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Nonwovens

An Advanced Tutorial

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Foreword

Anyone seeking to become acquainted with nonwoven technology soon realizes that a concise source of information is difficult to find. While many excellent articles have appeared they are so scattered that it is quite time consuming to obtain an overview of the field. In an attempt to overcome this problem, a 3-day "TUTORIAL" was held at the 10th International Chemical Congress in Toronto, Canada on June 6-8, 1988.

The sessions were designed to give an in-depth view of methods for making nonwovens, a thorough examination of absorbency testing and theory, fundamentals of fibers, process engineering and matrix interactions and some new end-use applications. The outstanding scientists responsible for coordinating and chairing symposia sessions were:

Dry and Wet Laid Systems - David R. Buchanan - N.C. State University
Alan Meierhofer - Dexter Corp.

Melt Blown and Spunbonded Technology - Larry Wadsworth -
University of Tennessee - Knoxville

Novel Applications - Richard Heitmiller - Consultant, Nashua, N.H.
Absorbency and Testing - P. K. Chatterjee - Personal Prod. Div.,
J & J., Milltown, N.J.

Without their dedicated efforts this venture could not have been accomplished and the editors express their deep gratitude for their cooperation.

The quality and scope of the papers exceeded the expectations of even the most seasoned colleagues in the nonwoven field. This book is being published to preserve and share this valuable information on fundamental and advanced nonwoven technology.

The book is organized in three sections:

Dry Laid and Wet Laid Systems
Melt Blown and Spun Bonded Technology
Absorbency and Testing

In the first section Subhash Batra outlines the history and definition of nonwovens and follows this with an excellent engineering approach of fibers as structural elements. Karl Pflugel then describes how Fehrer's machines can deliver uniform webs and Karl Maitre shows how Dilo's technology is involved in needle punching these air laid structures. For areas better served by binder technology rather than by mechanical or thermal bonding, Bill deVry shares the Rohm and Haas technology of latex bonding chemistry and processes. Finally, Philippe Coppin covers the latest in composites from wet laid high modulus fiber based structures.

The second section focuses on the direct formation on nonwovens from polymer melts. Herman Balk supplies an excellent overview of the latest Reicofil Technology for producing spun bonded and melt blown webs and then describes the Reicotex process for in-line bonding of such webs to structural fabrics, films and other substrates. Mancil Milligan of the University of Tennessee, Knoxville delineates the theory and fundamental engineering requirements for supplying the

large volumes of high velocity air required in melt blown operation, while his cohort Larry Wadsworth reports his extensive and detailed experience in evaluating the melt blown technology, parameters and product properties on the multimillion dollar pilot plant donated to the University by Exxon. Then Joe Spruiell shows how to get on-line characterization of product during melt blown operations. Chris Davey of Exxon reports on the important parameters of polyethylene and polypropylene if they are to be useful for melt blown applications while Steve Krupp reviews Dow's position in olefins and copolymers such as their new ethylene/octene ultra soft "ASPUN" fibers.

Having made the nonwovens, it is necessary to evaluate them. The third section is absolutely loaded with exciting new testing techniques and results on absorbency. To begin, J. E. Rice from Buckeye Cellulose Co. takes a critical look at the fundamental assumptions used in deriving the Lucas equation for predicting fluid flow in fibrous matrices and develops a mathematical model which gives better correlations of flow in low density structures. B. S. Gupta then details the effects of various common fluids and body fluids on absorbency, and John Berg reports his work at the University of Washington on single fiber wetting measurements for assessing absorbency. H. O. Wilson shows how he applied an ingenious technique at Hercules to overcome the inadequacy of the Lucas-Washburn equation to predict performance as the absorbent material changes density, pore size and wicking during wet out.

The frosting on the cake, however, comes in the final three papers. In a classic paper, Carl Gryte of Columbia University reveals the initial use of x-ray computer tomography to give a three-dimensional distribution of water flow through diapers and cateminal products with and without super absorbent gels. Bernie Miller reports a totally new method for monitoring resistance to forced passage of liquid and accurately measuring break through time and pressure. This method should certainly replace the present inadequate techniques. Finally, Ty Vigo tells how he applied his thermal storage and release treatments to not only obtain these new properties but also to significantly improve absorbency.

As conference chairmen and editors, it has been our privilege to be part of this attempt to produce a concise source of fundamental, current and new information in the nonwoven field. The results of this tutorial exceeded our fondest hopes. We hope you, the readers, agree that the effort was worthwhile.

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A New System for Classifying Textiles*

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1. Introduction

To establish the beginnings of "nonwovens" is a problem of considerable challenge.

First, there are examples in nature which can be construed as the forerunners of "nonwovens". These include the nests of many birds, mammals, and invertebrates; the "spunbonded"-like cocoon produced by the silk worm; the spider webs, which can also be thought of as "spunbonded" "nonwovens"; the root structures of trees, shrubs, and grasses, which serve as natural erosion control akin to modern day geotextiles; the fibrous network, which constitute the skeletal structure of bark and leaf components of shrubs and trees; the extremely fine, "spunbonded"-like, fibrous structure of the every day egg-shell; the paper-like hornets nets; and so on.

Second, fibrous network structures were made from papyrus by the ancient Egyptians as early as 2400 B.C. [1]. Beaten bark cloth made from the inner bark of the trees of the mulberry family (paper mulberry, bread fruit, wild fig, etc.) has been in use in Oceania, Africa, Southeast Asia, and the Americas from prehistoric times; several layers of this cloth can be bonded together by further use of moisture and beating which presumably releases the "inner resins" [2] and provide the needed binding material.

Third, according to Gordon [2]:

"There are several similar recurring legends about the discovery of felt. In one, Noah attempted to make his ark more comfortable by padding the floor with sheep's wool. By the time the journey of 40 days and nights had passed, the loose wool had turned to a matted fabric (felt) by pressure and moisture the animals had subjected it to.

"Another legend placed the discovery in France in the Middle Ages. A monk who lived in the city of Caen decided to make a pilgrimage to a distant shrine. He set out wearing a pair of new sandals and his feet soon became sore and tired. To make them more comfortable, he picked some wool from the backs of the sheep and put it in his sandals. When he arrived at his destination after 15 days of walking, he found a strong soft cloth had been created by the constant moisture and pressure of his feet.

"An almost identical story takes place in the Middle East, where a tired, foot-sore camel driver took some of the soft hair from his camels and put it in his sandals. By the time the caravan reached its destination, he, too, had discovered felt.

"...it is generally believed that it was the nomadic people of Central Asia who first learned to make it...

"The oldest pieces of fabric to have been found date from about 1500 - 1000 B.C. Felt caps as old as 3,500 years have been found in Scandinavia, and several items from the later Bronze Age (1400 - 1200 B.C.) have been found in tombs in northern Germany and Siberia..."

* Part of this paper was presented at the 13th Annual Technical Symposium INDA, held in Boston, June 1985.

Fourth, Ruiji and Ke'an [3] speculate that the invention of paper, which is a form of "nonwovens", "during the early Western Han Dynasty (206 B.C. - 8 A.D.) was most probably introduced from silk floss making. To make the silk floss the "waste" cocoons were beaten on a bamboo screen in water. After thorough beating, there was a layer of silk fiberweb left on the screen. This web-form material might have been called and used as paper..." They also suggest that, according to "recent" archaeological evidence, paper made from hemp was introduced in China, as early as the second century B.C. More reliable records in Han Shu (The History of the Han Dynasty) state that Cai Lun made great improvements in paper making in 105 A.D. Other sources assert that paper was introduced, in China, by Ts'ai Lun; it could be made from "bark, hemp, fish net and rags" [4]. The art of Washi, paper making by hand, using hemp and fibers from mulberry or certain deciduous trees, dates back to 6th century A.D. in Japan [5].

Fifth, if a silk worm, ready to spin, drops on the floor, it will not stop spinning; the spinning effort will thus result in a "flat cocoon" or "cocoon paper", akin to the modern day spunbonded web. This was known to the ancient Chinese [3]. Earliest records of the technological utilization of this phenomenon date back to the Song Dynasty (960 - 1279 A.D) [3].

Sixth, the industrial process of paper making was first introduced in France by Henry and Sealy Fourdrinier in 1803 [4]. It was based on adaptation of a machine invented by another Frenchman, Louis Robert, in 1799 [1]. Similarly the industrial process of wool felt making was invented by an American, J. R. Williams, in 1820 [2].

Seventh, both Millman [6] and Wagner [1] suggest that the invention of "nonwovens" can be traced to a British patent issued in 1853. According to Wagner [1], "the invention of making 'nonwovens' can be traced to British Letters Patent Number 114, granted in 1853 to Belford, which revealed the use of cards, conveyor belting, impregnating, mattresses, cushions, etc."

Eighth, Tyas [7] claims that the production of needle looms was established around 1870 by William Bywater Ltd.; whereas, Ward [8] asserts that "one of the first, if not the first, needleloom was built by ... Bywater ... in 1878 and delivered to a Leeds woolen manufacturer, Hargreave & Mussey, for making horse blankets."

Thus, while the antecedents of the "nonwovens" technology and products can be traced to various points in human history, most writers tend to place the beginning of the "nonwovens" industry, as we know it today, to the late 1920s or early 1930s. Nottebohm [9] cites a German patent (DRP Nr.544324), dated 26.8 1928, which claimed the production of a fleece in combination with a glued web to serve as a shoe reinforcing material; it is still (1968) produced by the Kalff firm. He also cites a Dutch Blaupotten Cate process disclosure, in 1930, for the manufacture of artificial leather backing from fiber fleece to be glued with natural rubber. The earliest patent cited by Buresh [10] is dated March 12, 1929 or April 29, 1930, depending upon one's interpretation as to their relevance specifically to "nonwovens" technology.

According to Hayes, cited in [11], laboratory and pilot scale production of "nonwovens" started in the 1930s. Shearer [12], and Millman [6] put the industrial production of "bonded-fiber nonwoven fabrics" in commercial quantities (a few thousand pounds) around 1942.

To conclude this overview of the history of nonwoven product development, it is useful to recognize some subtle, but very important differences in the development of technologies for making woven, knit, and nonwoven fabrics.

The principles of weaving have been embodied in the loom ever since its earliest inception. Most subsequent developments in weaving technology have been aimed at improving the design potential of the loom, quality of the cloth it produces, its efficiency and productivity, and so on. Similarly, the principles of knitting have been embodied in the circular or warp knitting machines, which have been improved, over the years, for their design potential, the quality of the cloth they produce, their efficiency and productivity.

The introduction of fiber-to-web (or sheet) structures, as viable textile materials, was a radically new idea -- a very significant departure from the conventional thought. It appeared concomitantly with another radically new idea: namely, that quite useful textile fibers could be obtained from sources other than plants, sheep, or worms. These two innovative notions nurtured each other and unleashed the creative energies of many individuals and organizations.

Thus were born the many technologies to fabricate products presently called "nonwovens." In the early stages of their development, more often than not, the fabrication technologies evolved independent of each other. As such they differ quite markedly in their use of the necessary engineering systems and subsystems to accomplish their goals. The common theme, if any, seemed to be that they all tried to accomplish four basic functions of (1) fiber preparation or extrusion, (2) web formation, (3) bonding, and (4) finishing, sequentially or simultaneously. Not all so called "nonwovens", however, seemed to result from processes which could be described so uniformly and neatly.

2. History of the Definitions

The "evolutionary" growth rate of this industry and the performance versatility of the resulting products, has bordered on being revolutionary, even chaotic, albeit a healthy chaos. This has been reflected in the problem of defining the nature of the products. Kovacs, in attempting to define the term "nonwovens" in his book [13], said something to the effect (in translation): paradoxically, the term nonwoven is difficult to define; the best definition is that it is not woven. Others have been less discouraged.

Krcma [14], who struggled with this issue, suggested that the term "nonwoven fabric" was coined in the early stages of development of the technology and products. Subsequently, as the development continued, the scope of the original term changed gradually. Shearer in 1952 [12] called the then existing structures "bonded-fiber fabrics" and "bonded-fiber nonwoven fabrics." Sherwood [15] quoted a definition by the Textile Organon which he asserted had received wide acceptance. It was: "a web of parallel, cross-laid or randomly dispersed fibers is held together by an applied bonding material or by the fusion of self-contained thermoplastic fibers through heat and/or pressure." Buresh [10] in 1962 used the ASTM (Committee D-13 on Textile Materials) definition which defined these materials as textile structures consisting of a web or a mat of fibers held together with a bonding material. Both the ASTM and the Textile Organon definitions seemed to be attempts at describing the fibrous web-like structures as "nonwovens".

Slightly earlier, in Europe, Meyer (1960) [16], Jorder (1961) [17] and Krcma [14] made their contributions to the definition of the term "nonwoven fabrics". The first two of these are not available to us at this time for inclusion here. Krcma, however, suggested that the term also include "production techniques and products containing fibers in higher textile processed forms, i.e. yarns or threads, as well as combined (laminated) products both chemically and mechanically bonded." According to him, these suggestions were accepted by the East European countries, as well as incorporated in the German standard, DIN Norm 61 210 (Textilien-Grundbegriffe). Thus Krcma was acknowledging the use of yarns together with fibrous webs in making some of the "nonwovens". The pressure to

enlarge the scope of the definition to include wider range of structures was mounting. Even the German term "Vliesstoffe", literally meaning fleece material, or fleece fabric, or fiberweb, originally coined to represent "nonwovens" was becoming increasingly inadequate [14].

Millman [6] in 1969 suggested: "The term 'nonwoven' has many interpretations, none widely accepted. In a broad sense nonwovens are products made without a loom, a literal meaning that encompasses felt, knitted and tufted goods as well as a long list of non-textile products." This was apparently far too broad; indeed, even the narrower interpretation of Krcma was not being accepted. For Newton in his review of the progress in "nonwovens" [18] in 1973 stated: "The definition of 'nonwoven fabric' and its negative implications are still topics that fascinate journalists; the subject was discussed at the 11th International Colloquium on Nonwovens at Brno. The generally accepted understanding of the term as referring to fabrics made from fiber rather than from yarn tends to put stitch-bonded fabrics on the fringe. Even though a number of nonwoven fabrics contain yarn, usually as a reinforcement, it is chiefly the stitch-bonded fabrics that have yarn as a major constituent."

During the 1970s, the ASTM definition(s) [19] of "nonwovens" were:

"... n. - a structure produced by bonding or interlocking of fibers, or both, accomplished by mechanical, chemical, thermal, or solvent means, and combinations thereof. The term does not include paper or fabrics that are woven, knitted, tufted, or those made by wool or other felting processes.
D 1117, D-13

"... - a planar structure produced by the bonding or interlocking of textile fibers, or both, normally accomplished by mechanical work, thermal treatments, chemical or solvent actions or combinations thereof.
D 2646, D-13

"... n. - a textile structure produced by bonding or interlocking of fibers, or both, accomplished by mechanical, chemical, or solvent means, and combinations thereof. ...

"Note - The term does not include paper, or fabrics which are woven, knitted, tufted, or those made by wool or other felting processes.
D 123, D-13."

In other words, ASTM definitions of "nonwoven" structures were still restricted to bonded fibrous webs; any structure containing yarn in any form was excluded. Thus not only stitch-bonded but fibrillated film network, scrimmed and various other modern day combined structures were excluded.

In 1978, INDA [20] offered its version of the "nonwoven" fabric definition:

"Nonwoven fabrics are generally defined as sheet or web structures made by bonding and interlocking fibers, yarns or filaments by mechanical, thermal, chemical or solvent means."

While this definition is very precise, it is also broad enough to include the stitch-bonded and other modern structures, together with paper, wovens, knits, and every other conceivable textile structure.

In contrast, the current European definition as formulated by EDANA [21] is:

"Nonwovens are a manufactured sheet, web, or batt of directional or random orientated fibers, produced and bonded by mechanical, chemical, or physical means (excluding weaving, knitting, tufting, stitch-bonding, and traditional felting).

"The fibers may be of natural or man-made origin. In the latter case, they may be staple or continuous filaments or be formed in situ.

"N.B. Nonwovens are distinguished from paper, notably by the bulk and rupture energy criteria as outlined as follows (in order of ease of control).

- a) if it is made of 100% man-made fibers
or then
- b) if its bulk index is above 7
or
if its total rupture energy index is higher than 7 joules
or then
- c) if together its total rupture index is between 7 and 1, 2 joules AND its bulk index between 7 and 2."

As Purdy [22] points out: "Such a definition would appear to exclude many structures produced for industrial applications that are made by needling fibers onto woven scrims or basecloths." Moreover, the distinction between "paper" and "nonwoven" appears to be somewhat arbitrary.

From the above review of the history of definitions, it becomes painfully obvious that as long as we stick to the single phrase "nonwoven" and expect it to include a very broad range of textile structures, while at the same time append to it a long list of exclusions, we condemn ourselves to live with chaos and confusion. The question is: must we? Or is it possible to break through the conundrum? We propose a possible solution.

3. Proposed Classification Scheme and Definitions

Let us divide all textile fabrics which are neither woven or knit into three classes:

1. Fiberweb structures. This class should include all textile sheet structures made from fibrous web, bonded by frictional/mechanical constraints and/or covalent bonds obtained through the use of added resins, thermal fusion or formation of chemical complexes. Here, fibers (or filaments) are the fundamental units of structure arranged into a web and bonded so that the average bond-to-bond distances are greater than, say, 50-100 times the fiber diameter. It is this fundamental characteristic feature which gives them the "textile-like" quality of lower bending and in-plane shear stiffness and distinguishes them from paper.

Paper, here, is viewed as a fiberweb containing cellulosic wood pulp fibers ranging in length from 2-4 mm, which are bonded together by hydrogen bonding, and wherein the bond-to-bond distances are of the order of fiber diameter.

This class therefore excludes paper, but includes carded- or air-laid/bonded, spunlaced, spunbonded, meltblown and needlepunched structures as we know them today. It will also include Malivlies and Arabeva type structures. Properties of structures in this class are determined by the geometrical arrangement of the fibers in the web, the properties of the constituent fibers and the properties of the binders, if any. Finishing treatments may be involved in altering the mechanical properties of the fiber (as in heat setting), fiber web geometry (as in calendering or embossing), or physico-chemical characteristics of some or all of the constituent fibers (as in the modification of the electrostatic, surface tension, hydrophobicity-to-hydrophilicity, etc., characteristics).

Together with 'singles' yarns, structures in this class should be considered as primary structures. In this sense, plied yarns, woven and knit fabrics are secondary structures, inasmuch as they represent the next level in the hierarchy of fibrous structures.

2. Net-like structures. This class should include all textile structures formed by extruding one or more fiber-forming polymers in the form of a film or a network of ligaments. The film may be subsequently stretched uniaxially/biaxially to fibrillate it into an irregular net-like structure, as in the case of Sharnet®, a proprietary product made with the help of blowing agents in the molten polymer, or highly regular as in the case of Conwed® netting. There may be other variants.

3. Multiplex structures. The term multiplex is commonly used in audio and communication technology. One of its dictionary meanings is: multiple or manifold; another is: being or relating to a system of transmitting several messages simultaneously on the same circuit or channel. Extending these concepts for our purposes, we might define multiplex structures as that class of textile fabrics which combine and utilize the characteristic properties of several (two or more) primary and/or secondary structures, at least one of which is a recognized textile structure, into a single unitized structure. This, as we shall see, is a very broad yet intuitively satisfactory definition. For example, it would include all the stitch-bonded structures which may use unbonded or bonded fiberwebs, paper, 'warp' and/or cross ('filling') yarns of varying color and/or texture, any sheet material which can be penetrated by the stitch/knitting needles, in some desirable combination. It would also include laminates, tufted structures, scrimmed structures, and those that are loosely called composites.

In the past, three terms have been used, somewhat informally, to represent the class of fabrics which do not belong in either the fiberweb category or the net-like category. These are: composites, complex, and hybrid. Each of these in our opinion has limitations. First, the term composites has had a very well established meaning in material science. It connotes two (or more) phase materials in which one phase, serving as the matrix, completely encapsulates the other; there is a well established bond at every point of the interface between the two phases. These are not the type of structures we wish to describe here. Second, the term complex connotes complexity in manufacturing, describing, or understanding the behavior of the structures. While this may be true in some cases, it is not true in all of the diversity of structures we wish to deal with in some unified conceptual sense. Therefore, we believe the term complex is an undesirable descriptor of this class of structures.

The term hybrid is a very attractive one, for it suggests the result of combining two or more heterogeneous species, varieties or races. This much is applicable to our class of structures. But while hybridization in botany or biology produces a product which maintains a certain combination of the desirable characteristics of the precursor species, it also produces a new homogeneity in which the individuality of the precursors is lost. Such is not the case with the class of fabrics we wish to classify here. The multiplexing concept does not impose this restriction. In fact, in multiplexing, the component structures enhance each other more often than diminish each other. Multiplexing, therefore, is more descriptive of our needs.

Finally, the term unconventional fabrics has been often used, loosely, to categorize structures that are neither woven nor knit. The term is totally nondescriptive. Moreover, most of these structures have been around for twenty or more years. It is high time we thought of them in more descriptive terms.

4. Advantages of the Proposed System

Unlike previous classification schemes, the proposed system is not technology specific; it puts the emphasis on the structural characteristics and performance of the products. Even more to the point, it acknowledges a philosophy of structure development (or engineering)

which has always existed in the textile industrial activity, but not as part of the mainstream. It has been rooted in the need to develop textile structures with special features or properties not attainable in the conventional woven, knit, or fiberweb structures. This has been accomplished by modification of the existing technologies (e.g. velcro, weft inserted warp knitting) to be sure, as well as by development of new technologies (e.g. tufting, Malimo), or combination of technologies and structures (or multiplexing) (e.g. production of flocked blankets, production of scrimmed "nonwovens", etc.). As such, it has served limited, though quite sophisticated specialty markets.

In the last ten to fifteen years, this activity has gained considerable momentum, resulting in the appearance of a very broad range of products. Multiplex structures, as defined above, have been and are being developed at an accelerated rate through the use of newly developed technologies or through the combination of technologies. Indeed, the whole concept of what textiles are, what they are supposed to accomplish and how they ought to be fabricated, has been undergoing a quiet but invigorating renaissance. To acknowledge it as such is both timely and beneficial.

Benefits of this acknowledgment can be many-fold. It frees the imagination of the Textile Engineer/Designer from the constraints of the conventional or existing technologies and the limitations of the products made therefrom. As such, it opens up the possibility of product innovation and its consequences in the market place. That is, specially designed products with superior performance characteristics and competitive price structure will challenge the commodity approach to textiles in specific markets; for example, industrial fabrics. Innovative products could also penetrate the "high-technology" structural materials markets wherein fibrous structures will outperform traditional materials in cost or in performance or both.

Finally, the proposed classification system has built-in versatility. While it will be possible to accommodate most new flexible, textile-like structures in the Multiplex category, it will also be able to admit new generic categories not yet conceived.

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FIBERS AS STRUCTURAL ELEMENTS IN NONWOVENS*

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Abstract

This paper focuses on the role of fibers as the basic elements of structure in the fabrication of nonwoven fabrics. As such, the mechanical properties of fibers contribute to the mechanical performance of the nonwoven. The nature of this contribution is influenced by the structure of the nonwoven assembly, the presence of binders and other additives if any, and environmental factors. In this connection a few selected mechanical properties of fibers, in the small strain range, are reviewed and their influence on fabric properties is discussed.

1. Introduction

In point of fact the mechanical properties constitute a subgroup of the totality of the physical properties of fibers. And yet, to acknowledge their immense importance, the mechanical properties are sometimes spoken of as though they were distinct from physical properties. In this framework, the physical properties generally encompass the physical size (length, fineness), cross-sectional shapes, optical characteristics, fluid imbibition, thermal and electrical conductivity, density, melting or degradation temperatures, dimensional changes related to changes in ambient temperature and relative humidity, frictional characteristics, etc. Mechanical properties, on the other hand, relate to those properties that determine deformational response of fibers (including failure) to imposed forces (including moments), or vice versa, modification of these responses under changing conditions of temperature and humidity, and the influence of time as a parameter during the duration of imposed loading or deformation.

Technologically, the interest in the knowledge of the mechanical/physical properties of the fibers as such is principally at the phenomenological level. Fibers can be therefore treated as continuum entities and their properties can be determined/measured, albeit often with considerable degree of difficulty posed by their fineness and slenderness (or aspect) ratio. From a practical point of view, the properties of fibrous assemblies, which govern their end use performance, are a function of (1) the properties of the fibers, (2) the characteristics of the structure of the assembly (3) the properties of additives, if any, and (4) the environmental conditions. More to the point, the properties of the assembly are often determined by a variety of complex interactions among these four factors. The nature of these interactions, more often than not, and most regrettably, is poorly understood.

It is in this context that the role of a few selected physical and mechanical properties of fibers, as elements of structure, in nonwoven applications is briefly examined and reviewed. In particular, small local-deformations is emphasized.

From the macroscopic point of view, fibers in common usage are usually homogeneous materials. For special effects, or other reasons, sometimes sheath-core or conjugate bicomponent fibers may be used. Discussion in this paper is restricted to the characteristic properties of homogeneous fibers with solid or hollow cross sections.

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2. Stress-Strain Behavior

Fibers most commonly used in nonwoven applications are oriented polymeric materials wherein the fiber axis coincides predominantly with the direction of molecular orientation. Therefore, at very low levels of local deformations (small strains), their mechanical behavior is modeled quite adequately by transverse isotropy, characterized by five elastic constants [1]. These are the elastic modulus along the fiber axis, elastic modulus and shear modulus in the plane of the cross section, shear modulus and Poisson's ratio in the axial planes. In this characterization, it is implicit that the two elastic modulus values are independent of whether the corresponding normal strains are extensional or compressive. From a practical point of view, however, it is the modulus along the axial direction and the shear modulus in the cross-sectional plane that are most important, unless the fibers are subjected to lateral forces or deformations.

The stress (or specific stress) vs. strain curves of typical fibers appear as shown in Figure 1 [2]. They are obtained by deforming the fibers in axial extension. The slope of each curve for very low values of strain (at $\epsilon = 0$) yields the so called initial modulus value which represents the modulus along the axial direction. For inorganic engineering materials this slope does not change if the sample is subjected to compression instead of extension, but for some polymeric fibers the limited experimental evidence suggests that this may not be true [3]. The different values of the elastic modulus in (axial) extension and compression have a specific implication in the bending behavior the fibers which will be discussed in a later section.

Cox [4] was one of the earliest to recognize the role of the (axial) elastic modulus of fibers in the nonwoven fabric properties, and proposed a rigorous theoretical model relating the two. He postulated that at small strains the elastic modulus of the constituent fibers together with their orientation distribution function determine the initial stiffness and other elastic properties of fiber-network-structures, in which the fibers lie essentially straight over distances somewhat larger than the average bond-to-bond distance. Such fiber-network structures include spunbonded and other area bonded, dense, planar nonwovens.

Techniques for measuring the shear modulus in the plane of the cross section of the fiber have been developed [5,6], and data for several fibers with circular cross sections have been obtained [1,5,6]. For larger size fibers (eg. monofilaments) even other constants can be measured [1,7,8,9], but their determination for finer fibers is nearly impossible. The role of the shear modulus (in the plane of the cross section) of the fiber in nonwovens is discussed in a later section, which deals with the deformational behavior of crimped fibers. The importance of the other elastic parameters of the fibers [7,8,9] in nonwoven structures has not yet been recognized to any significant degree.

3. Bending Behavior

The bending behavior considered here is restricted to deformations in which local strains in the fiber are small. Thus the small-strain theory of elasticity provides an adequate description of the phenomenon. Under these conditions the so-called Engineering-Beam Theory [10] provides the framework in which the bending behavior of the fibers can be examined. The cross-sectional shape and its orientation relative to the plane of bending play a significant role in determining the behavior.

Consider a fiber (beam, prismatic bar) and its cross section, somewhere along its length, as shown in Figure 2(a); the x-axis is directed parallel to the axis of the fiber. If the fiber is being used to transmit pure bending, then every element of its length is subjected to the bending moment M_b in the OXY-plane and it bends into a circular arc of "radius" R , as shown in Figure 2(b). The longitudinal strain ϵ_x at any point, surrounded by area element dA , in the cross section and at a height y from the OZ-axis is given by

$$\epsilon_x = \frac{y}{R} \quad (1)$$

provided the OXZ-plane is the neutral plane; that is, all points in the OXZ-plane have zero strain. If the OXY-plane is a plane of symmetry and O is the centroid of the area of cross-section ($y \, dA = z \, dA = 0$), then the radius of curvature R of the OXZ-plane is related to the applied bending moment M_b by

$$\frac{1}{R} = \frac{M_b}{EI_{zz}} \quad (2)$$

where E is the elastic modulus of the material along the x-direction (both in tension and compression) and I_{zz} is the area moment of inertia of the cross section about the z-axis given by

$$\int_A y^2 \, dy \, dz = I_{zz} \quad (3)$$

The reciprocal of the radius of curvature R is often called the curvature κ of bending; $\kappa = 0$ implies $R \rightarrow \infty$, i.e., the fiber is straight. From Eq. (2) it is clear that the curvature κ is inversely proportional to the factor $E I_{zz}$, which is therefore called the "Bending Rigidity or Stiffness" B . Equation (2) can therefore be rewritten as

$$\kappa = \frac{1}{R} = \frac{M_b}{B} \quad (4)$$

Two points are worth noting at this stage: (a) If the (axial) elastic modulus in tension is different from that in compression, the neutral plane will shift away from, but remain parallel, to the indicated OYZ-plane. (b) The bending stiffness B depends not only on the initial elastic modulus in extension (and in compression, if they are different) but also on the geometrical shape of the cross section and its orientation to the plane of bending; the latter factors determine I_{zz} .

Inasmuch as the fiber cross-sectional shapes are engineered to accomplish other objectives (reflectance properties, power to hide soil, etc.), their influence on bending rigidity should be kept in a proper perspective. For fiber cross sections with two or more planes of symmetry, such as those shown in Figure 3, there will always be two planes in which the bending rigidity will attain maximum and minimum values. The fibers will, of course, be much more prone to bend in the plane of minimum bending rigidity. Circular cross section fibers will have, naturally, no such preferred planes of bending.

For fibers with cross-sectional shapes, with no planes of symmetry, such as one shown in the Figure 4, the bending behavior still can be defined in terms of a bending rigidity, similar to the symmetrical case, provided a special set of coordinate axes is defined. Let O be the centroid of the area of cross section. Let axes Y, Z be chosen as the principal axes of inertia, which means that the product of inertia $I_{yz} = \int_A y \, z \, dy \, dz = 0$.

Now if a bending moment M_{bz} along the z-direction is applied, the OXZ-plane will be the neutral plane, whose radius of curvature R will be given by

$$\frac{1}{R} = \frac{M_{bz}}{EI_{zz}} \quad (5)$$

where I_{zz} is the integral defined in Eq(3). Similarly, if a bending moment M_{by} along the y-direction is applied the OXY-plane becomes the neutral plane, whose radius of curvature R is given by

$$\frac{1}{R} = -\frac{M_{by}}{EI_{yy}} \quad (6)$$

where $I_{yy} = \int z^2 dy dz$. The preferred plane of bending for such a cross-sectional shape will be OXZ or OXY depending upon whether $I_{yy} < I_{zz}$ or vice versa.

The bending behavior of fibers constitutes an important influence on the (fabric) bending stiffness, the inplane (fabric) compressive stiffness and the drape of dense nonwovens made from noncrimped fibers. It is of equal importance in determining the resilience and hand properties of bulky, air-laid nonwovens using uncrimped fibers. Similarly the bending stiffness of fibers will influence the needle punching process and the bulk, stiffness and hand of the resulting products.

4. Transmission of Shear

Fibers may be bent by laterally applied, localized forces as illustrated in Figure 5. In Figure 5 (a) the shear forces transmitted in sections AB and CD equal F ; between points B and C the fiber is bent by a pure bending moment with magnitude Fm . When used to transmit shear forces, shear stresses develop in the fiber as shown in Figure 5 (b) and (c). If the shear force transmitted by a cross section along the fiber axis is F , in the y-direction, then the shear stress τ_{xy} in the fiber at a point, at height y from the OXZ-plane (Figure 5c), is given by

$$\tau_{xy} = \tau_{yx} = \frac{FQ}{bI_{zz}} \quad (7)$$

$$\text{and } Q = \int_{A'} y dA \quad (8)$$

where Q is the first moment of the shaded area A' about the z-axis and b is the width of the cross section at height y .

Inasmuch as some materials are prone to failure in shear (eg. Kevlar), calculation of the maximum shear stress generated in the fiber under certain loading conditions may become important. In nonwoven manufacture such critical situations may arise in the needle punching process. For the present purpose it is particularly important to note that the fiber cross section shapes play an important role (through factors Q , b , and I_{zz}) in determining the intensity of shear stress generated in the fiber.

5. Buckling Behavior

A critical issue in the load-transmission of slender rods is their buckling behavior. Three cases of fiber (slender rods) buckling are of particular interest in the case of nonwovens. These are called hinged/hinged, built-in/built-in and hinged/built-in cases [10] and are sketched in Figure 6. The critical load beyond which a fiber in a nonwoven could buckle is determined by (1) its bending rigidity (EI), (2) its length between two consecutive

bonds involved in load-transmission, (3) a multiplicative constant which depends upon the nature of bonds (whether they can be closely modeled by a hinge or a built-in end condition), and (4), most importantly, the nature and extent of any interference from the neighboring fibers. In any event, the buckling behavior of the fiber influences significantly the inplane compression, bending/buckling/drape of the nonwoven structures.

6. Torsional Behavior

A fiber with a circular cross section subjected to a twisting moment M_t deforms (twists) as shown in Figure 7. The angle of twist per unit length α is related to the twisting moment M_t by

$$\alpha = \frac{d\phi}{dz} = \frac{M_t}{GI_p} \quad (9)$$

where G is the shear modulus of the material in the plane of the cross section, I_p is called the polar moment of inertia (about the centroidal z -axis) defined by

$$I_p = \int_0^R \int_0^{2\pi} r^2 (r d\theta) dr = \frac{\pi R^4}{2} \quad (10)$$

The shear stress $\tau_{\theta z}$ at a point in the cross section at radius r from the center is given by

$$\tau_{\theta z} = \frac{M_t r}{GI_p} \quad (11)$$

Equation (9) suggests that quantity GI_p represents the resistance of the fiber to the twisting deformation and is, therefore, termed the torsional rigidity of the fiber.

The torsional behavior of a fiber with non-circular cross section is not as simple. For fibers with elliptical cross section (major axis = a , minor axis = b , area $A = 4\pi ab$, $I = \pi ab(b^2 + a^2)/4$), the torsional rigidity is given by

$$\frac{GA^4}{4\pi^2 I_p} = \frac{G\pi a^3 b^3}{a^2 + b^2} \quad (12)$$

Similarly, for a fiber with equilateral triangle cross section the torsional rigidity is given by

$$\frac{3}{5} GI_p \quad (13)$$

where I is the polar moment of inertia of the cross section about its centroidal axis.

The torsional rigidity of any other cross-sectional shaped fiber can be determined through fairly elaborate theoretical analyses and/or experimental procedures based on the membrane analogy of Prandtl or the soap bubble analogy [11], etc. In this connection it is useful to bear in mind St. Venant's conclusion [11]: For a given cross-sectional area of a (non hollow) fiber, the torsional rigidity increases if the polar moment of inertia of the cross section decreases. Consequently, for a given area of cross section the circular cross section

gives the greatest torsional rigidity. For an arbitrary shaped cross section the torsional rigidity C can be estimated by

$$C = G \frac{\pi a^3 b^3}{a^2 + b^2} \quad (14)$$

where the arbitrary cross section is replaced by an ellipse of equal area and equal polar moment of inertia.

The torsional properties of fibers become particularly important in influencing the behavior of nonwovens when crimped fibers are used, as is illustrated in the next section.

7. Load-Extension of Crimped Fibers

Two models of crimp in fibers are considered here: First, is the planar crimp in which the stable configuration can be modeled by a series of semi-circular half waves, as shown in Figure 8(a). Second, is the three dimensional crimp in which the stable configuration of the fiber is modeled by a helical spring, as shown in Figure 8(b).

The load-extension behavior of the planar crimp fiber can be estimated [11] by

$$\delta = \frac{\pi P R^3}{EI} \quad (15)$$

where δ is the extension of one full wave-length, made up of two semi-circular segments of radius R , under an extensional load P with EI as the bending stiffness in the plane of the crimp. Equation (15) can be expressed as an apparent strain ϵ , defined by

$$\epsilon = \frac{\delta}{4R} = \frac{\pi P R^2}{4EI} \quad (16)$$

$$\text{or} \quad P = \frac{4EI}{\pi R^2} \epsilon \quad (17)$$

Thus stiffness of the planar crimp is directly proportional to the elastic modulus of the fiber modified by the factor $\frac{4I}{\pi R^2}$. For a typical textile grade 1.5 denier polyester fiber with circular cross section with 4.0 crimps/cm (10 cpi), the effective modulus becomes $31.32 \times 10^{-6}E$; that is, it is reduced by about five orders of magnitude.

The load-extension behavior of a helically wound crimped fiber must be divided into two categories: (a) close pitch coils (or very high degree of crimp), and (b) large pitch coils. For the close pitch coil case, the deflection δ under an axial load P is given by [12]

$$\delta = \frac{P R^3 2\pi n \sin q}{C} \quad (18)$$

where R is the radius of the helix, q is its helix angle, n the number of turns and C the torsional rigidity of the fiber. Alternatively,

$$P = \frac{C \cos q}{R^2 \sin^2 q} \epsilon \quad (19)$$

where ϵ , the apparent strain, is based on the length of the helix (n turns $\times$ pitch). Thus the stiffness of the fiber is directly proportional to the torsional rigidity of the fiber, inversely proportional to the square of the radius R of the helix and modified by the helix angle function $\cos q / \sin^2 q$.

For a 1.5 denier polyester fiber of circular cross section with 10 crimps/cm and a helix radius of 1mm, the effective modulus becomes $3.09 \times 10^{-6}G$ --a value six orders of magnitude lower than the shear modulus of the fiber in the plane of its cross section.

For the large pitch coil case, the deflection δ under an axial load P is given by [12]

$$\delta = \frac{2\pi n PR^3}{\sin q} \left[\frac{\sin^2 q}{C} + \frac{\cos^2 q}{EI} \right] \quad (20)$$

Alternatively,

$$P = \epsilon \left\{ \frac{\cos q}{R^2} \left[\frac{\sin^2 q}{C} + \frac{\cos^2 q}{EI} \right]^{-1} \right\} \quad (21)$$

Thus the stiffness of the fiber is $\frac{\cos q}{R^2} \left[\frac{\sin^2 q}{C} + \frac{\cos^2 q}{EI} \right]^{-1}$. For a 1.5 denier polyester fiber with 2 crimps/cm and radius of helix 1 mm, the effective modulus of the fiber becomes

$$19.45 \times 10^{-6} \frac{G}{[1+1.27 G/E]} ;$$

in other words, a reduction of five orders of magnitude relative to the shear modulus of the fiber in the plane of its cross section.

Thus a nonwoven fabric made from crimped fibers has the potential to be very soft, extensible and flexible, with good drape and hand, provided the structure of the fiber assembly and the bonding systems used do not, unduly, restrict the fiber from performing at its optimum. The crimp configuration is exploited to obtain bulky nonwoven structures with adequate degree of resilience. In the carding process crimp in the fiber aids immeasurably in developing web cohesion by increasing considerably the frictional contact points between fibers.

8. Influence of Ambient Conditions

The discussion, thus far, has tacitly assumed that the values of elastic parameters of the fiber material remain constant. This, in general, is not true. For fiber forming polymeric materials, particularly, the values of these parameters often change significantly with changing ambient conditions [14,15,16,17]. Depending upon their chemical composition and morphological structure, different fibers have different propensity to absorb moisture (measured as moisture regain) which is further influenced by the ambient temperature and relative humidity. To the extent the absorbed moisture penetrates into the morphological structure of the fiber it influences its initial modulus. Comparison of values in the S and W₂₀ columns in Table I reveals that this influence (reduction) can be quite significant with fibers that have water retentions of 12 percent or better when wet or moisture regains (at standard conditions) of 4 percent or better (rayons, acetates, nylons). The initial modulus values of fibers such as polyester, polypropylene, polyethylene and acrylics remain essentially unaffected by this phenomenon.

The initial modulus of fibers in the wet state is further reduced by increasing ambient temperature (addition of heat). Comparisons of values in the S, W₂₀ and W₉₅ columns in Table I show that for fibers which are affected significantly by moisture (water) absorption, additional reduction in the initial modulus can be modest (wool and rayons) to significant (acetate, nylons). In contrast those that are essentially unaffected by the presence of moisture (polyester, acrylics, polyethylene, polypropylene) show relatively dramatic reductions in their initial modulus.

TABLE I Typical Values of Moisture Regain, Water Retention and the Initial Modulus of Selected Fibers --- the Latter at Various Treatments and Conditions [16].

Fiber (gpd)	Moisture Regain	Water Retention		Initial Modulus		
	%(a)	%(b)	S	S _T	W ₂₀	W ₉₅
Wool	16-18	44	25	-	10	5
Viscose Rayon	12.5	101	100	50	5	5
High-Tenacity Viscose Rayon	14.1	79	100	50	2	1.5
Polynosic Rayon	11.8	75	150	100	12	10
Cellulose Acetate	6.9	26	35	28	6	0.4
Nylon 66 (Type 200)	4.3	15	40	14	25	4.8
Nylon 6	4.4	12	30	7	15	2.5
Polyester	0.4	4	100	70	100	30
Acrylic	1.3	10	40	40	40	0.8
Polyethylene	0	2	50	30	50	6
Polypropylene	0	5	80	50	80	17
Glass	0	0	220	220	220	190
Kevlar 49*	3.5	-	880	-	-	-
Kevlar*	7.0	-	525	-	-	-
Nomex*	4.5	-	100	-	-	-

a. At standard conditions of 65% RH and 20 degrees C

b. After soaking in water and centrifuging for 7 minutes

c. S - As received, tested at standard conditions;

S_T - Treated in water at 95C, tested at standard conditions;

W₂₀ - As received, tested in water at 20 degrees C;

W₉₅ - As received, tested in water at 95 degrees C.

*Data obtained from manufacturer's (duPont) literature.

The influence of ambient conditions on other elastic properties has received less extensive or little attention. Based on Clayton and Pierce's observations, Meredith [17] showed that the shear modulus (in the plane of cross section) of cotton fiber decreases exponentially with increasing moisture regain. Similar behavior has been reported for acetate, nylon and viscose rayon by Permanyer, cited in reference (15).

From a structural property point of view, changes in the initial and shear moduli of fibers would cause changes in the stiffness properties of nonwoven fabrics to the extent that those (stiffness) properties are a consequence of the moduli of the constituent fibers.

9. Other Properties

Other mechanical properties of fibers that would influence the performance of a nonwoven include: visco-elastic behavior, large deformation behavior, recovery behavior, fatigue behavior, influence of thermo-mechanical treatments, etc. Investigation of the influence of these properties on the nonwoven fabric characteristics is clearly beyond the scope of this paper.

10. Summary

In summary, it is useful to reiterate that the performance characteristics of nonwovens are determined by a complex interaction of the properties of the constituent fibers, the structure of the assembly, the properties of the additives (binders) if used, and environmental influences. Prediction of the performance characteristics of any nonwoven fabric, therefore, requires a very careful analysis of all factors involved in the specific application.

11. Acknowledgements

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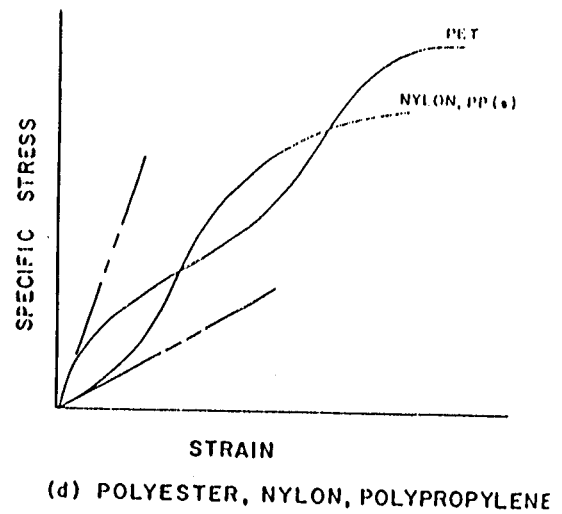
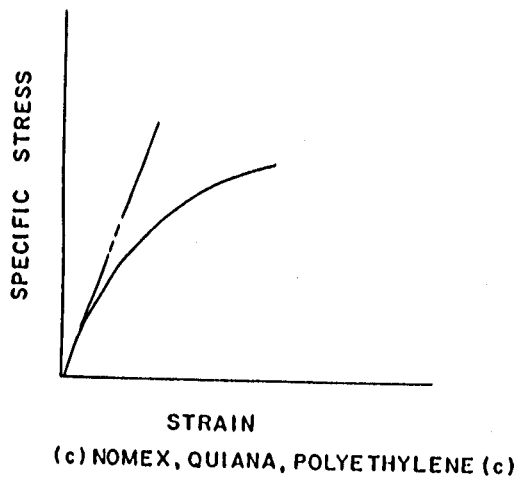
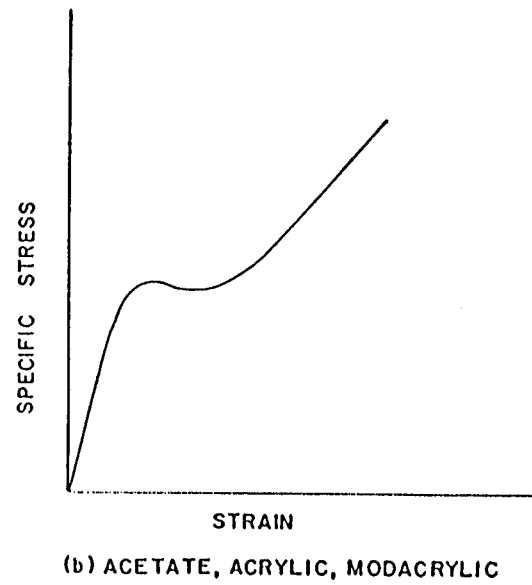
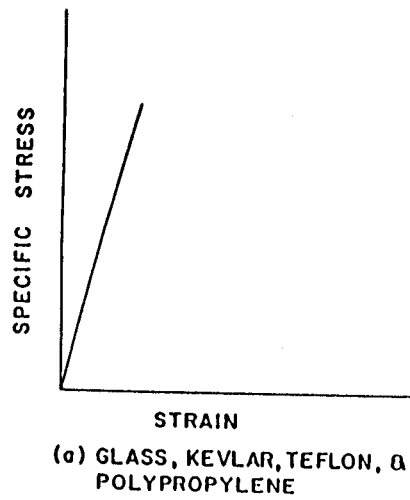


Figure 1. Qualitative Shapes of Stress Strain Curves of a Few Typical Fibers

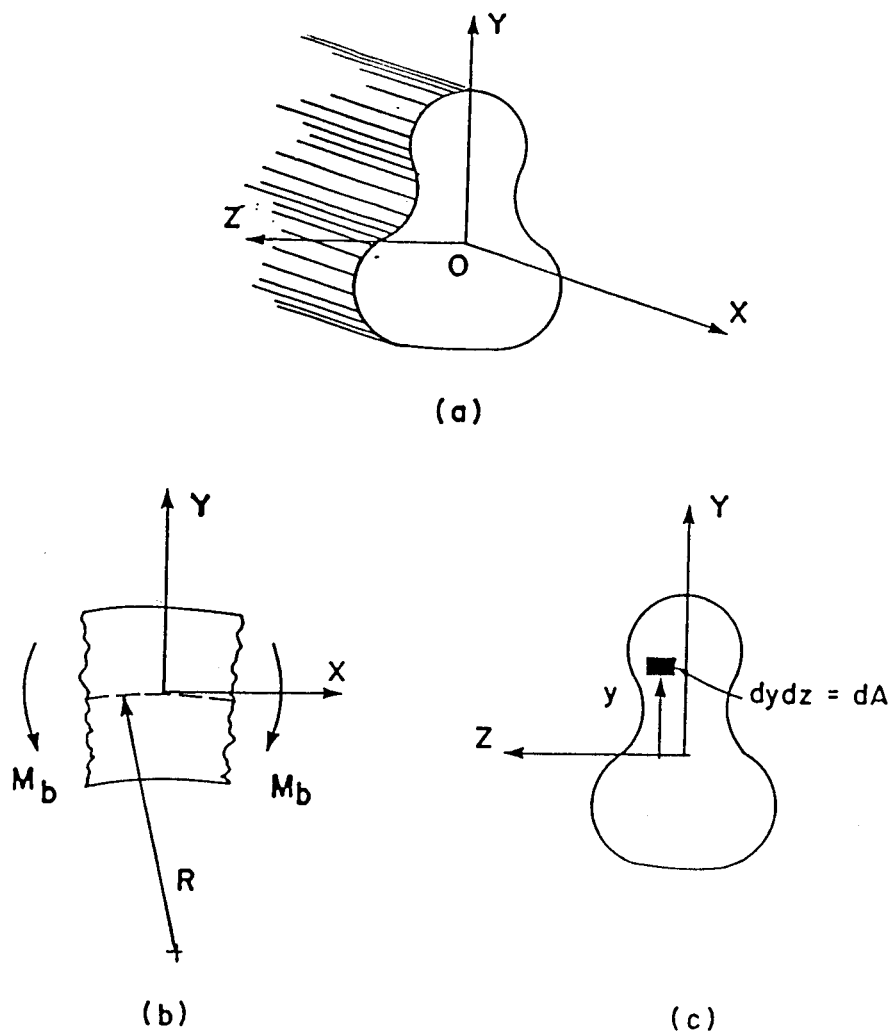


Figure 2. Illustration for Bending Behavior of Fibers

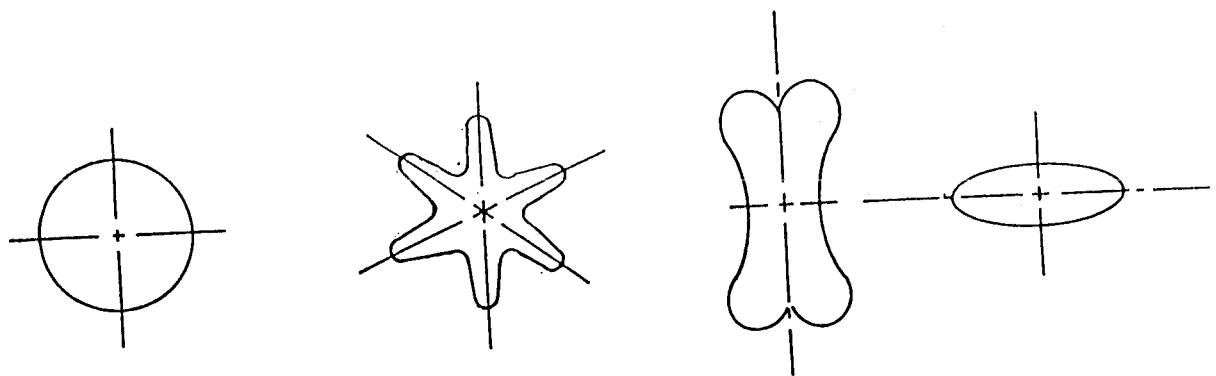


Figure 3. Typical and Hypothetical Fiber Cross-sectional Shapes With Two or More Planes of Symmetry.

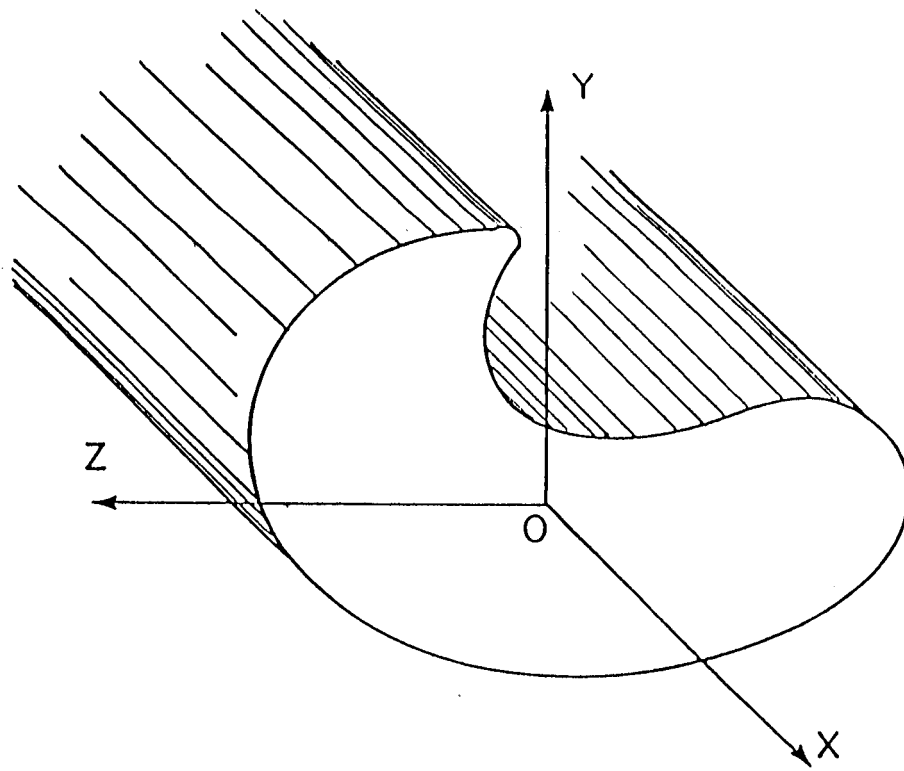


Figure 4. Fiber Cross Section With No Plane of Symmetry

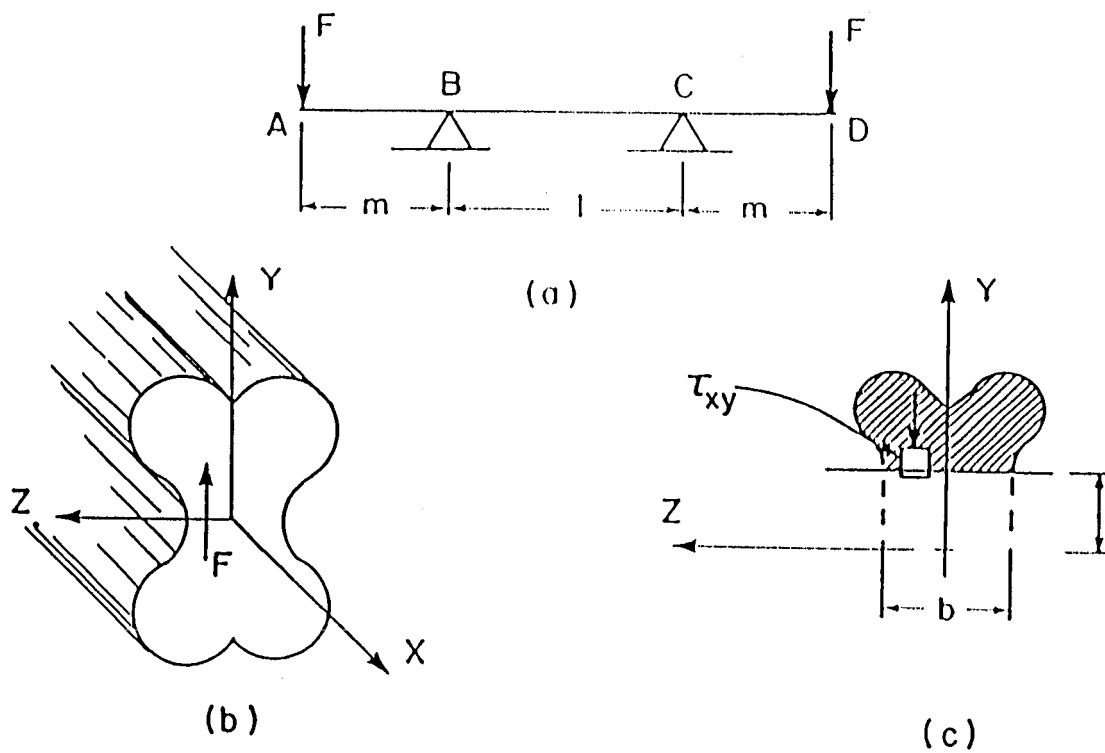


Figure 5. Fiber Bent by Laterally Applied Forces

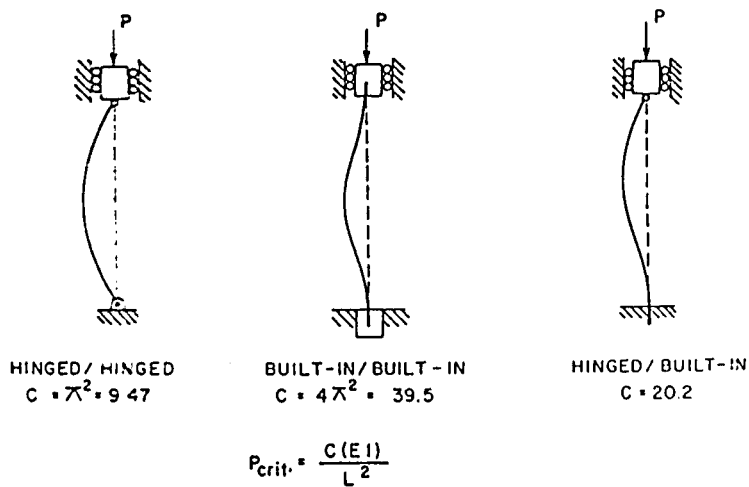


Figure 6. Buckling Behavior of Fibers

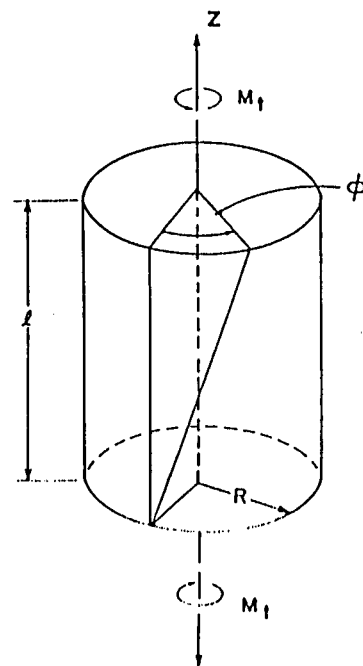
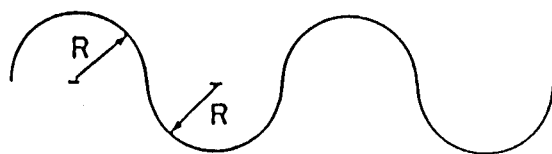
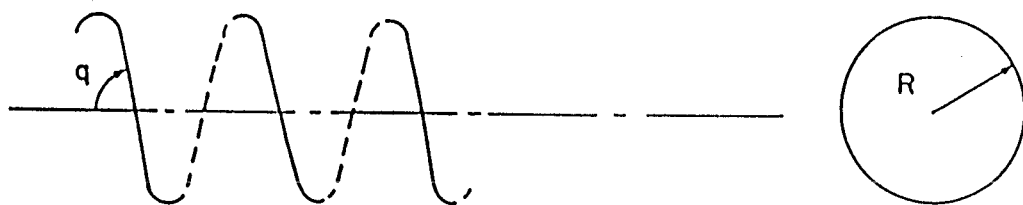


Figure 7. Torsional Deformation of a Circular Fiber



(a) Planar Crimp



(b) Helical Crimp

Figure 8. Geometry of Crimped Fibers

Control of Fibers in the Webstructure and Its Influence on Properties

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Introduction

We are going to make a review over Random Webforming Systems with special consideration of the Fehrer Nonwoven Plant type V 21/K 12 for the medium to the higher web-weight-range. A line which offers an optimum fibre distribution in the webs for needling or other bonding techniques. In comparison to conventional carding machines in conjunction with crossfolders you find at the random carding machine a fibre distribution with a strength ratio of nearly 1:1 (machine direction and the cross direction). At cards and crossfolders you find the strength distribution in the range of approximately 3 or 4:1 CD/MD. This is caused by the influence of the crossfolder.

By mechanical bonding the influence of the type of machine and type of needle as well as the needling density is studied.

Preneedling

The preneedling is the basic requirement to influence or to stabilize the strength distribution in a fibre web. On random webs as produced on the Fehrer nonwoven plant type V 21/K 12 it is essential to needle the web under very low draft conditions. This means that special preparations on the preneedler have been made to eliminate any air turbulences from the unbonded web during the preneedling process as well as special guiding of the unbonded web into the needling zone. Figure 1 shows the new Fehrer compound stripper in conjunction with the new stripper support. These two features are removing all air turbulences caused by the up and down movement of the needle-beam from the unbonded web. With this new feature it is possible to achieve draft ratios as low as 3% to 5% in preneedling depending on the fibre characteristics. A stabilized web, stabilized by preneedling is now further treated by additional needling from both sides to achieve enough strength for being used for example as a filter substrate or geotextile.

Uniform Web Formation

Now we return to the webforming system itself. The Fehrer nonwoven plant type V 21/K 12 has been proving its efficiency on the market for many years and only recently this line has been equipped with an additional, very important feature which opens up further marketing possibilities. This new feature is a continuous belt weighing station introduced between the chute feed and the feeding section of the V 21. This new continuous belt weighing station is also the base for a fully automatic plant control.

The data input is effected by a keyboard. On a monitor, the operator is guided by a menu to achieve the required web weights or line speeds. The continuous belt weighing station checks the incoming weight and regulates the feedling speed to achieve a constant fibre weight input into the width distribution system of the V

21/K12 which is shown in Figure 2.

The very simple calculation is showing the relationship between the web weight, belt speed and width of the belt weighing station in conjunction with the web weight, line speed as well as width of the finished product.

$$W1 \times V1 \times g1 = W2 \times V2 \times g2$$

For a given area weight 2, the operator just has to make the simple calculation.

$$V2 = \frac{W1 \times V1 \times g1}{W2 \times g2}$$

Web Strength

Another subject is showing the influence of the needling on the strength distribution in the finished product. For example one machine in quadropunch configuration which is a Fehrer model NL42 (Figure 3) needling the webs from both sides alternately. By this alternating needling the free transport time for the web is much lower compared to two doubleboard-machines which would be the Fehrer models NL 21 (Figure 4) and NL 21/R (Figure 5) one doubleboard-machine downstroke and one doubleboard-machine upstroke. The relationship is shown in Figure 6.

By the reduced free transport time of the web in the NL 42 the fibres are orientated in the machine-direction much more than one two machines type NL 21 and NL 21/R.

The result is, that the tensile strength distribution is quite different between a web which is needled on a NL 42 and the same basic web needled on NL 21 and NL 21/R. The tensile strength ratio will be approximately 2:1 (MD:CD) needled on NL 42 while the same basic product would end up with a ratio of about 1,5: 1 (MD:CD) needled on NL 21 and NL 21/R.

Geotextile Webs

Another subject related to the control of fibre orientation is shown in the diagram which was laid out for introducing a new method for the production of geotextiles (Figure 7). The line layout is showing the Fehrer nonwoven plant V 21/K 12. The NL 9/S preneedling machine downstrokes, the NL 21/R doubleboard upstroke and the NL 21 doubleboard downstroke. Following the fibre distribution or following the strength distribution you find that at the entry into the preneedle loom the random web has a strength distribution of nearly 1:1. During the needling process the strength distribution is changing caused by the draft in the needlelooms to approximately 1,4 or 1,5:1 (MD:CD). Our idea was now to bring the fibre introduced the stentering technique to reorientate the fibres in the needled web into the strength distribution which is again close to 1:1 (MD:CD). The stretch required is in the range of 30%. This means that from a 4m needling line a 5,2m finished product is achieved. By additional heat treatment of PP fibre webs the overall strength can be increased additionally.

Uniformity Testing

The next subject which is discussed during this short lecture is a very important new development which allows the continuous inspection of felts (Figure 8). Inspection of felts by checking their physical properties such as web weight, air permeability and thickness. This device, called the "felt quality control system," was developed mainly for paper felt producers, but it also could be of increasing importance for filter producers or nonwoven producers in general. The measuring equipment serves for continuous determination of the felt thickness, the air permeability and the mass per unit area. While felt measuring, the felt must be under tension otherwise the necessary contact between felt and measuring head will not be given. The adjustment of the system to several measuring ranges and the data processing is effected by a microprocessor. The measuring data are displayed on a screen and will be stored on discs.

A matrix printer provides hard copies of the measuring data, which can serve as quality certificate for the product. The system consists of the following parts (Figure 9):

- Linear guide system.
- Measuring carriage including measuring head. It is height-adjustable and driven by a DC-motor.
- Measuring head for the mass per unit area, this is a one sided measurement and the measuring head is equipped with a gamma source of americium 241 in the radiation collection tube. This is a low energy radiation source with a long half life period. The radiation collection tube is also equipped with an automatic safety cover.
- Measuring head no. 2 is equipped with the take up for the air permeability measurement. It operates by suction and air speed measurement. A radial fan with electronically controlled DC-motor creates the required air velocity and ultralow range pressure transducers and a speed sensing devices are delivering the required data.
- Measuring head no. 3 is responsible for measuring the felt thickness. It is equipped with an inductive proximity switch and holding magnets in the table of the carriage. These holding magnets are pulling a counter plate with the carriage over the felt.
- Control cabinet and interface computer including software and a personal computer are additional features to complete the system.

The following measuring ranges are available:

Technical Data:

A) Area weight

Measuring range: 300 - 4000 g/m² (8 - 118 oz/yd²)

Accuracy: +/- 1%

B) Air permeability

Measuring range: 300 - 3000 m³/hm² (16 - 160 cfm) ext. measuring range optional Accuracy: +/- 5%

Static pressures in steps of 1 Pa variable up to 200 Pa = 0,8" water gauge

C) Felt thickness

Measuring range: 0 - 6 mm (0 - 1/4 in.)

Accuracy: +/- 1%

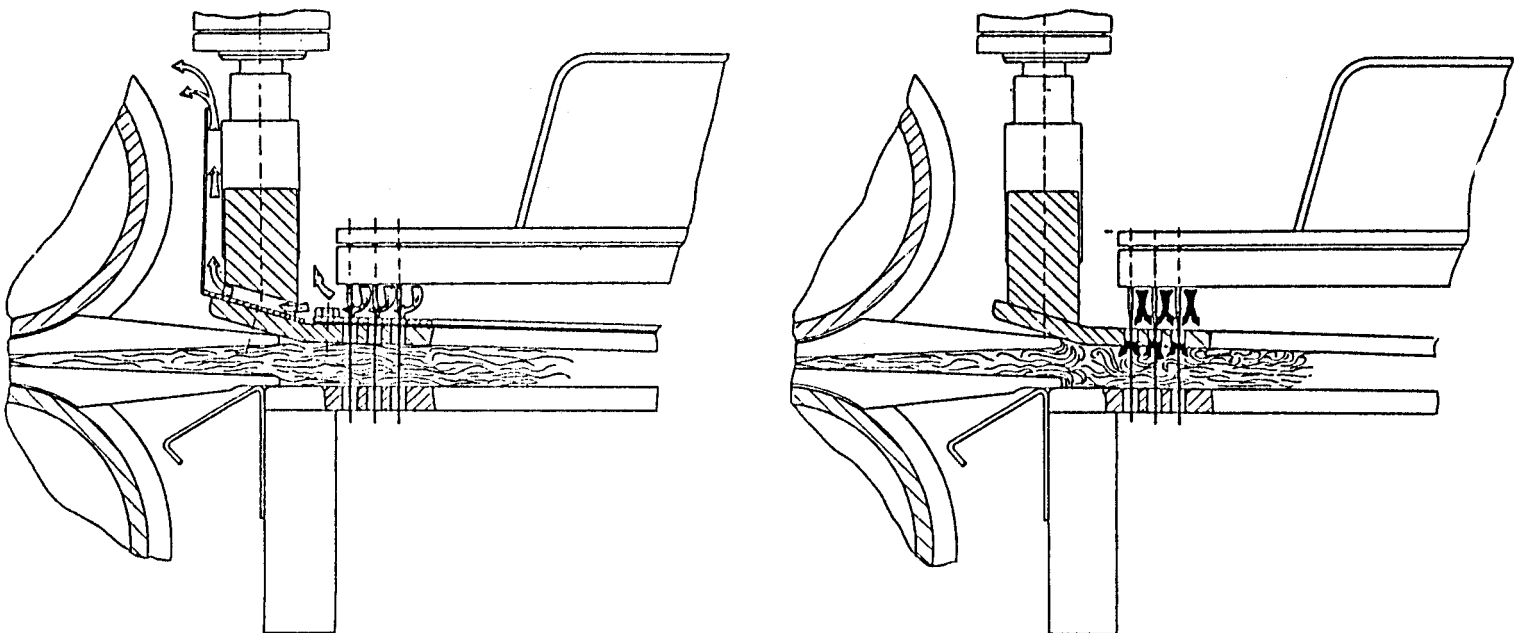
scanned surface: approx. 0,25 cm² (0, 04 in.²)

The Random Carding Machine

The most important subject in this paper is designated to the latest development of the Fehrer company, and the so-called "High capacity random carding machine K 21." This completely new development was first introduced to the public during the recent ITMA Textile Machinery Exhibition in Paris. First demonstrations have started in our laboratory in Linz/Austria on a 2 m wide machine. As shown in Figure 10 this high capacity random carding machine X 21 was designed to achieve equal tensile strength distribution in lightweight nonwovens. The web weight range is between approximately 10 and 100 g/m².

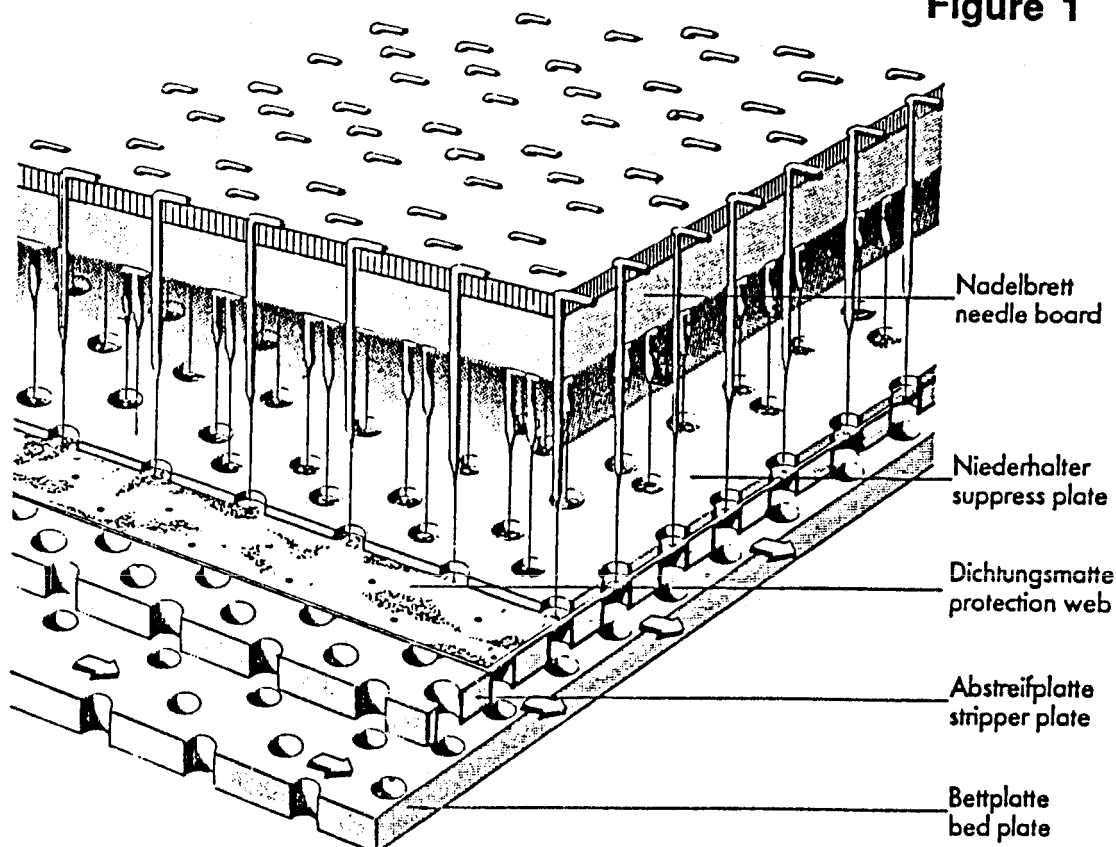
Many nonwoven producers in this low web weight range have been looking for such a unit for several years and can now offer a solution. The marketing of this new machine will help many producers to achieve better products. As shown in Figure 10 the K 21 has an arrangement of four fast rotating opening cylinders, each of them equipped with a pair of worker/stripper rolls. The K 21 is capable of transforming prewebs into random lightweight nonwovens. A very high web regularity is achieved by constructing the web out of four equal fibre layers.

It is also capable of dispersing parallel fibres into absolutely random fibre webs. The K 21 also can open up carded and crosslapped webs from a weight of approximately 200 g/m² to produce for example a 20 g/m² web consisting of 4 equal layers 5 g/m² each. The speed range of the K 21 is up to 150 m/min. The capacity is up to 300 kg/hour/m based on 1,5 den/1,5" and 3 den/2" as shown in Figure 11. More details of this new machine will be published as they are created in our laboratory in Linz/Austria. Trial possibilities are given on the nonwoven plant type V 21/K 12 for the medium to higher web weight range as well as on the new K 21.



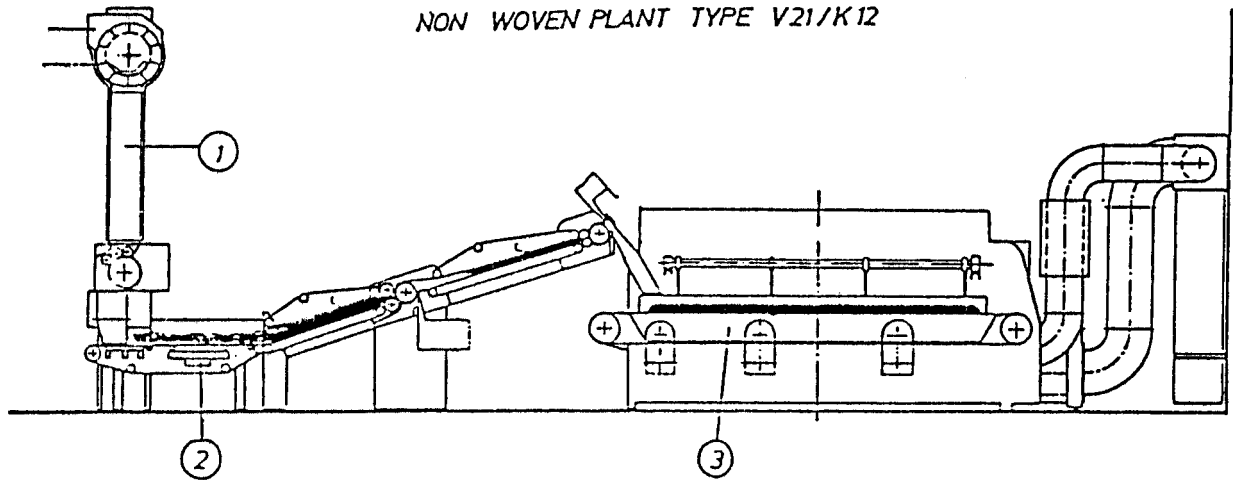
COMPOUND STRIPPER

Figure 1



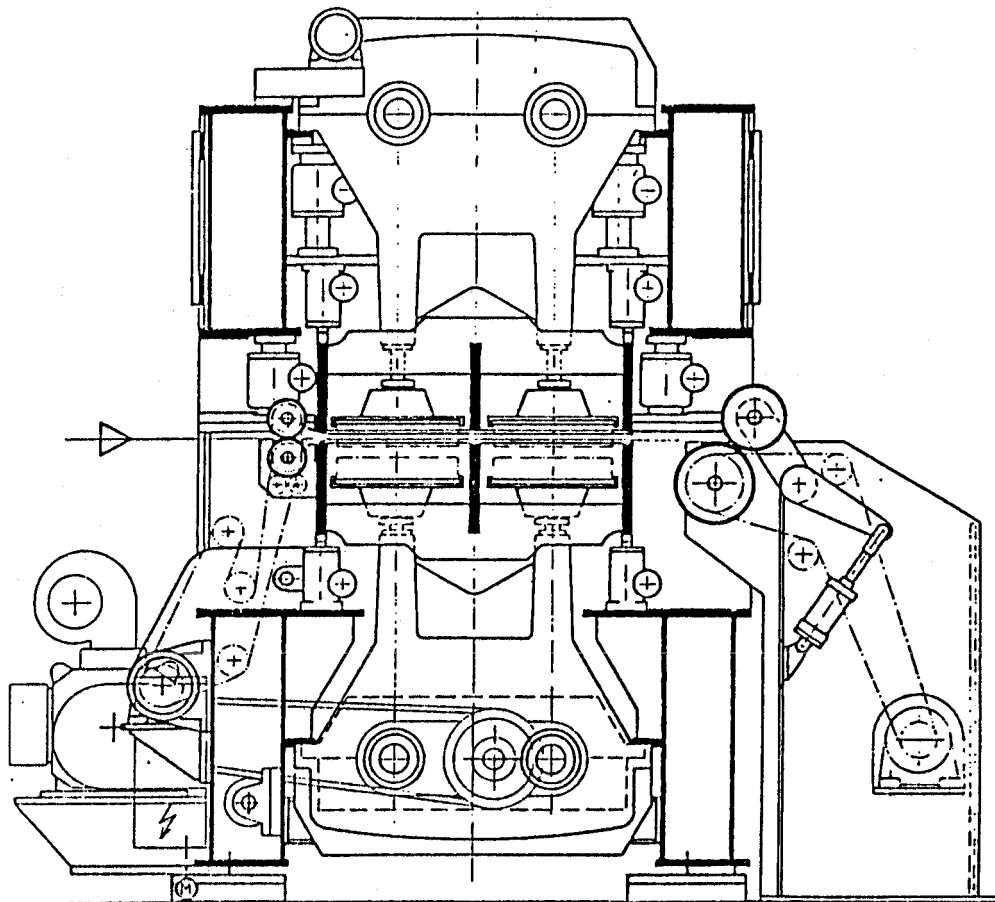
Abstreiferdichtung · Compound-Stripper

FEEDING SECTION OF THE FEHRER
NON WOVEN PLANT TYPE V21/K12



- ① CHUTE FEED
- ② CONTINUOUS BELT WEIGHING STATION
- ③ WIDTH DISTRIBUTION UP TO 5,4 m (212")

Figure 2



NL 42

Figure 3

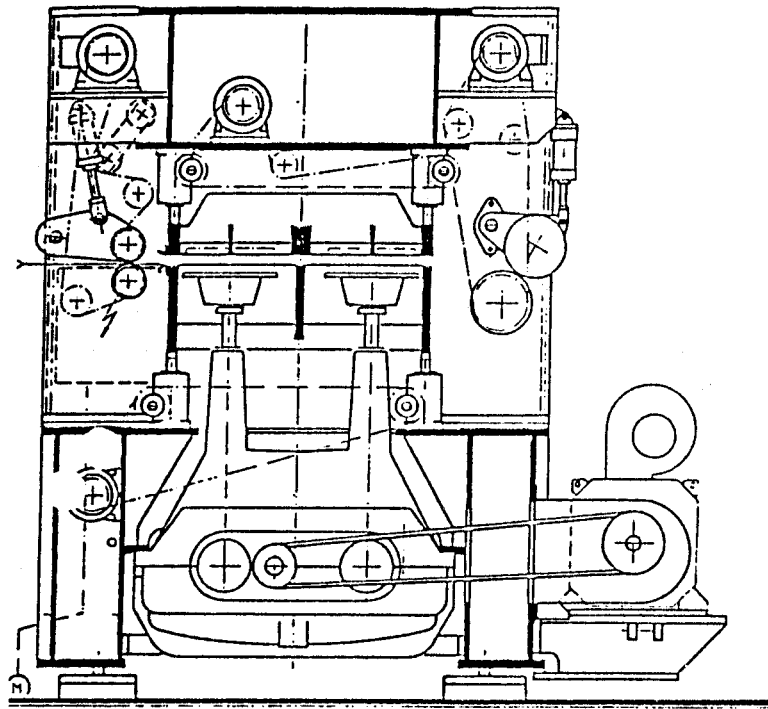


Figure 4

NL 21 R

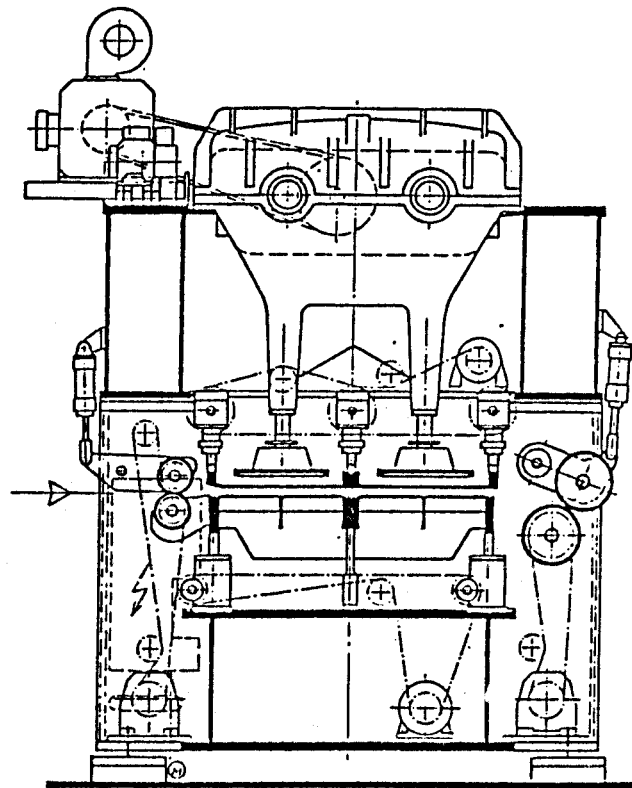
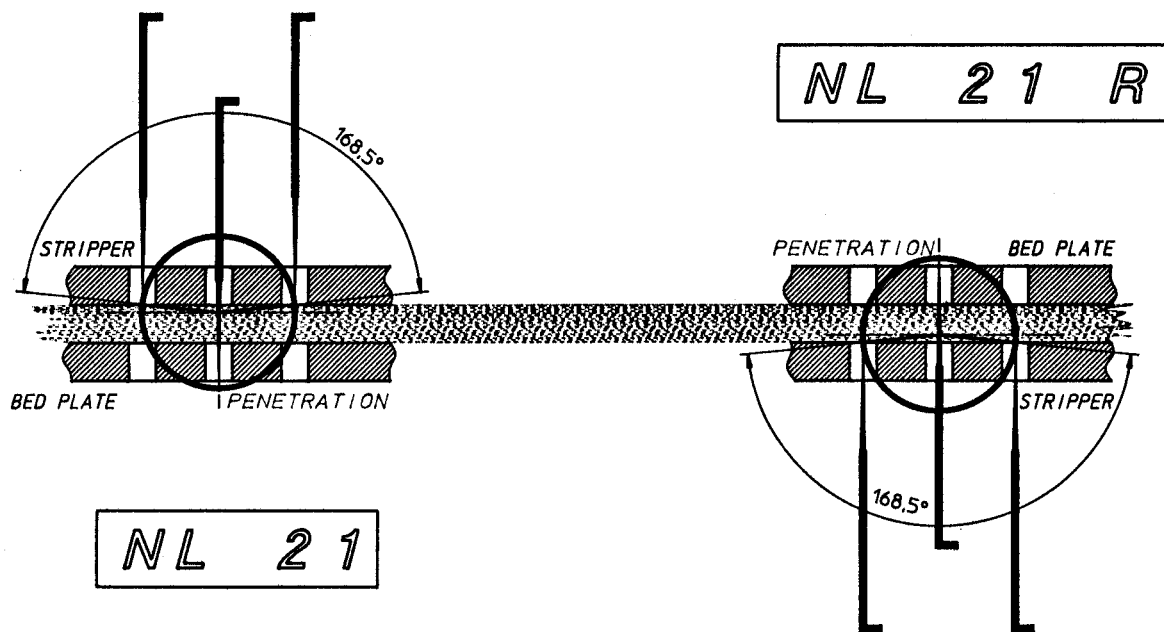


Figure 5

NL 21



Comparison of free transport time between NL 21 / NL 21 R and NL 42

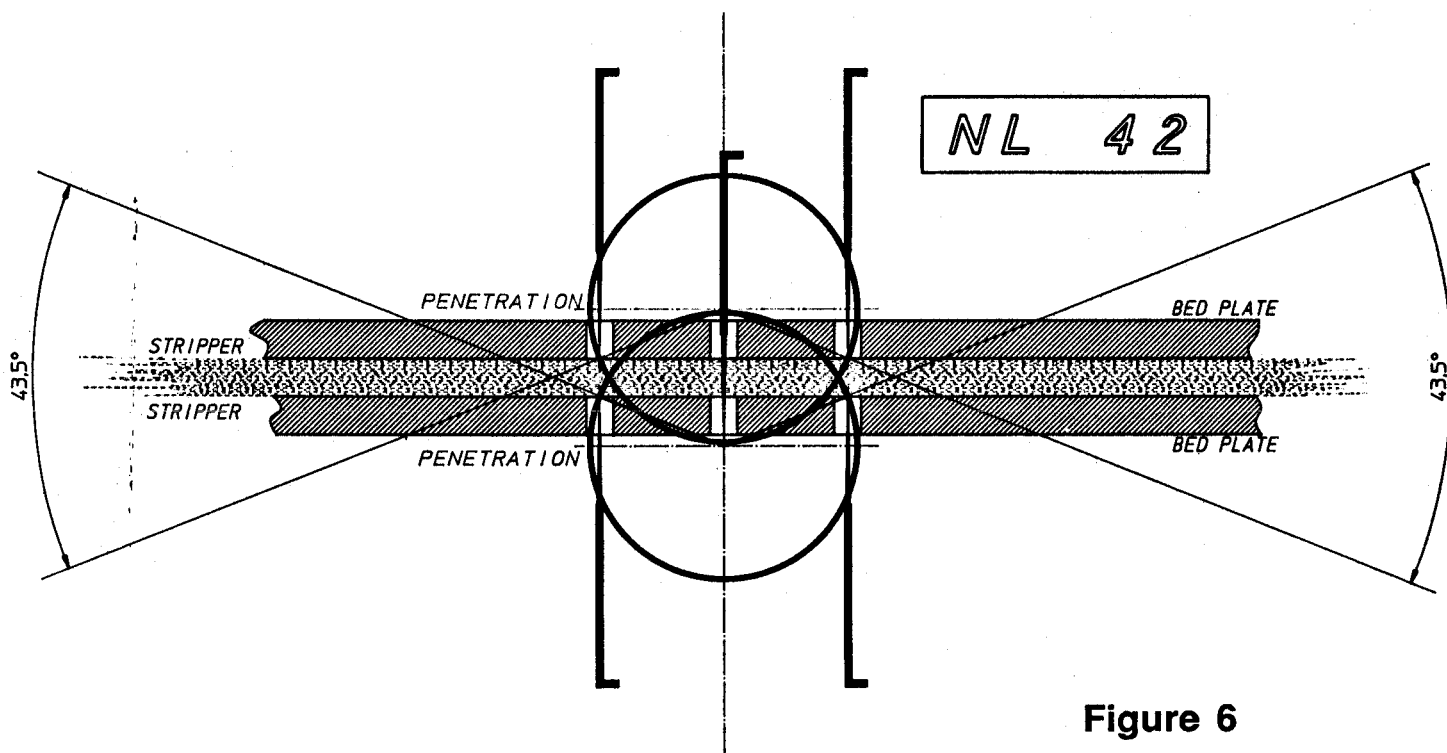


Figure 6

UTILIZATION OF THE RANDOM WEBS CHARACTERISTICS

A NEW WEB TECHNOLOGY FOR THE PRODUCTION OF GEOTEXTILES

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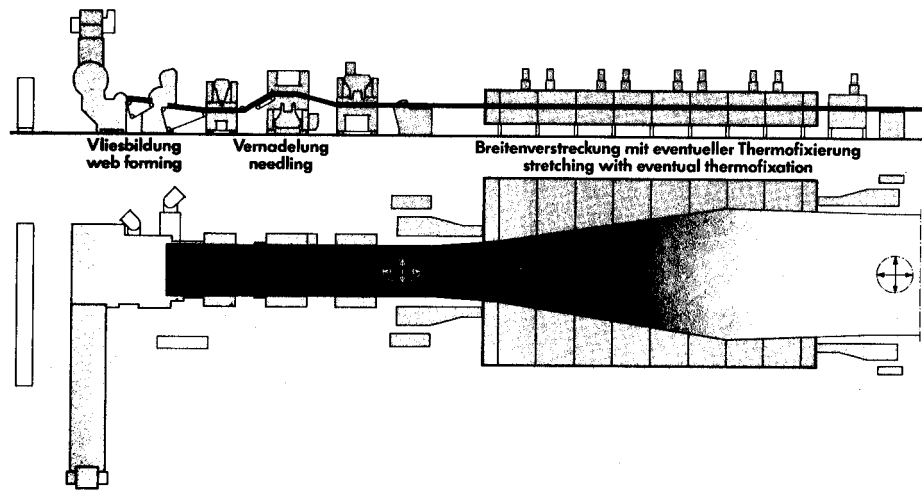


Figure 7

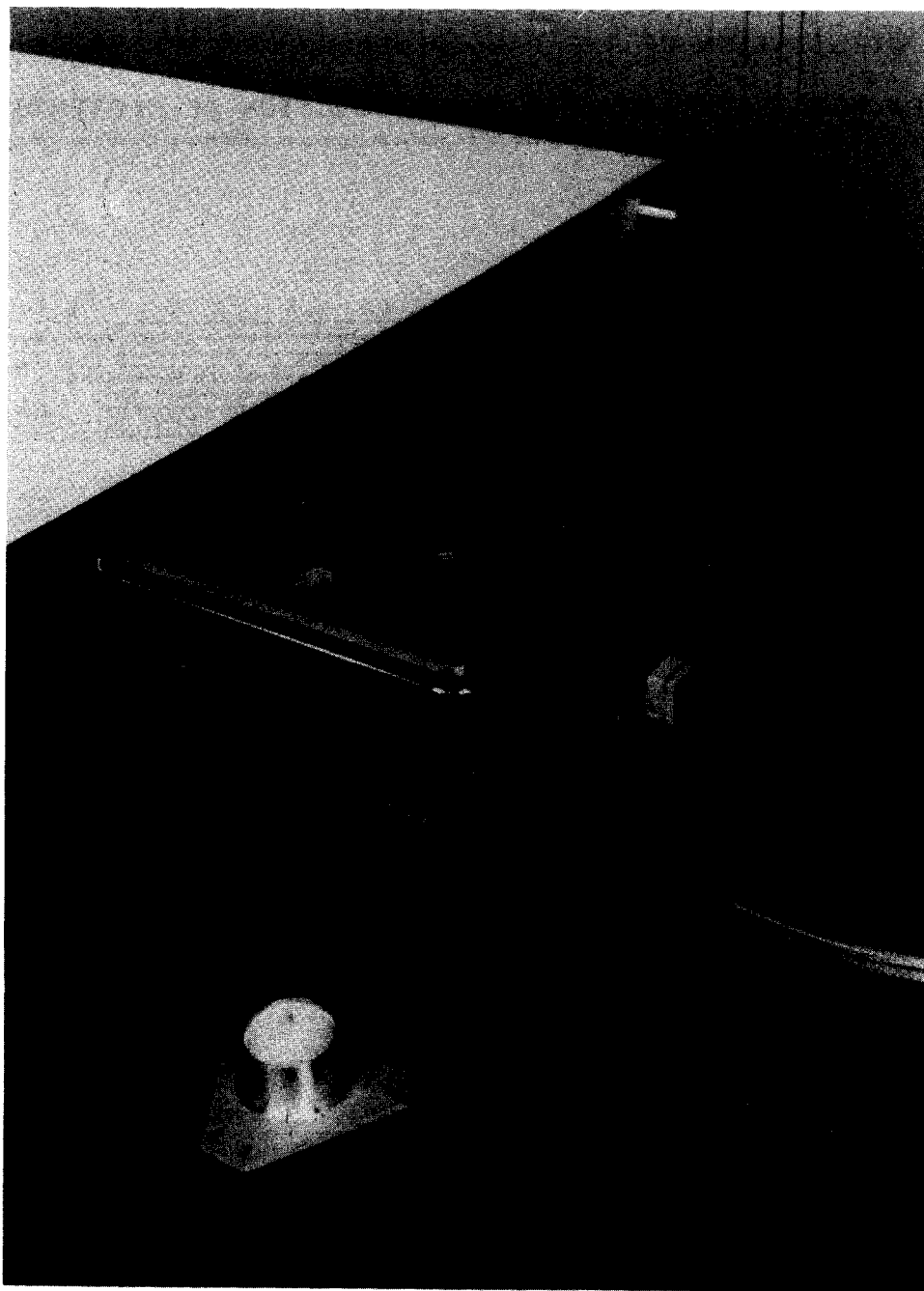


Figure 8

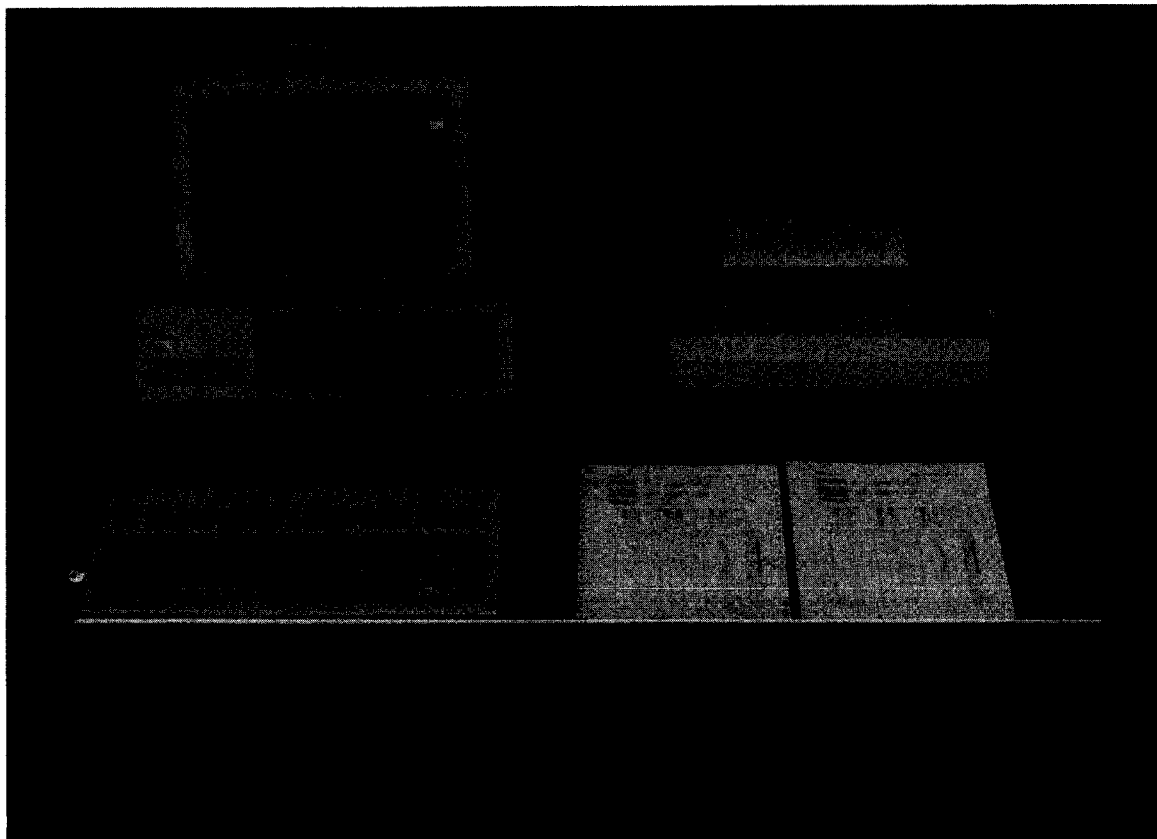
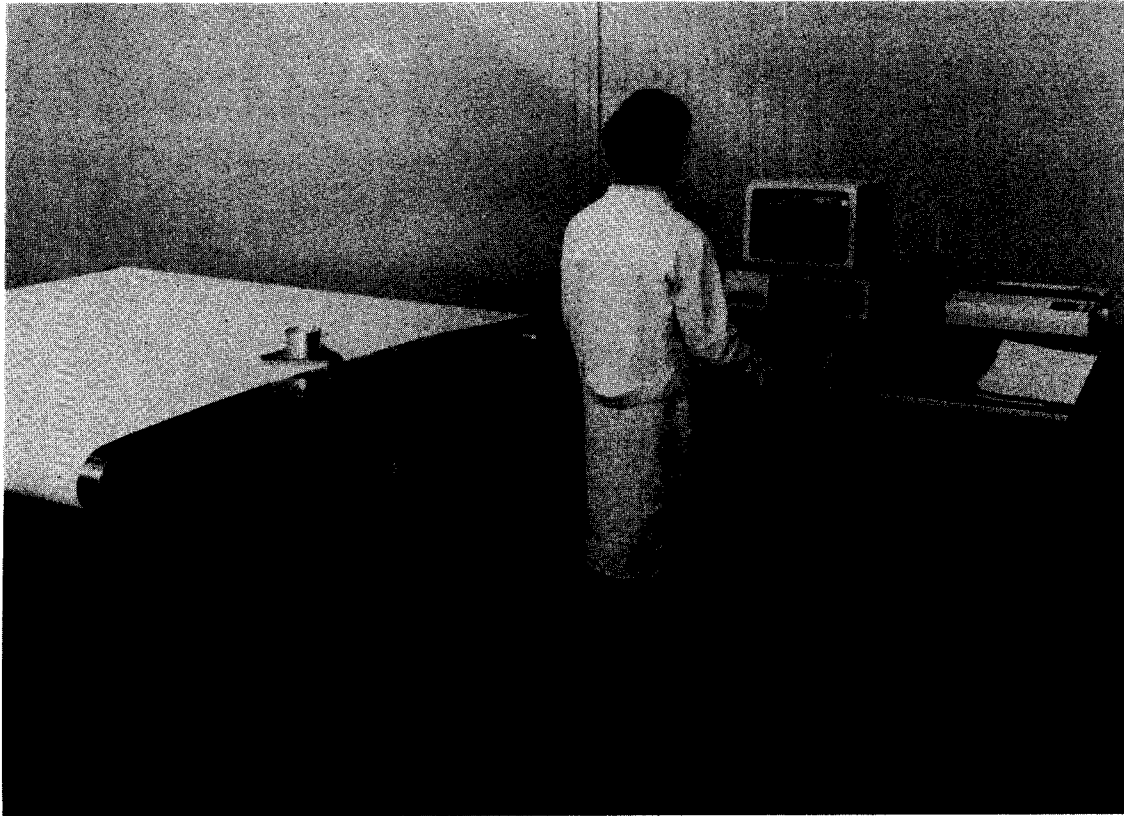


Figure 9
Control of Fibers in the Webstructure and Its Influence on Properties/33

High-performance random carding machine "K 21"

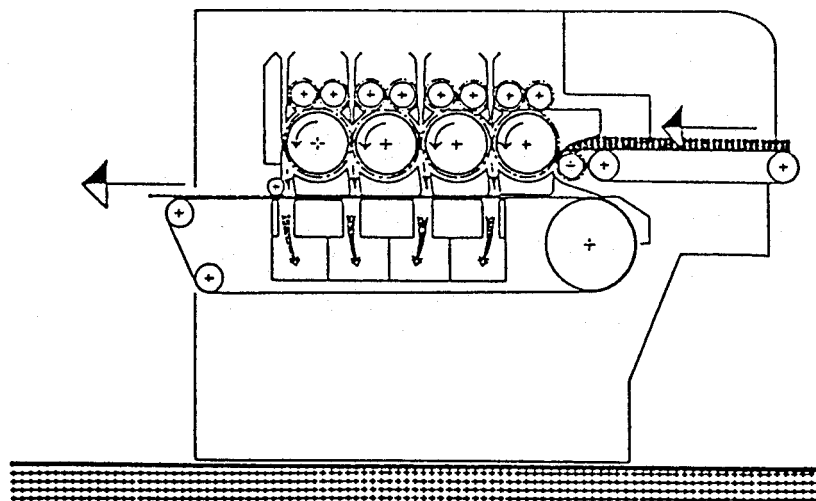
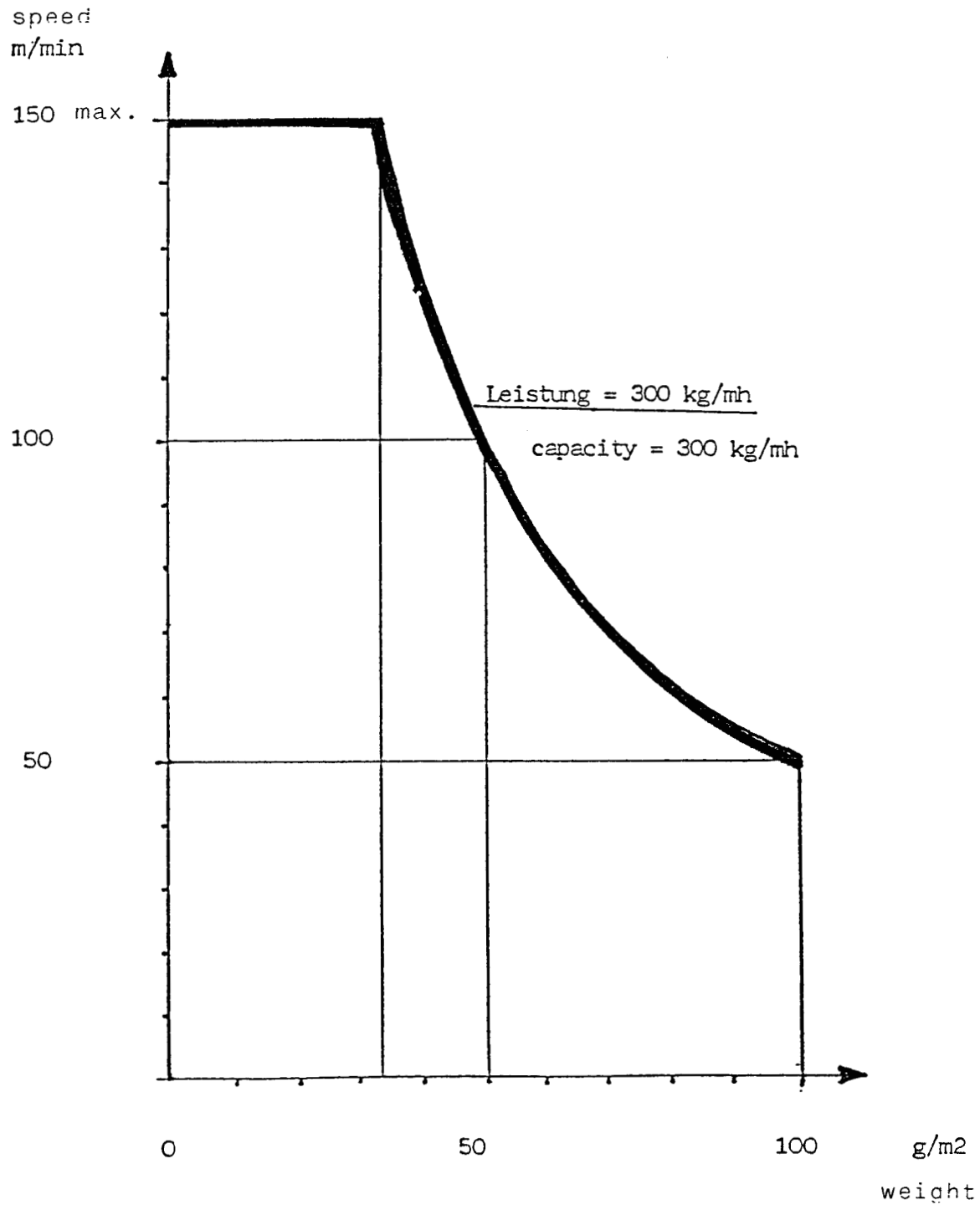


Figure 10

Possible production speeds of the K 21 at a maximal capacity of 300 kg per hour and meter in dependence of the weight to be produced



Fasermaterial: PES, Viskose 1,5 - 3 den

Fibre material: PES, viscose 1,5 - 3 den

Figure 11

FRONTIERS OF NEEDLE PUNCHING

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ABSTRACT

Within the advances of non-wovens in general and needle felting in particular, developments of products, processes, technologies and fibres have often been evolutionary and, in many cases, revolutionary. To wit: production width versus surface structuring, respectively. Mechanical factors such as stroke frequency, needle density, advance, penetration depth, etc. are interrelated and approach limits beyond which process economics and fiber properties cannot follow. The paper deals with the limitations of presently known processes, technologies, raw materials and economics and the need for development of more specific means to create a wider product and economic spectrum and reach beyond present established goals and standards.

INTRODUCTION

We have heard papers dealing with topics associated to needle punching, but adhering to the concept of a tutorial, I may be permitted to delineate some specific aspects of the original concept of this paper as sketched out in the abstract.

Some developments of a mechanical nature, such as equipment design, might not be for general publication at this time, or this audience. Some developments in process technologies, however, are. Let me, therefore, strike a compromise between some basics, which should really be known by most of you, and the very specifics of proprietary nature known only to us.

DISCUSSION

To start, let us look at a short list, which is intended to provide a perspective of needle punching or needle felting, as it is also referred to. (FIGURE 1)

As you can see, the definitions are rather confusing. Nonwovens could be knitted products, tufted products, stitched products, etc., but none of them are considered nonwovens. The more accurate description for needle punching should be fiber-woven; however, this is a registered trade mark and could not be used. We are dealing with an interweaving and intanglement of fibers rather than yarns or filaments, which would make that term much more descriptive. The other aspect of defining nonwovens are in their methods of preparation. We know of the wet-laid as opposed to dry-laid methods and will ignore the wet-laid method here, as it belongs in a different category, and concentrate instead on dry-laid. This has been explained previously and is now being defined in regard to the method of bonding these fiber webs together. The bonding technique can be thermal, ultrasonic, saturated, spray, spun and, last but not least, mechanical.

MECHANICAL BONDING - Within the mechanical bonding category there are only two groups: stitch bonding and the rather domineering process of needle felting or needle punching. As we concentrate on needle felting in this paper, we subdivide those products into two groups again, namely, flat felts and structured products. Flat felts exist in a variety of industrial products, geotextiles, automotives, interlinings, home furnishings, but not all products can be part of these categories. In the structural processes there is a narrower product mix, namely, automotives, floor and wall coverings, as well as home furnishings and a small segment reserved for special applications. Hopefully, this provides some additional perspective and we can more specifically address the differences between flat felt and structured product technologies.

As we examine differences between flat felts versus structured felts, it is obvious that the preparatory process technologies required for either are different and are based on the intended end product. Traditionally, the evolution of these processes and the machinery and equipment necessary was a result of efforts between the universities, the manufacturing industry, the machine manufacturer and the fiber producers.

Unfortunately, in the area of needle punching, the machinery manufacturer and the manufacturing industries are almost the only activists in advanced know-how, vision, development and market demands. If this seems to be critical of fiber producers' efforts in this field and the academic community, then so be it. But facts are that the overwhelming majority of new inventions, developments, equipment and processes have been attributable to the efforts of the machine designers and the drive of the product manufacturers. For example, the start of a conventional textile career of a young man was to attend a university and learn how to design a fabric or how to manufacture a bed sheet. None of these avenues are open to anyone in nonwovens other than from the background of a textile manufacturer who transferred into nonwoven manufacturing and/or nonwoven equipment design at the almost total exclusion of significant contributions by the fiber producers and/or the academic community.

We have, therefore, a rather closed loop of manufacturing techniques and machinery designs, with the intended purpose to provide process means for specific end products. Figure 2 shows basic flat felt needling principles. A web is being fed between a feed apron and roll pair, which in reality is of more sophisticated design, but the principle is clear. The web is introduced to two plates - the upper one is the stripper plate, the bottom one is the bed plate. The fiber is entangled and interwoven by barbed needles in an interchange between horizontal movement of the web and vertical action of the needles forming a needled and consolidated substrate emerging from the needle zone by action of the delivery rolls. Some variations of this theme can be seen in figure 3, a downstroke and upstroke action, either in the same machinery concept, or in an alternation machine process. The up and downstroke, or simultaneous needling, is shown in the figures 4 and 5 in its alternate and the principle is clear.

In addition to this basic needling technique, the element of controlled drafting as illustrated in figure 6 is being used to provide additional economic means for the manufacture of light-weight materials, but in the more modern forms, controlled drafting is a separate process between preneedling and finish needling, in order to also redistribute the strength of a product. The conventional process produces a strength bias in the cross machine direction, stemming from the fiber orientation off the card as it is cross-lapped onto a floor apron. In order to randomize this strength in both directions, a controlled draft, as illustrated, is being employed and the results are shown in the next illustration, figure 7, with an almost equal strengths distribution in machine direction.

This principle will be explored a little further later, but let it suffice to say that flat felts with high quality standards need that strengths distribution. If the end product is structured, fiber orientation in one direction is to be maintained.

The next illustration, figure 8, shows the basic flat felt needle schematic and figure 9 illustrates the basic configuration of a fork needle, then shown in figure 10 in operation. Figure 11 illustrates the principle behind structuring. The preneedled material introduced between a perforated stripper plate and a lamella table is being "structured" through the action of the fork needle through the lamella spaces and its withdrawal leaves a fiber rib or tuft or velour on the underside of the table, which constitutes the face of the product. This form of structuring in velour, pile and riv surface effects in various designs is being accomplished on the DI-LOOP machine, figure 13, widely operated throughout the industry and used for plain structures and sophisticated surface designs. This variety stems from the use of multi-colored fiber webs as well as the interrelationship between needle action, the needleboard pattern, the needle density and the contrast between flat and structured surface area.

A further development of basic structuring is the design of a machine model solely intended for the manufacture of velour and velour-like products.

The DI-LOUR principle is shown in figure 12 where some of the design elements are the same, like the needle beam, the needle boards, the needles and the stripper plate. But, unlike the DI-LOOP principle, the preneedled substrate is being carried tensionless by a moveable brush conveyor substituting for the stationary bed plate. The action of crown needles into this preneedled web will form a totally random, pliable, drapable velour surface into the brush conveyor. The schematics of the model DI-LOUR II reveals two beams, two boards and the brush conveyor and the delivery rolls, which withdraw the velour material, figure 14.

In the interest of perspective, the next illustration, figure 15, shows the wide array of equipment models and needling variations available to the non-woven needle felt manufacturer and its variety in needling direction, needle densities, speeds and working width. Quite clearly, the working width requirement of the manufacturing industry has been an evolutionary process dictated primarily by the demands of product design and market criteria. On the other hand, the equipment principles, such as structuring through the invention of the DI-LOFT process of years ago and its further refinements of the DI-LOOP and DI-LOUR variations, are process related and inventions of revolutionary magnitude within this segment of the industry. These principles were adapted by other machinery makers, duplicated and simulated, and we are now in the company of a small number of different machinery manufacturers marketing the same design principle, thus indicating some maturity within this equipment category. It was the creation of these design principles which encouraged the manufacturer who created the product, which developed the market because of the uniqueness with which these products could be adapted to its demands, for instance, geotextiles and automotive end products, just to mention a very few. These revolutionary aspects of equipment design and process development influenced the evolutionary efforts within the area of mechanical equipment design, providing the tooling for a whole industry primarily engaged in producing needle punched fabrics or needle felts in their various forms and product categories mentioned earlier. It is outside the realm of this audience to mention needle board design, needle board installation, and the ability to remove and reinsert needle boards into looms at a rapid interval.

Other mechanical aspects are the design of high speed machines achieving, with the use of various stroke heights, speeds of up to 2200 to 2400 RPM, much outside of what even the most optimistic designers of 50 years ago thought possible. This was in addition to the demands made of the machine designer in regard to dealing with excessive heat build-up, and its removal, during the high speed needling process the safety aspects, noise abatement and mechanical and electrical maturity of the support systems. All these factors have to be fitted into the operational controls of the equipment demanded by the process, such as depth of penetration adjustments, drafts, material thickness and the various process adjustments. It requires steps to provide maximum controls, economics and processability, demanded by continuous product developments.

PROCESS VARIATIONS - After some of these, by necessity, sketchy accounts of basic principles and mechanical requirements in needle punching, let's concentrate a little on the processes mentioned and in which ways they vary from flat to structured and within geotextiles, automotives and/or upholstery end uses for instance. On page 17, as an example, we find the presently accepted state of the art configuration of a geotextile or industrial product installation. These installations and their width requirements range from 3.5 to 6 meters with the majority of all installations producing at 5.5 meters width for a trimmed end product. In the illustration, figure 16, the fiber web, coming from preparatory processes such as opening, blending, carding and cross-lapping, is being introduced by the compressive batt feed, type CBF, insuring tensionless and draft-free controlled introduction of the fiber into the needle zone of the preneedling stage. After approximately 100 to 120 ppi, the material is introduced to a four-stage draw frame, type VE-4, designed to reorient the fibers at their cross-over points and to randomize the strength distribution, which up to here was in the cross machine direction. The finish needle pass is being accomplished by a double needle loom with four boards, two striking from above and two from below, needling both sides of the substrate and providing final dimensionally stable, as well as physical qualitative properties the material was designed for.

Geotextile uses are wide spread, but the mentioned process is not limited only to geotextiles. A large and growing number of manufacturers of industrial felts and industrial composites, filters and a variety of specialty end uses exist but a detailed discussion of them would out-strip the scope of this paper.

In summation, let it suffice to say that wherever flat, finished products with dimensional stability and strength and evenness characteristics are mandated, compressive batt feed, preneedling, controlled draw and double finish needling are the required and accepted stages in various width and different production parameters.

The next illustration, Fig.17, shows the typical stages for structural products. Coming from the card and cross-lapper, the material is being fed into the CBF unit of a preneedling machine and into the two stage down-stroke passes of needle looms, which could be combined into one machine. For better dimensional stabilization characteristics, however, they are preferred to be run as shown, namely in two separate stages, in order to assure that the preneedled material intended for structuring is of optimum quality. Separate from this line configuration is the actual structuring machine - in this case, the DI-LOOP model. This concept requires the winding of the preneedled material after preneedling and unwinding again for passage through the structural DI-LOOP, and again winding of the end product. Very few manufacturers chose to operate an in-line installation, wherein the necessity of winding and unwinding between preneedling and structuring is omitted. The greatest majority of manufacturers chose off-line structuring,

enabling them to utilize the base installation for other than structured materials and products as well. In the case where the structuring is limited to the basic rib, pile and some velour products, the equipment can be operated without the hydraulic design attachment. But, in cases of sophisticated floor and wall cover products, the design feature of this process must be utilized. The equipment with the hydraulic design features enables the manufacturer and product designer to create an endless variety of patterns, which are unique and cannot be duplicated or simulated through any other process.

In the more specific case of velours, the DI-LOOP machine, shown separately in illustration number 13, can be substituted with the DI-LOUR machine, shown in illustration number 14, which enables the manufacturer to create a light weight velour product, ranging anywhere from 100 to 600 g/m² with superior drapability, random pile, moldability, strength and abrasion resistance. If the proper fibers have been selected for the end product, both process categories, the first one used for virtually all industrial products, composites and geotextiles, and the latter for all the automobiles, floor and wall cover products, are now contributing to the formation of yet a third and in its implication probably the most significant advance in the industry. It is being used in the manufacture of upholstery fabrics and, thus, provides a cross-over into the sphere of conventional textile fabrics. The frontiers of needle punching are indeed challenges to the role of conventional products and processes, such as weaving, tufting and knitting.

Figures 18, 19, and 20 of the following illustration show the principles of the various methods of needle punched upholstery products. The web structure, strength and elastic properties are of great importance to the upholstery product, as the fabric has to absorb the plain forces in all directions. It is stretched, and compressed, and has to withstand high abrasion and friction. During use, the fabric, as all fabrics, should elongate elastically to avoid wrinkling and resist dynamic loads. In order to fulfill this function, special emphasis has to be laid upon the fiber type, blend and formation of a proper web structure. The DI-LOUR upholstery fabric may be made of spun bonded material or staple fiber. A spun bonded usually has a rather high degree of uniformity and a random fiber orientation. Carded and cross-laid batts naturally have a cross-oriented fiber structure. Since forces during use of the fabric are multi-directional, within the substrate fiber orientation should also be multi-directional or random. This may be achieved by two different approaches. One, by using two layers of fiber batts with cross-directional fiber orientation, the first layer to be delivered by a double doffer card and a second layer on top of it as a cross laid batt. The now right angular structure will be more or less randomized by needling.

The second way is for the fiber of the preneedled cross laid materials to be reoriented through a VE drafting unit as described earlier in the processes of geotextiles, in order to gain equal strength in all directions. The principle of drawing is simple and shown in illustration 6. In running through the draw unit, the web is wrapped successively around the surfaces of the upper and lower rolls in a series of passes and the frictional forces created by the wrap are controlling the motion of the fibers, which gradually pivots around their cross-over points received through preneedling. A four stage draw, usually in a ratio of 1:2 is not only randomizing the strength, but also reducing the web weight. This controlled draw creates the randomization of the fiber orientation and results in rather equal distribution of strengths in all directions, which is a prerequisite for any fabric for upholstery and other related uses. In figure 18 of the illustration, the process of the lowest cost of investment would consist of carding, cross-lapping, preneedling, drawing and the DI-LOUR structuring model II/S, with a maximum throughput of around 8 to 10 yards per minute. Comparing that with conventional speeds, it becomes clear that the potential for this process is phenomenal.

The described process using only one card is designed to manufacture only unicolor products. After preneedling and drawing, the DI-LOUR machine is fed with a substrate of totally random strength characteristics. The DI-LOUR machine not only creates a pile structure, but also increases the strengths of the material and end product through the vertical entanglement of the fibers, something which cannot be done to this degree with conventional tufted, woven or knitted products. They are weakening the surface and increasing the danger of pilling. A two layer or two color product is illustrated in figure 19. As mentioned earlier, one can also create a totally random web structure by using two different cards and separate needling for each. In the example in figure 19, the first card, with a double doffer, is used for the longitudinally oriented base layer in weights of up to 3 oz/yd², while the second card, with a cross-lapper, is intended for the cross-laid batt to be laid upon the base layer. Both webs have to be needled separately in order to prevent color blinding; hence, the double acting needle loom prior to introducing the laminated web to the DI-LOUR machine for structuring. In the case of a multi-color process, the DI-LOUR crown needle transports the fiber pile through the base layer fabric. Therefore, the color of the top layer shows up in the pile surface of the bottom layer with striking results. One can vary the appearance of the product by omitting some rows of needles in the DI-LOUR machine and the difference now becomes visible as small stripes, simulating cords or stripes or interesting patterns for not only upholstery material, but for wall coverings as well.

FINISHING - Thermal bonded DI-LOUR materials have a high degree of abrasional resistance, strength and elasticity. For this end result a portion of low melt fibers have to be blended as a component to the overall blend. After DI-LOUR structuring, the material is thermally bonded in a separate dryer passage. A tenter frame will soften the low melt fiber and a vertical flow of hot air through the material will bind the total fiber mass together. It is quite obvious that this method does not require any evaporation of liquids or waters as in a chemical bonding process, but rather glues the fibers together at a relatively low energy consumption. After the thermal bonding process, a separate shearing passage is required in order to cut the pile surfaces in those areas where fiber loops are apparent in order to give the end fabric good cleaning and vacuum properties. In appearance, a sheared DI-LOUR product is almost impossible to distinguish from a woven velvet of conventional manufacturing means.

These have only been a few small examples into the development work for producing limited quantities of upholstery products. The development work is not concluded and a great deal of additional work remains to be done. Weight ranges in the DI-LOUR furniture fabrics are between 200 and 350 g/m², or about the 4.5 to 9.5 oz/yd² range. Early conversion and sewing tests have proven very satisfactory and so have a series of other wear tests in various areas in the industry. We are very encouraged and enthusiastic about the present state of progress and will be only too happy to assist others in their own product development efforts.

SUMMARY

In summary, one can conclude that frontiers in needle punching are continuous. During the civil war the frontiers in needle punching centered around the manufacture of horse blankets for the army and in later years a gradual infiltration of needle punched fabrics into the area of conventional woven products occurred. Small examples are interlinings, which are now almost exclusively nonwoven, and automobiles which approach an almost 80% level of total nonwoven products in the average passenger car. Carpets and wall covers are greatly substituted by nonwoven products. Conventional

geotextiles would not be economically viable and have been discussed. Nonwovens in many other areas, too numerous to mention here, are replacing conventional structures or at least supplementing woven, tufted and knitted products. The frontiers in nonwovens at least are continuously moving.

DILLO-Needle Looms
Table of models

FIGURE 15

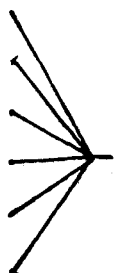
Type type	Nadelbretter needle boards	Nadelanzahl/1m number of needles/1m	Arbeitsbreite (m) working width (m)	Hub (mm) stroke (mm)	max. Hubfrequenz/min max. strokes/min
OD-II*	2 = 	4000-15000	1,5-15	40/50/60	1200
UD-II	2 = 	4000-15000	2,5- 6	40/50/60	1200
OD-II S	2 = 	4000-15000	2,5- 6	25/30/40/50/60	2200/1500
UD-II S	2 = 	4000-15000	2,5- 6	25/30/40/50/60	2200/1500
OUG-I	2 = 	4000-15000	2,5- 6	60	1200
OUG-I C	2 = 	4000-15000	1,5	70/100	700/500
OUG-II*	4 = 	8000-30000	2,5- 8	60	1200
OUG-II S	4 = 	8000-30000	2,5- 6	60	1500
DI-LOOP OD-SV*	1 = 	3000- 7200	2,5- 5	40	1200
DI-LOOP OD-SD*	1 = 	3000- 7200	2,5- 5	40	1200
DI-LOOP VS	1 = 	3000- 9500	2,5 / 3,5 / 4,5	40	1700
DI-LOOP DS	1 = 	3000- 9500	2,5 / 3,5 / 4,5	40	1700
DI-LOUR II*	2 = 	8000	2,5 / 3,5 / 4,5	40	1200
DI-LOUR II S	2 = 	8000-15000	2,5 / 3,5 / 4,5	40	1800
RONTEX 50	2 = 	3000	25-170 mm ø	40	1500
RONTEX 75	2 = 	3560	40-500 mm ø	60	800
ORC 8	1 = 	3000	0,8	60	750
OD-OR	1 = 	1600-4000	2,5 / 3,5 / 4 / 5 / 6,5	70	1000
BELTEX	4 = 	8000-20000	bis/up to 15	60	1200
PMF 2-0-2-0	4 = 	12000	bis/up to 15	60	800
PMF 2-0-2-2	6 = 	18000	bis/up to 15	60	800

FIGURE 1

NON - WOVEN PRODUCTS

DRY - LAID
(Web-Forming)

THERMAL
ULTRA-SONIC
SPRAY
SPUN
SATURATED
MECHANICAL



BONDING

MECHANICAL - BONDING

WET LAID
(Web-Forming)



STITCH-BONDING



NEEDLE FELTING
(Punching)

FLAT FELTS
INDUSTRIAL
GEOTEXTILE
AUTOMOTIVE
HOME FURNISHINGS
INTERLININGS
DISPOSABLES
COMPOSITES
PAPER MAKERS FELT
MARINE



STRUCTURED FELTS

AUTOMOTIVES
FLOOR COVERINGS
HOME FURNISHINGS
UPHOLSTERY/FURNITURE
INDUSTRIALS

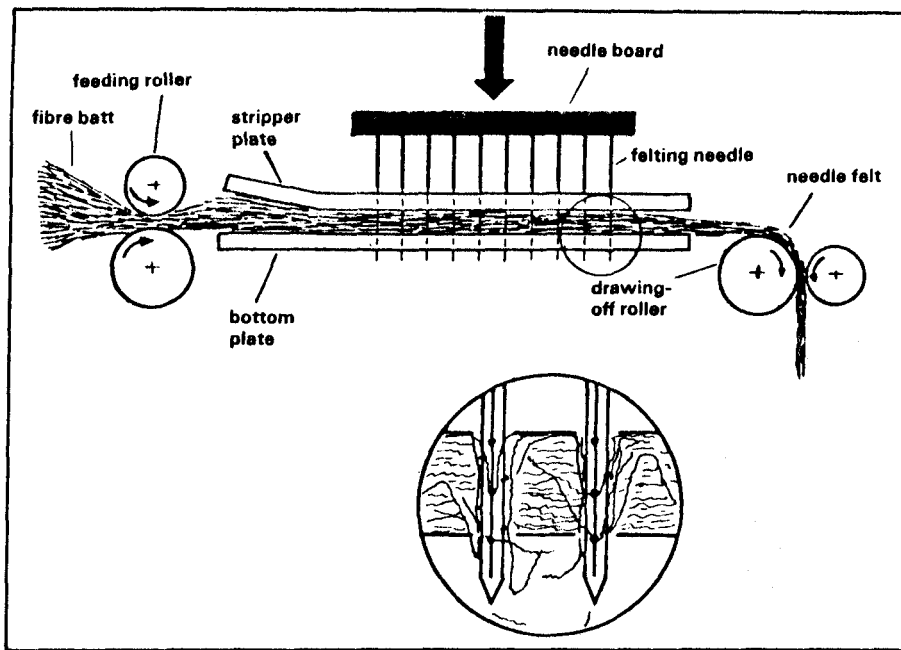


Figure 2: basic principles of needle felting technology.

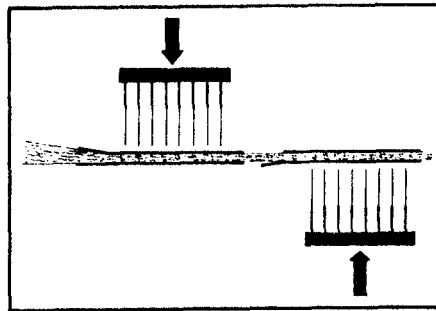


Figure 3: double-sided needling, needle board arrangement shifted.

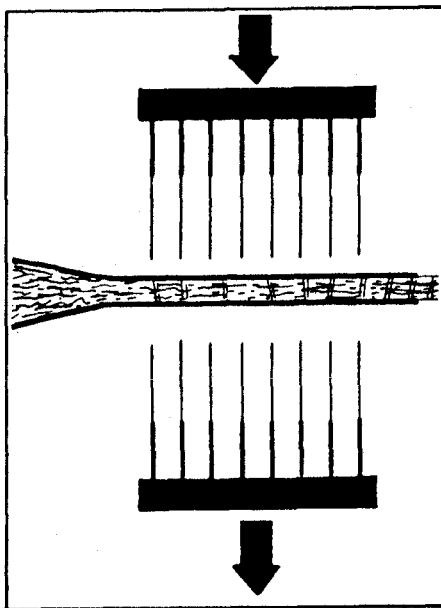


Figure 4: double-sided needling, needle board arrangement, with simultaneous needling sequence.

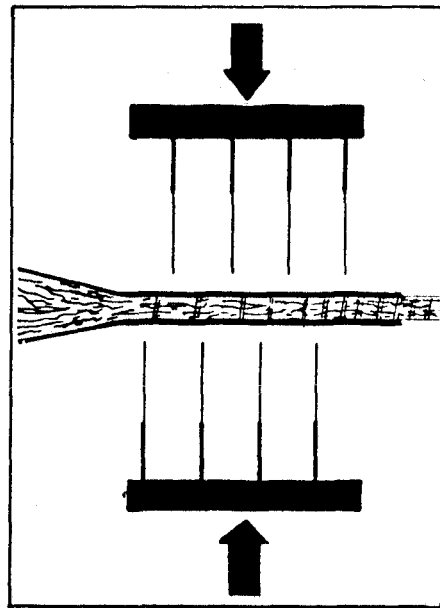


Figure 5: double-sided needling, needle board arrangement opposite each other, alternating needling sequence.

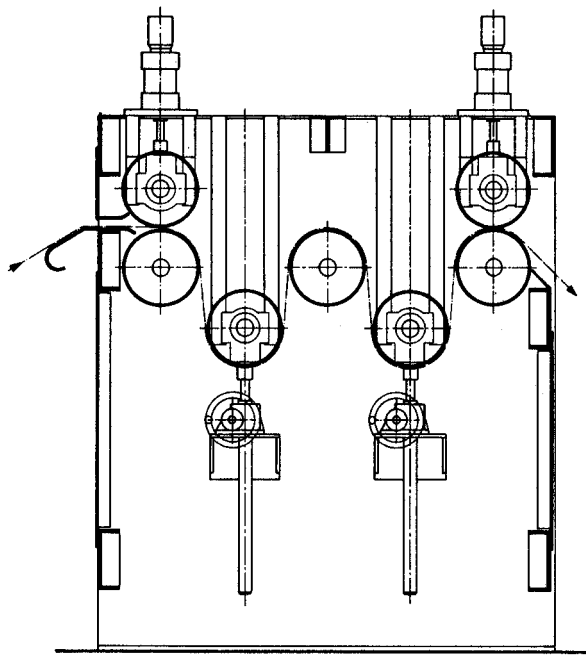


FIGURE 6

DILO-Drafting Machine type VE

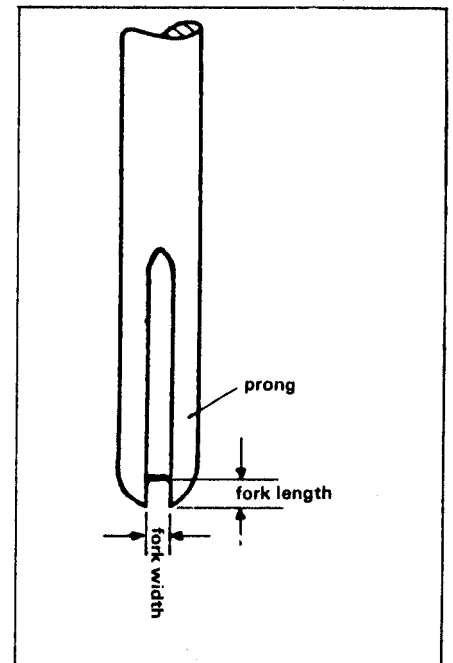


Figure 9 : constructing the fork of a fork needle.

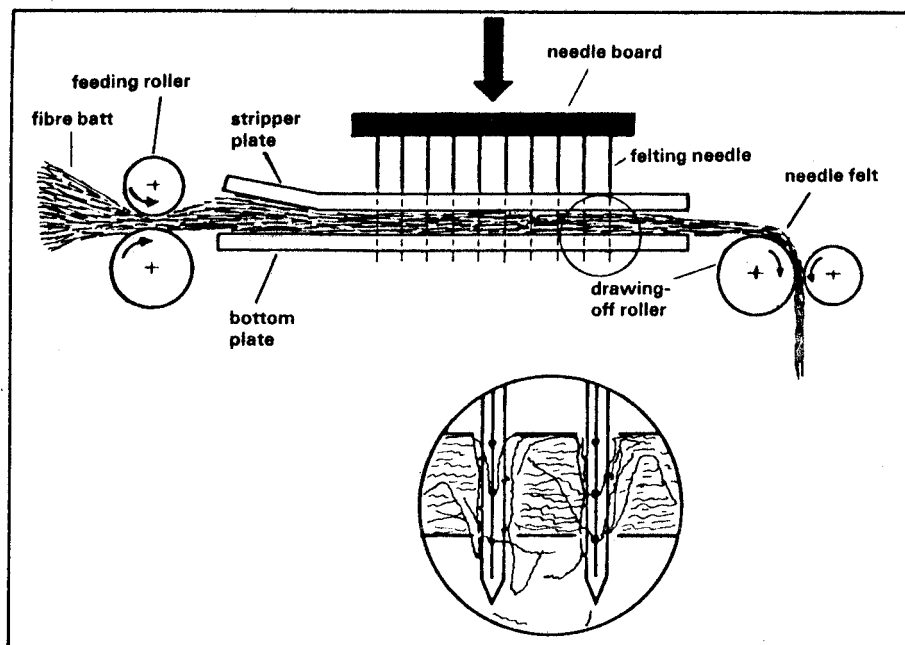


Figure 8: basic principles of needle felting technology.

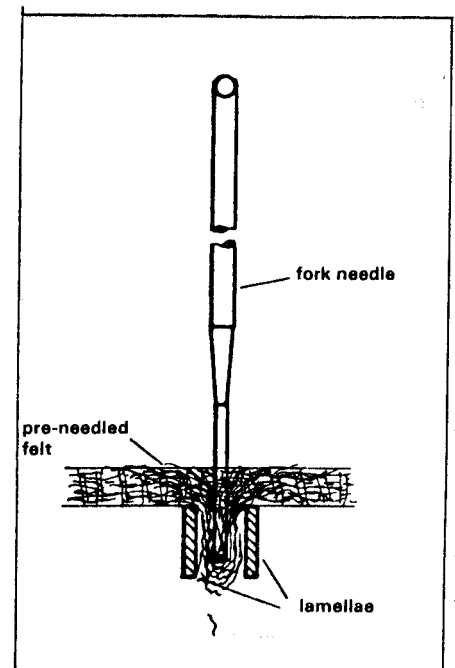
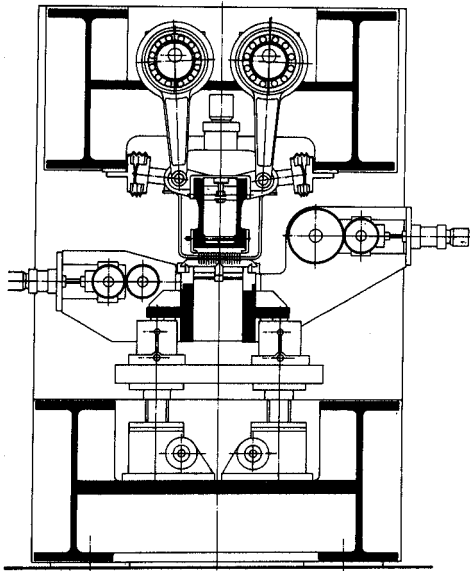
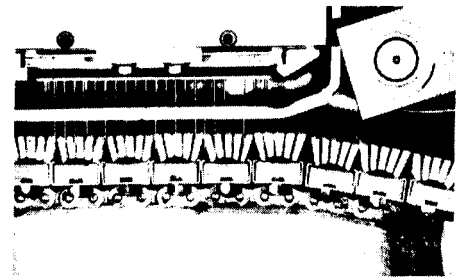


Figure 10: building a miniature loop during needling with a fork needle.

FIGURE 13



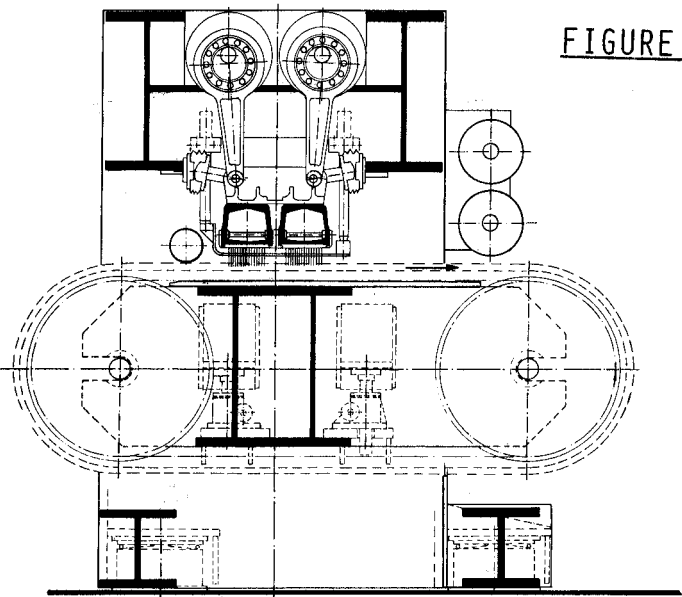
DILO - Needle Loom type DI-LOOP OD-SV
DILO - Needle Loom type DI-LOOP OD-SD



Partial view of brush belt

Figure 12

FIGURE 14



DILO-Needle Loom type DI-LOUR II

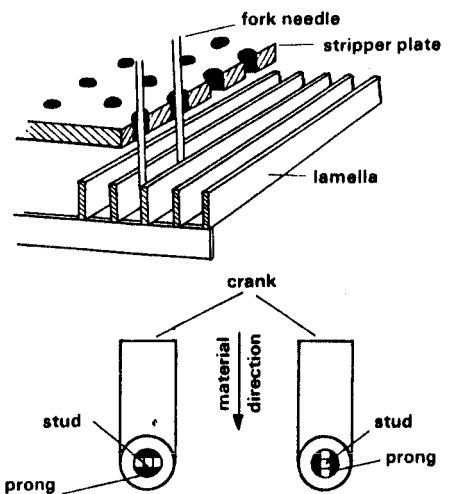


Figure 11.: design of the grid table consisting of lamellae arranged parallel to each other.
Figure 5B: position of the fork for producing ribbed or velours-like effects.

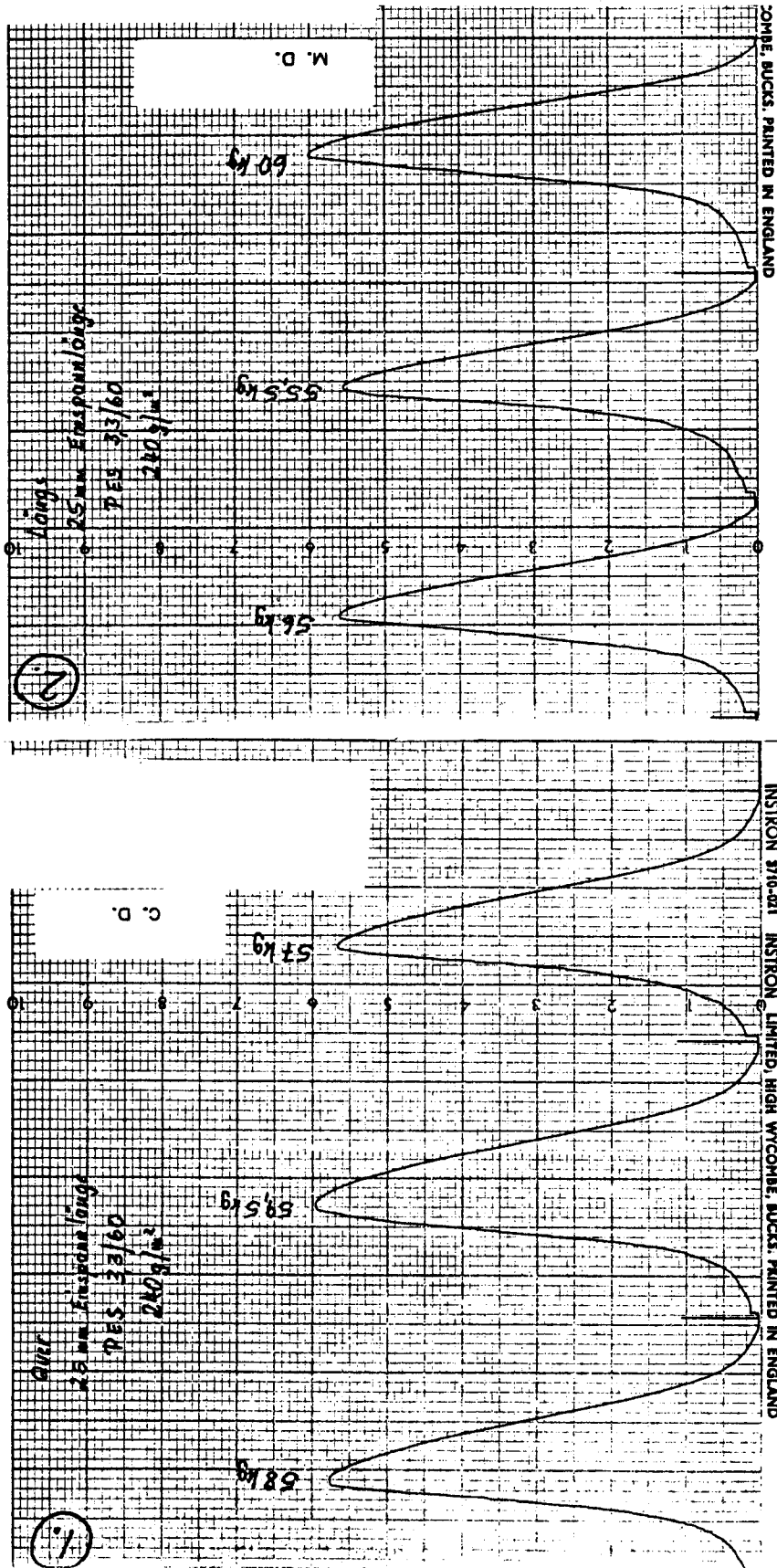
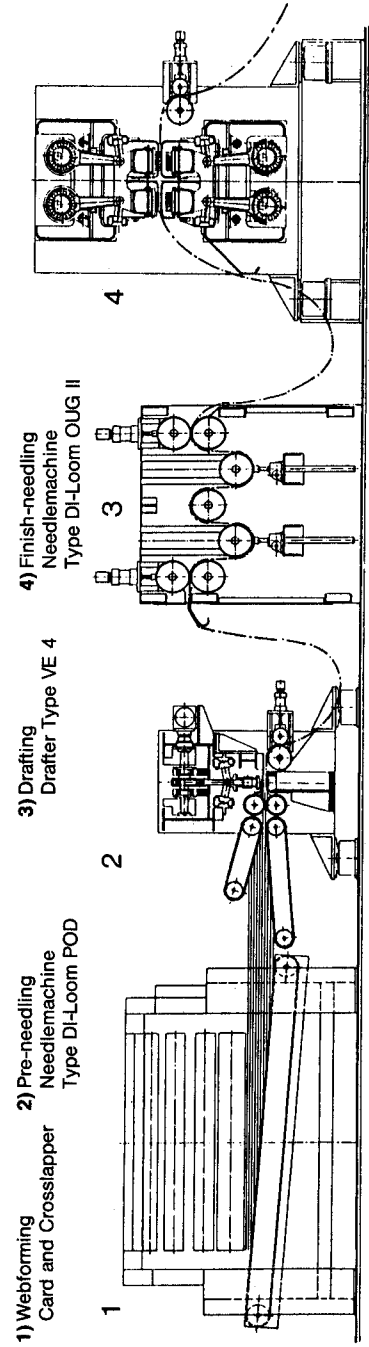


FIGURE 7
Strength distribution of nonwoven material in cross and length direction after stretch pass through DILO VE-4 Machine.



Nonwoven plant for Geotextiles

FIGURE 16

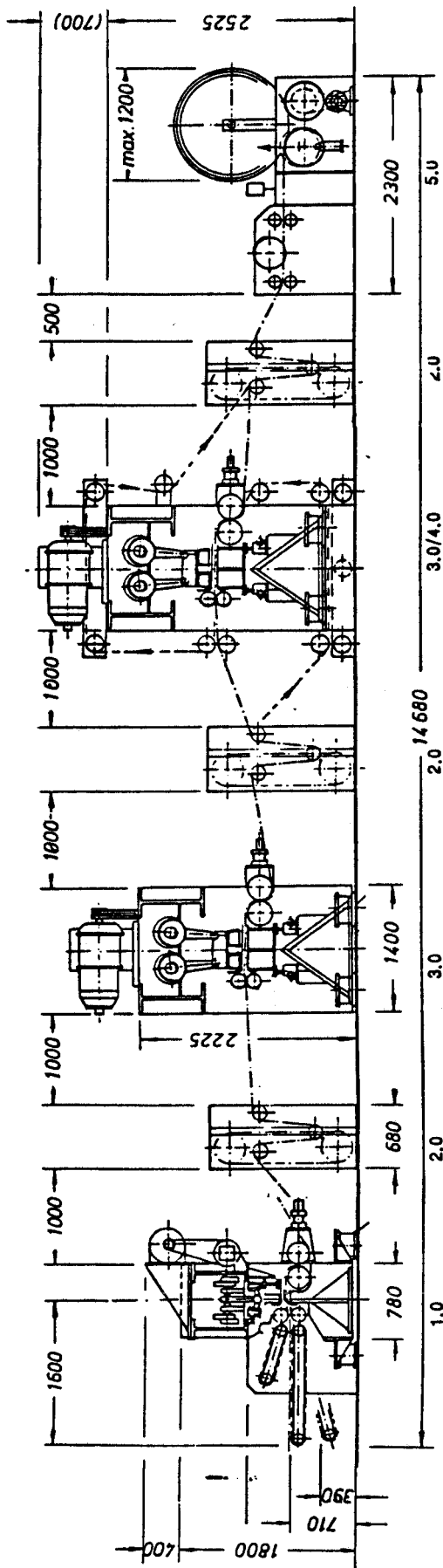
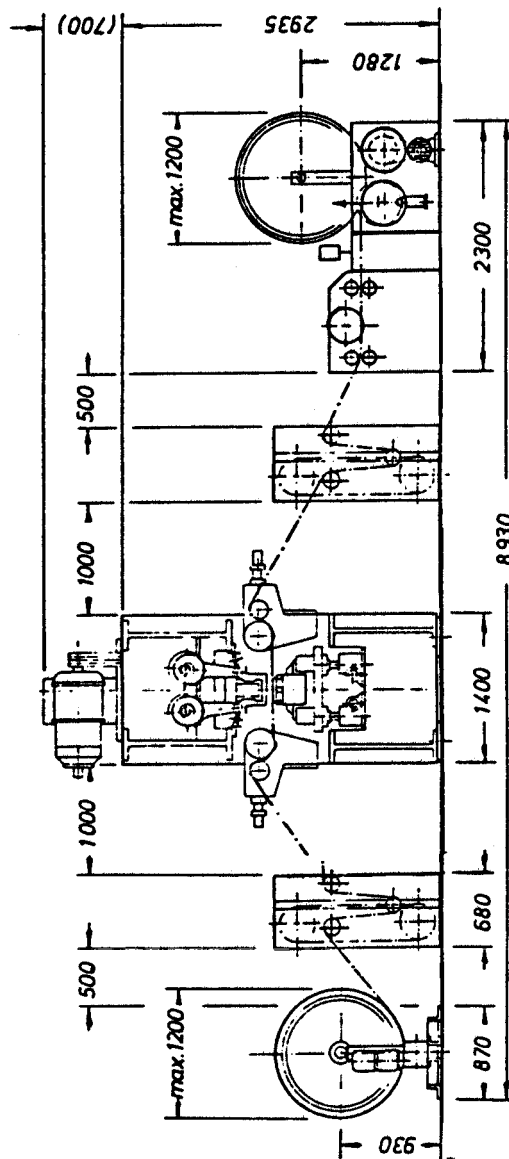


FIGURE 17

TYPICAL STAGES
FOR STRUCTURAL PRODUCTS.



- 6.0 - Abwickler Typ AWZ
- 6.0 - Unwinder type AWZ
- 7.0 - Durchhangsteuerung
- 7.0 - tension control
- 8.0 - Nadelmaschine Typ DI-LOOP OD-SV
- 8.0 - Needle Loom type DI-LOOP OD-SV
- 9.0 - Kantenschneidemaschine Typ LSE mit
- 9.0 - Querschneider Typ RS/m und
- 9.0 - Wickler Typ SW/A
- 9.0 - Edgetrimming Machine type LSE with
- 9.0 - Cross Cutter type RS/m and
- 9.0 - Winder type SW/A

- 1.0 - Rollengang
- 1.0 - conveyor system
- 2.0 - Kantenschneidemaschine Typ LSE mit
- 2.0 - Querschneider Typ RS/m und
- 2.0 - Wickler Typ SW/A
- 2.0 - Edgetrimming Machine type LSE with
- 2.0 - Cross Cutter type RS/m and
- 2.0 - Winder type SW/A

- 1.0 - Vornadelmaschine Typ POD
- 1.0 - Pre-Needle Loom type POD
- 2.0 - Durchhangsteuerung
- 2.0 - tension control
- 3.0 - Nadelmaschine Typ OD-II
- 3.0 - Needle Loom type OD-II

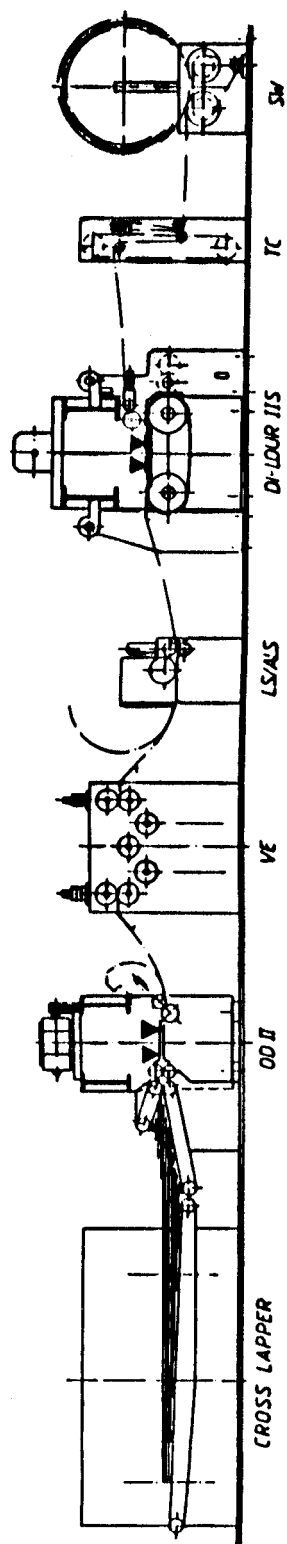


Figure 18:

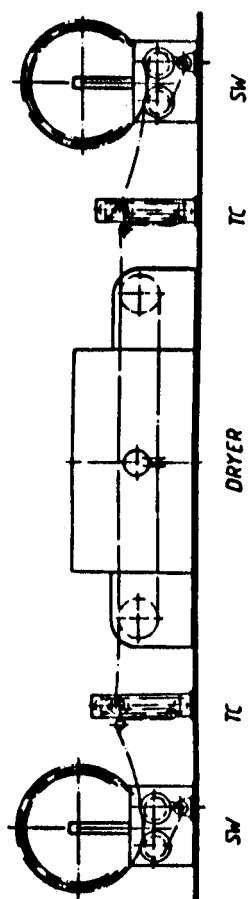


Figure 19:

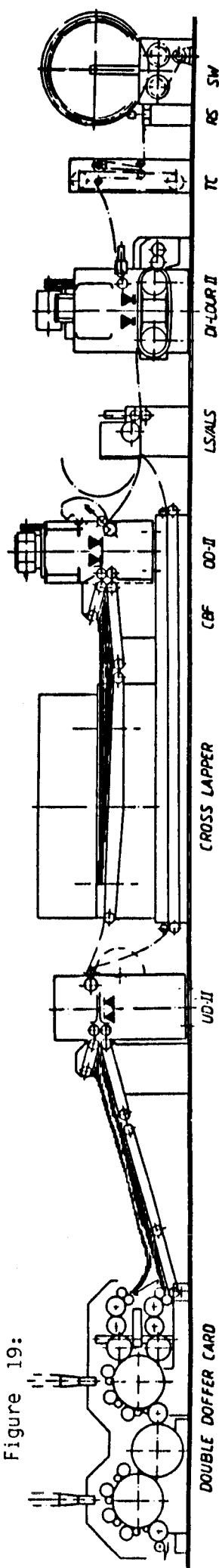
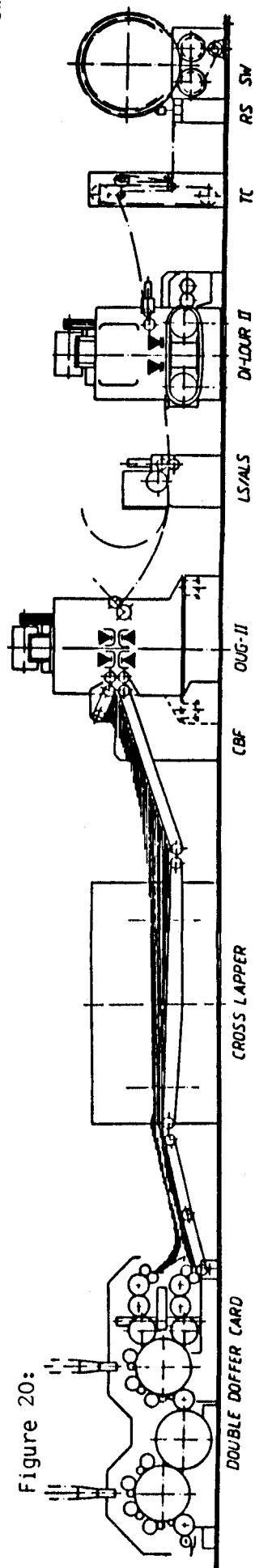


Figure 20:



Figures 18, 19, and 20 Principles of various methods for needle punched upholstery products. Web structure, strength and elastic properties are important so fabric can absorb forces in all directions.

William E. deVry, Rohm and Haas Company

The fibers of nonwoven fabrics are bonded together to give the structure strength and durability. This bonding can be the result of melt fusion of fiber during web formation, such as the case of spunbonded fabrics, or it can be a post-fusion of heat sensitive fibers mixed with non-heat sensitive ones, i.e., the thermal bonding process. Or, lastly, and by far most frequently, it can be a latex polymer binder applied wet to a formed fabric and then dried and cured to provide strength and durability. This technology is the subject of my talk today.

The popularity of latex binder has its foundation built on four important cornerstones:

- Low viscosity - ease of application
- High molecular weight -- toughness
- Binder variety and versatility
- Low cost and economy of use

The variety of latex binders can be bewildering to novice and experienced hand alike. Why so many? To answer this question, let's consider the factors used to differentiate among binders in order to arrive at the best binder for a particular application:

- Backbone composition. The choice of composition influences the glass transition temperature or how hard or how soft a binder is, how much dry tensile strength it has. We are talking here about the chemistry of the polymer chain, influencing the hydrophobic, hydrophylic balance, the polymer's elasticity and its long term aging characteristics.
- Functional groups or the chemistry of the polymer side chain that give the polymer certain special properties, such as adhesion or solvent resistance.
- Molecular weight. With typically very high molecular weights for emulsion polymers, there isn't much variation in this property. Often the polymer is lightly crosslinked during polymerization.
- Surfactant. What type of surfactant, the amount? This important item affects both how the polymer is made and how it is used in an application.
- Maintaining the same ingredients, the polymerization process itself can be varied to yield different polymer structure, for example a core/shell configuration.

The latex polymer is the result of a free radical emulsion polymerization carried out in water where a vinyl monomer is coupled with many, many others through its double bond to yield a high molecular weight polymer dispersed in water. The simplest vinyl monomer is ethylene where R is a hydrogen. The

homopolymer of ethylene, a very important plastic, is not made via emulsion polymerization. Latex binders using ethylene are copolymers with other monomers, such as vinyl acetate or vinyl chloride, where the ethylene serves to provide internal plasticization. Polyethylene emulsions are useful as additives to binders to reduce needle sticking in interlinings for sewn garments or to impart a slick, waxy feel to any nonwoven.

If "R" on the vinyl monomer is a benzyl group, we have styrene and when this monomer is polymerized with butadiene, where R is an ethylene group, we have styrene butadiene rubber or SBR, a polymer used extensively in the tire industry. As a nonwoven binder, the styrene promotes hydrophobicity and tensile strength, while butadiene contributes elasticity, resiliency and toughness. Because of the residual double bond left over in butadiene polymerization, the SBR binder is susceptible to oxidation during heat and light exposure and must be protected with antioxidant.

Butadiene also is copolymerized with acrylonitrile to yield the elastomer and latex polymer acrylonitrile-butadiene, commonly called nitrile, and known for its high oil and solvent resistance combined with elasticity.

Another important monomer is vinyl acetate. Polyvinyl acetate is not a popular nonwoven binder because of its stiffness (primarily a fiberfill or high loft nonwoven binder) but copolymers with ethylene and acrylate monomer, which serve as internal plasticizers, are widely used.

When R in the vinyl monomer is a chlorine atom, we have vinyl chloride, the homopolymer of which is the plastic polyvinyl chloride or PVC. The homopolymer

is a very hard, rigid polymer and must be plasticized, so nonwoven binders using vinyl chloride are copolymers of softening monomers such as ethylene or acrylates. External plasticizers also are added to soften. Chlorine in the polymer promotes flame retardancy but gives the polymer a tendency to yellow on heat aging due to dehydrohalogenation.

Acrylic binders are the predominant binder in the nonwoven industry and, from this point on, I will concentrate on this type because of their utility in a wide range of applications.

Acrylics are versatile, that is their premier advantage. They avoid the tendency to oxidize, embrittle and discolor as in polymers with residual unsaturation, such as the butadiene copolymers. They avoid the susceptibility toward polymer degradation, as with vinyl chloride copolymers under heat aging or vinyl acetate copolymers which tend to hydrolyze when exposed longer term to water, particularly at alkaline pH. Acrylics range from very soft, pressure-sensitive adhesive polymers to very hard, plastic polymers such as Plexiglas and a myriad of combinations between. This range in softness/hardness or in glass transition temperature is a result of compositional variation through monomer choice rather than external modification, such as adding a plasticizer.

Let's review the chemistry of acrylics which leads to their good balance of properties and versatility. The simplest acrylic monomer is acrylic acid. Polyacrylic acids are useful as thickeners due to their tendency to swell when neutralized with base. With nonwoven binders, a small amount of acrylic acid can be copolymerized for colloidal stability, adhesion promotion and to provide sites for crosslinking.

Methacrylic acid, where a methyl group has replaced the hydrogen on the beta carbon of acrylic acid, is also an important acid functional monomer.

These vinyl acids are converted to the acrylate esters in the familiar esterification process with alkyl alcohols. For example, acrylic acid reacted with ethanol gives ethyl acrylate. When butanol is the alcohol, butyl acrylate is the result. Using methacrylic acid instead of acrylic acid, the methyl ester is methyl methacrylate, the monomer for the hard, clear, plastic Plexiglas. These monomers can be copolymerized to yield intermediate Tg polymers.

The acrylate monomer is the primary one and gives the polymer its generic name. But often acrylates are copolymerized with other non-acrylate, but vinyl monomers, for various reasons:

(1) Styrene -	Hardness, hydrophobicity, embossability, lower cost.
(2) Acrylonitrile -	Solvent resistance, durability to dry cleaning.
(3) Vinylidene Chloride -	Flame retardancy, and moisture vapor transmission resistance. Vinyl chloride is also used for flame retardancy but doesn't offer as much chlorine per molecule.
(4) Vinyl Acetate -	Lower cost.

Functional groups are added to a polymer via copolymerization of small amounts of generally more expensive but special-purpose monomers. The reasons for functional groups in a nonwoven binder are primarily to resist hostile environments:

Durability – Washing and dry cleaning resistance

Deformation under heat and pressure -- e.g., blocking resistance or hot elongation resistance

Resistance to solvent immersion

Colloidal stability (mechanical stability, compoundability)

Note that the polymer backbone heavily influences a binder's dry tensile strength while polymer functionality heavily influences wet and solvent tensile strength.

Some of the functional groups commonly put on acrylic polymers and the monomers used to do this are:

<u>Group</u>	<u>Monomer</u>
Hydroxyl	Hydroxyethyl Acrylate
Carboxyl	Acrylic Acid
Amide	Acrylamide
n-Methylol Amide	n-Methylol Acrylamide

Carboxyl functionality is incorporated to provide colloidal stability, adhesion, hydrophilicity, self-thickening and sites for reaction with added crosslinking resin. Incorporating hydroxyl functionality isn't common for nonwoven binders; in the industrial coating arena it is more popular and done to provide crosslinking sites.

Amide functionality through copolymerization with acrylamide provides crosslinking sites. To make a polymer self-crosslinking, a monomer is used which is acrylamide reacted with formaldehyde: n-methylol acrylamide. These monomers often are used together in a polymer to give higher crosslink density.

The acrylic latex polymers can be classified according to their reactivity. I believe there exists some confusion in terminology within the nonwoven industry regarding reactivity. By non-crosslinking we meant that the polymer does not have any of the functional groups above so it does not crosslink with itself or added curing resin. By crosslinkable is meant the polymer has functional groups, such as carboxyl or amide, which will react with added curing resin but the polymer will not crosslink with itself. By self-crosslinking is meant that the polymer reacts with itself to provide a crosslink density for resisting solvents, deformation, etc. This polymer also reacts with added curing resin. And lastly, a thermosetting binder is one which has the curing resin already added and is capable of high crosslink density after curing.

The conventional self-crosslinking acrylic latex needs between 280-300°F polymer (not oven) temperature to effect the intended cure. Adding an acid catalyst brings this temperature down in the range of 260-280°F. Rohm and

Haas has introduced some new chemistry in a series of much lower temperature, in fact, room temperature-curing, cationic, self-reactive, base-catalyzed binders. These low energy cure (LEC) binders employ a nonionic surfactant but have a cationic backbone, giving them unique compatibility with cationic additives, such as water repellants, and unique adhesion to such difficult-to-adhere-to fibers as polypropylene.

The most common curing resin added to latex binders is the melamine-formaldehyde condensate resin. This chemistry is that of n-methylol groups, just as with the conventional self-crosslinking latex polymer. The curing resin reacts with hydroxyl, carboxyl, amide and n-methylol amide groups and it reacts with itself. Other curing resins less commonly used with nonwoven binders are urea-formaldehyde, epoxy, phenolic and metal ion complexes (with carboxyl). The aim of the curing resin addition is to increase crosslink density, not lower curing temperature. By increasing this density, one improves durability and resistance to deformation.

Latex binders seldom are applied straight from the drum or tanktruck. Formulating is done with various ingredients to "fine-tune" the binder for a particular end-use. To be sure, a great deal of technology exists in nonwoven binder formulation. We have discussed already curing resins added for increased durability, solvent and water resistance. Consider the following ingredients commonly found in a nonwoven binder bath formulation:

Order of Addition

Water (most)

Defoamer

Surfactant

Crosslinker, salts, sewing aids or optical brighteners

Latex Binder

Thickener

Catalyst

Ammonia

Dye, Pigment or Filler

Water (dilution)

While it is important to design storage and handling equipment to minimize latex foaming, most baths will tend to foam at the applicator unless a defoamer is used. Defoamers are divided in two major categories, those which contain silicone oils and those which don't. The former (example: Dow H-10) are the more efficient defoaming agents but can cause poor wet-out if you later plan to coat the surface of the fabric. An example of a non-silicone is Nopco NXZ. Latex binder suppliers will have recommendations for their particular latex.

Surfactants or wetting agents are added to enhance binder penetration, modify the binder's hydrophobicity and increase the stability of the bath. They are generally either nonionic or anionic. Nonionics assist in wetting of hydrophobic fibers such as polyesters or polypropylene. Nonionics are the

surfactants of choice to promote chemical stability, such as when adding a cationic water repellent to an anionic latex. Nonionics also tend to act as plasticizers for acrylic latex polymers at higher concentrations. The anionic surfactants are excellent for wetting hydrophobic fibers and are particularly useful for imparting rewet characteristics to the nonwoven. Because of their greater tendency toward foaming (because they more effectively lower surface tension). The level of anionics should be held to a minimum.

The term "salts" refers to flame retardant salts, such as diammonium acid phosphate (DAP), which promote flame retardancy. Normally 50-100 parts of salt are added per 100 parts of polymer on a dry basis and this large amount of DAP requires a special, "salt-stable" latex where chemical stability has been enhanced. DAP is a so-called non-permanent flame retardant -- it can be washed out. A decabromophenyl oxide/antimony oxide mixture is widely used in the textile and nonwoven industries as a permanent flame retardant. The predispersion can be added to most latexes without concern. It is significantly more expensive than DAP, however.

Sewing aids are lubricating agents added to prolong sewing needle life when bonded nonwovens are fabricated in apparel, such as interlinings.

Optical brighteners are incorporated to enhance whiteness and brightness through a bluing action.

The topic of thickeners for latex binders in all applications would merit a seminar alone. For nonwoven binders, thickeners are not so much utilized for thickening action, because, in fact, baths are normally low in solids and low

in viscosity. Rather they function as water-holding agents to promote stability, non-drying on the applicator roll or as a suspending agent to keep a high density ingredient, such as flame retardant or filler, from settling. Thickeners also come into play for coating bonded nonwovens, where rheology is important. I have put the thickeners in two categories: the alkali soluble type and acid soluble type. The former requires neutralization with base, such as ammonium hydroxide and hence are thickeners only at higher pH. These thickeners generally are very efficient and yield pseudoplastic or shear thinning rheology. The acid soluble types can be used at any pH, are generally less efficient and yield more Newtonian or shear rate - independent viscosity. The latter type of rheology is useful for gravure or rotary screen printing of nonwovens.

Catalysts are used to facilitate curing at reduced temperature or to increase curing speed at high temperature. With the conventional self-crosslinking binders using n-methylol amide chemistry, latent or Lewis acid catalysts are recommended such as NH_4NO_3 or NH_4Cl because they decompose during drying to form strong acid catalysts (nitric or hydrochloric). The organic acid catalysts, such as oxalic acid or citric acid, are weaker and must be used at a pH less than 7 because salts of these acids are very poor catalysts.

Permanent alkaline catalysts, i.e., sodium carbonate or sodium sesquicarbonate, are employed with the new room temperature cure binders described earlier. Note: Remember that permanent base, such as caustic, retards the conventional self-crosslinking reaction -- the converse of catalysis.

Ammonia may be used to raise the pH of an acrylic latex to maximize the stability at the applicator. With certain binders, the so-called self-thickening types, ammonia can be used for raising viscosity without the need for external thickener. This technology can be useful in print bonding of nonwovens. The ammonia is driven off during the initial stages of drying.

Dyes and pigments are frequently added to the binder to color a nonwoven fabric, such as the familiar blue for medical/surgical gowns and drapes. Fillers are less frequently used, for while they are lower in cost, they detract from binder properties at levels where real savings accrue. Small amount of clay can serve to detackify a soft binder to facilitate fabric unwind.

At this latter stage of the formulation, a water repellent is added when needed, either to an anionic or nonionic latex which has been stabilized to accept it or to a cationic latex which accepts it without a problem.

The order in which the formulation ingredients are added in preparation of the bath is important. Generally, most materials are added to the dilution water prior to adding the latex in order to see that they are properly incorporated before the latex obscures incompatibility with milky whiteness. Exceptions are the thickener, added to the mix to adjust viscosity, catalyst which normally is left out until just before application to insure stability, ammonia to adjust pH and dye or pigment to color the whole mix. Water is added last to a final desired solids following a solids check.

What I hope to have left with you is a better understanding of the reason latex binder is used, the types of binders available and why they have their place, particularly the acrylics, and finally the technology of latex formulations which fine-tunes the latex binder into focus for the end use.

LATEX BONDING TECHNIQUES

Latex binder is applied to a nonwoven web using four general techniques:

1. Saturation
2. Froth
3. Spray
4. Print

Saturation

Saturation is the most popular because it is easy, fast and economical. It is a process well known in the paper industry. Generally, the coverage on the fiber is complete with add-on of 20–60% dry binder on fiber. Three methods of saturation are:

1. Dip/squeeze
2. Inclined padder or size press
3. Single screen

The binder add-on is controlled by bath solids, nip pressure, line speed and web structure (including fiber). Typical applications for saturation are fabric interlining, wall covering substrate and roofing mat.

Froth

Froth bonding involves the incorporation of air into the latex by mechanical agitation (for example, creating a foam of eight parts of air to one part of latex) and applying the foam to a nonwoven, followed by nip pressure or vacuum

extraction to remove excess. The latex foam in most cases is unstable and breaks down when it is applied to the web. The foam process yields higher binder solids, much less water dilution and thereby a reduced drying load. The bonded web is generally bulkier and of a softer hand due to better placement of binder at fiber crossover points. Some applications for froth bonding are fabric interlining, wipes and shoddy pad.

Spray

The spray bonding method is employed where a high bulk for the nonwoven product is important, as for example, in fiberfill and wipes made via an air laid web formation process. The spraying usually is done with an airless technique where air pressure forces the diluted latex out the nozzle and it is atomized. By controlling the air pressure, the spray nozzle size, the distance from the web, the angle of the spray fan and the line speed, binder add-on, coverage, and depth of penetration can be controlled. Typical add-ons of binder for spray are in the range 10-20% dry binder to fiber.

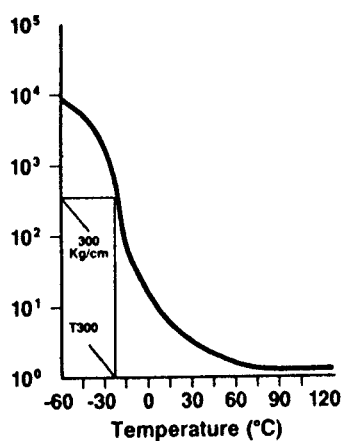
Print

The aim of print bonding is to apply the binder to discreet areas of the web and thereby allow for softer hand and improved absorbency. Because of the noncontinuous application of binder, a higher Tg binder often can be used to yield good strength and a softer hand. Two methods of printing are rotogravure and rotary screen. In both cases, the print design is important, as well as controlling binder penetration into the web; e.g., altering solids to optimize drying and rheology to optimize wet binder migration into the web. A typical application for print bonding is a wipe, such as a disposable kitchen wipe.

GENERAL COMPOSITION

Acrylic	Styrene-Butadiene
Vinyl Acetate	Vinyl Chloride
Ethylene Vinyl Acetate	Hybrids

TORSIONAL MODULUS - TEMPERATURE CURVE OF AN ACRYLIC BINDER



HARDNESS RANGE OF ACRYLIC BINDERS (TG°C.)

Ester	Acrylate	Methacrylate
Methyl	9	105
Ethyl	-22	65
Butyl	-52	20

$$\frac{1}{TG} = \frac{W_1}{TG_1} + \frac{W_2}{TG_2} \dots \text{Where } W_1 + W_2 + \dots = 1$$

BINDER FACTORS INFLUENCING NONWOVENS PERFORMANCE

- Backbone Composition
- Functional Groups
- Molecular Weight
- Surfactant
- Process

FUNCTIONAL GROUPS

- Durability
- Resistance
- Wet Tensile Strength
- Elasticity
- Hard
- Stability

BACKBONE COMPOSITION

- Dry Tensile Strength
- Hand
- Hydrophylicity
- Hydrophobicity
- Elasticity
- Color Retention

MOLECULAR WEIGHT

- Tensile Strength
- Toughness
- Elasticity
- Resilience

SURFACTANT TYPE AND LEVEL

- Adhesion
- Wettability
- Dermatological
- Stability
- (Foam Tendencies)

WHAT IS A LATEX BINDER?

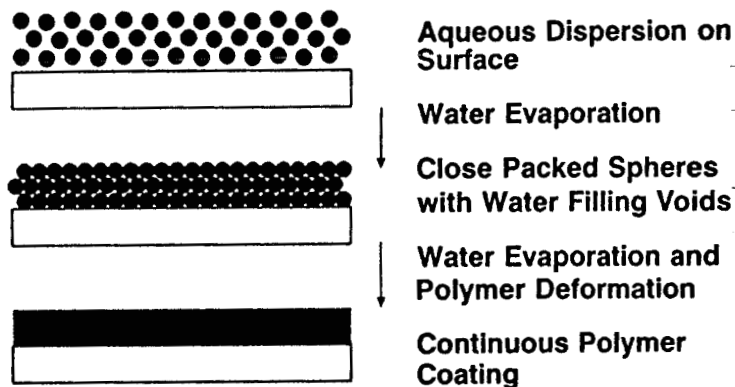
Water
Monomer
Surfactant
Initiator

} Polymerization

Colloidal Polymer Dispersion
Surfactant Stabilized
Water + Salts

PROCESS

- Resilience
- Tensile Strength
- Elasticity



FORMULATING

- Defoamers
- Wetting Agents
- Catalysts
- Crosslinkers
- Thickeners
- Colorants
- Flame Retardants
- Water Repellants
- Antistatic Agents
- Sewing Aids
- Optical Brighteners
- Fillers

DEFOAMERS

Hydrophobic Particulate Agents

- Fatty Amides and Acids
- Silicas

Surface Active Agents

- Silicones
- Fatty Alcohols and Esters

Starting Points: 0.15% Solids Based on Bath

FLAME RETARDANTS

Durable

- Halogenated Organics with Antimony Oxide
- Bromine-Phosphorus Compounds
- Aluminum Trihydrate

Non Durable

- Diammonium Acid Phosphate
- Ammonium Sulfamate
- Triaryl Phosphate Plasticizers

WATER REPELLANTS

Fluorocarbons

- Nonionic Sufactant for Compatibility
- Cationic Latex

Silicones

WETTING AGENTS

Anionic

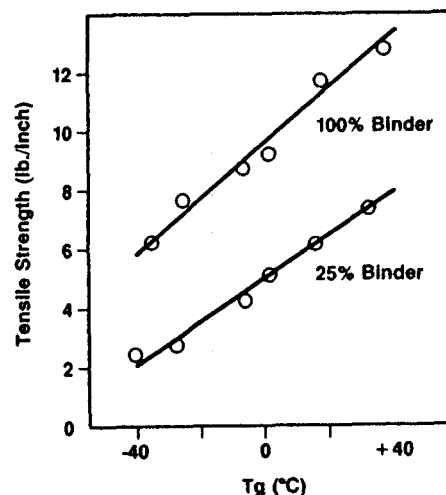
- Alkyl Sulfate
- Alkyl Aryl Sulfonate
- Dioctyl Sodium Sulfosuccinate

Nonionic

- Alkylophenol Ethoxylate
 - Octyl, Nonyl
- EO/PO Block Copolymers

NON-ALKALINE THICKENERS

- Hydroxyethyl Cellulose
- Hdroxypropyl Cellulose
- Methyl Cullulose
- Associative Rheology Modifiers



FILLERS

- Clay
 - 5-20%: Detackifying
 - 10-40%: Filling
- Titanium Dioxide
 - 1-5%: Opacity

CROSSLINKERS

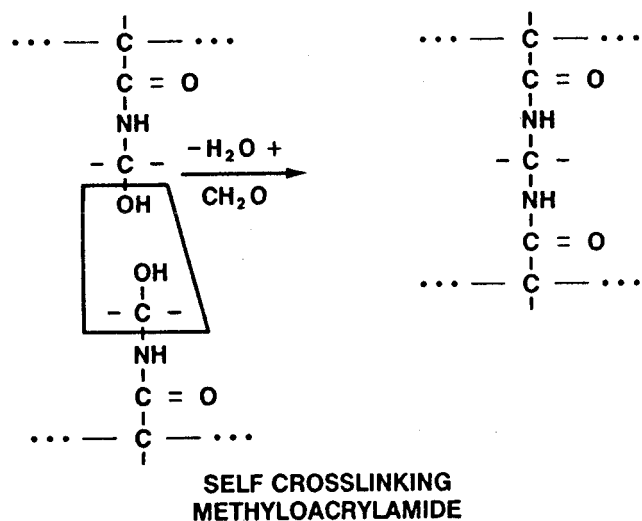
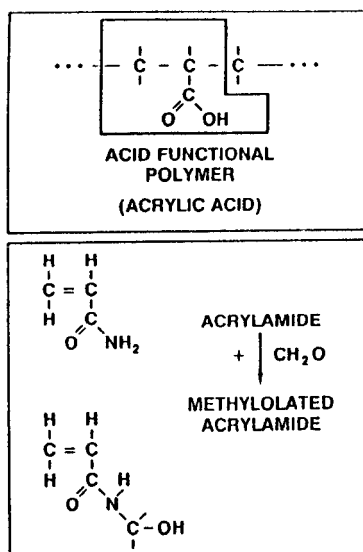
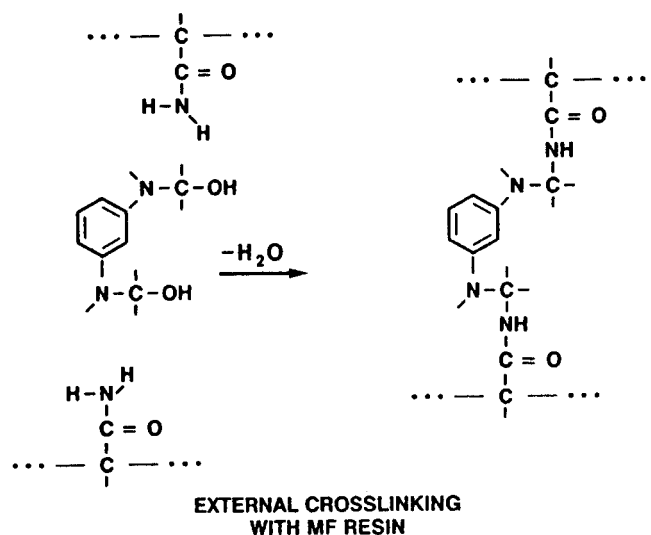
- Aminoplast Resins
 - Melamine/Formaldehyde
 - Hexamethoxymethyl Melamine
 - Trimethoxymethyl Melamine
 - Urea/Formaldehyde
 - Phenol/Formaldehyde
- Water-Borne Epoxies
- Metal Salts
 - Zinc and Zirconium Complexes

REACTIVITY

- Non-Crosslinking
- Crosslinkable
- Self-Crosslinking
- Thermosetting

CATALYSTS

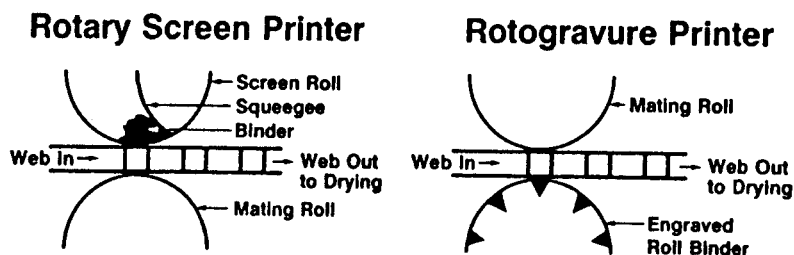
Latent Acid	% Solids/Solids
• NH_4NO_3	0.5 - 2.0
Organic Acid	
• Oxalic	0.2 - 0.5
• p-Toluene Sulfonic	0.5 - 1.0
Alkaline (LEC Polymer)	
• Sodium Sesquicarbonate	1.5 - 2.0
• MgO	0.5 - 1.0



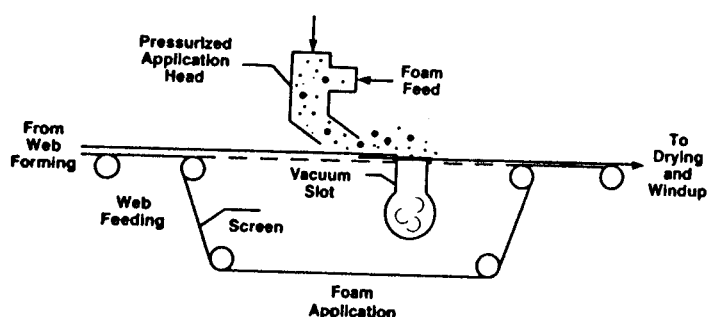
BONDING TECHNIQUES

- Saturation
- Froth
- Spray
- Print

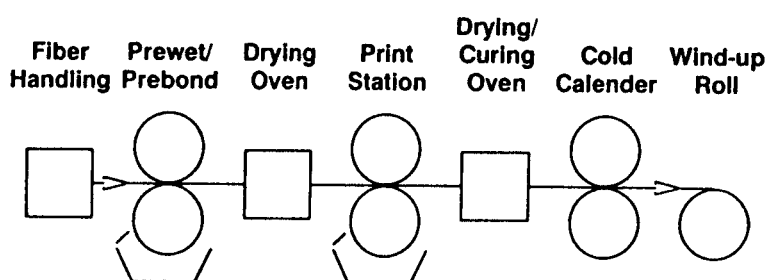
PRINTING EQUIPMENT



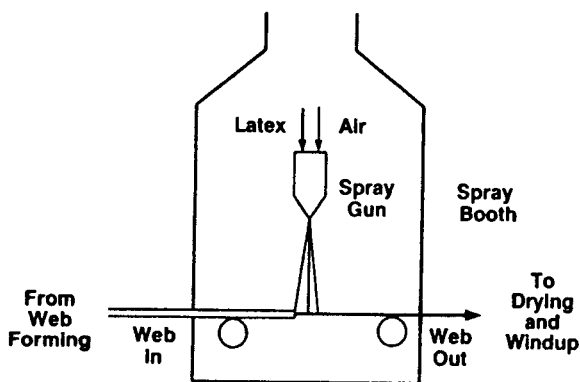
FROTH BONDING



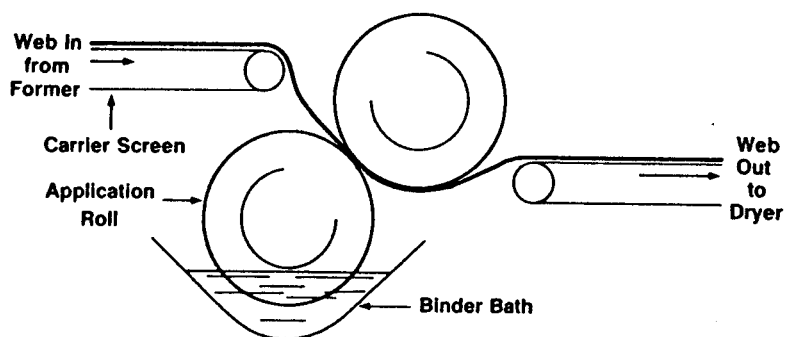
NONWOVEN PRINT BOND LINE



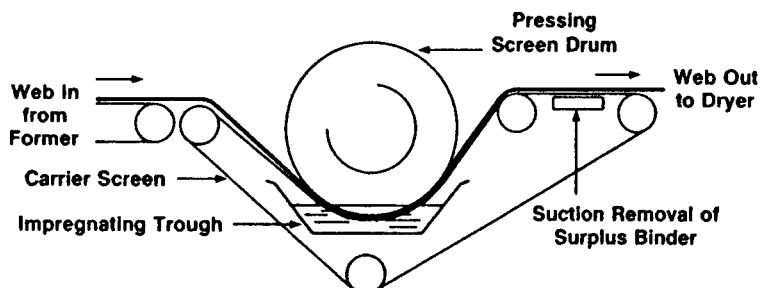
SPRAY BONDING



INCLINED PADDER OR SIZE PRESS



SINGLE SCREEN SATURATOR



NONWOVENS IN ADVANCED FIBER COMPOSITES

PHILIPPE COPPIN CONSULTING

15 NOD BROOK DRIVE, SIMSBURY, CONNECTICUT 06070

A composite, in the materials industry, is generally a fiber/matrix combination made of a fibrous structure embedded in a plastic - the matrix - and exhibiting enhanced structural properties. Those are imparted by "advanced fibers", developments of the last decades in high modulus and strength, the most significant for our purpose being carbon, aramids and high strength glass. This is far from exhausting the list of high technology fibers but the nonwovens industry's experience with the carbons, aramids and special glass fibers would apply to the others as well.

The nonwovens industry often calls nonwoven composites combinations of fiber webs or the resultant of fibers embedded into another web. The nonwovens industry is in the process of discovering the virtue of composites by its own definition, and much R & D effort is currently focused on the combination of nonwoven webs, particularly through water jet needling of fibers into another fibrous substrate. The process - spunlacing or hydroentanglement is not new, pioneered as it was by DuPont and commercialized with a very textile-like material, SONTARA.^R The sudden interest shown by the rest of the industry is expected to bring to the market within a short period, families of new "nonwoven composites", primarily constructed of the familiar fibers: polypropylene, polyester, rayon, woodpulp even cotton and E-glass.

Aside from this interest for "nonwoven composites", the nonwovens route for forming webs had also been explored by some companies to supply materials engineers with the core structure for their composites. It meant handling Nomex[®] and Kevlar[®] fibers, carbon, S-glass, etc... In short, with the exception of one success story - Nomex[®] "paper", the results of over a decade of dedicated work is notable technical success and minimal marketing success. The use of high technology fiber structures has grown considerably but not the nonwoven form. Yet, remarkable webs of carbon, Nomex[®] Kevlar[®], S-glass and all of those fibers in combination are available today - down to weights below 10 gr/m² with excellent formation and isotropic properties.

THE PROCESSES

Looking at the production technology first it is clear that of all the nonwoven processes, wet laid is the most appropriate. No other process compares when it comes to dispersion and uniform formation of individual fibers of whatever type, no other process allows the precise and infinite blending. The wet laid process modeled on paper making, but with important modified features, remains the most versatile tool in web forming. It is, of course, limited in the length of the fibers it can use but much progress has been made and the 20 to 30 mm range has been achieved.

Nonwoven webs do not pack the density of fibers that is usually demanded for structural composites. They can not approach the filament winding methods, tapes of highly packed filaments or even the woven webs when it comes to maximizing structural properties. Hence it was early recognized that the nonwoven form offered a better opportunity in pursuit of other remarkable properties of the advanced fibers: electrical conductivity and chemical resistance (carbon) or electrical resistivity and temperature resistance (aramids). Often these properties can be achieved with a modest amount of fibers, or can be incorporated in a substrate with a minimal concentration in advanced fibers. Even at these low basis weights or concentration prices are a major deterrent to penetration into large volume markets.

Profitability of wet laid production - as the economics of papermaking, is predicated on volume. The inability of advanced fiber nonwoven products to generate a sizeable demand has created a difficult dilemma : production on wide equipment would in a matter of days satisfy the yearly demand for carbon nonwoven webs, for instance. On a wide, versatile commercial machine it would also contaminate the systems and could jeopardize other production. Web prices from very short runs on narrow equipment range today four to five times or more the prices of the fibers - a final cost that few end users can afford.

A recent development may give a new life to the addition of advanced fibers to a substrate. Nordson Corporation known for its expertise in spraying equipment - dry powder, hot melt adhesives, superabsorbent, etc.- has disclosed success in the spraying of various fibers. Much of their work has been with Hoechst - Celanese's new cellulose acetate "fibrets", but also with other and longer fibers. They report in particular having successfully sprayed carbon fibers from 0.4 mm to 4 mm. Spraying allows the addition of variable amounts of fibers without contamination of any primary system. Delivery can take place, for instance, over the wire of paper machine or at the point of formation of a meltblown - it offers considerable flexibility. In the search for economical ways to add advanced fibers to a substrate we view this as a very promising development.

In a review of forming systems for advanced fibers, one can mention Dow Chemical's Random Fiber Composite process, basically a reinforced composite, fiber and matrix, produced in a flocculated wet laid system. The end product is a reinforced plastic in roll form, the reinforcing fibers being, at will, glass, carbon, Kevlar[®] or basically any fiber appropriate to the final properties demanded.

Hydroentanglement deserves special attention among the systems that may give new life to nonwovens with advanced fibers. It demands, of course, a precursor web or some method of laying fibers over a substrate. DuPont has spunlaced Kevlar[®] with Nomex[®], making successful commercial products. Water jet needling may not achieve interlocking with stiff fibers like carbon but can provide the embedding of parallel layers. It is easy to envision a sprayed layer of advanced fibers forced into a substrate by this system.

Current and emerging processes for producing nonwoven webs with advanced fibers are summarized in the Table: Processes for Advanced Fibers.

COMPANIES

DuPont, in this business holds a unique position having developed the most successful family of advanced fibers with Nomex^R and Kevlar^R, and commercialized the only advanced fiber product sold in the nonwoven form in the thousands of tons (Nomex^R paper). DuPont led the way also in applying their spunlacing system to the entanglement of Kevlar^R and Nomex^R.

In the area of wet forming of carbon fibers, International Paper has done pioneering work from the late seventies. The formation and binder capabilities demonstrated by IP sheets continue to be remarkable. It is also apparent that International Paper has dedicated a great amount of application engineering effort and technical contacts with potential users in the search for market openings for its advanced fiber products. This kind of effort is a prerequisite for anyone with the ambition to be a supplier, even at the present minimal demand level.

James Cropper, a UK paper company has been a relative newcomer but established a position and runs today the widest former: 1.2 m. Width may be imposed by the product criteria while production volume may remain

PROCESSES FOR ADVANCED FIBERS

Function	Process	Companies	Fibers
Web Formation	Wet Laid	DuPont Intl.Paper James Cropper Dexter Lydall	any advanced fiber
Fiber Layer deposition	Spray	Nordson (E)	Glass, Carbon others
Direct RP Composite formation	Random fiber composite	Dow Chemical	Any advanced fiber
Layer entanglement, consolidation	Hydroentang.	DuPont Perfojet (E) Honeycomb (E)	most advanced fibers
E = Equipment manufacturers			

more than modest. The small Technical Fiber Products (TFP) subsidiary of James Cropper has the flexibility of entrepreneurial companies and entertains runs of 50 kg - sometimes less.

Dexter proved its manufacturing capability but then shelved the program. This is a technology for "patient money". Lydall has been expanding its expertise in glass webs.

ECONOMICS

The main properties of the better known advanced fibers are by now quite familiar : better strength and modulus for S-glass (compared with E-glass); high modulus, strength, impact and abrasion resistance, temperature resistance, low density for Kevlar[®]; and for carbon very high modulus at low density, electrical and thermal conductivity and very high temperature and chemical resistance.

These properties come at a price.

\$4.00 / lb range for S-glass fibers

\$9.00 / lb range for Ashland Carboflex[®] Pitch Carbon

\$12.00 / lb up for Kevlar[®]

\$18.00 / lb up for PAN Carbon

As a point of comparison, practically all other fibers used in the nonwovens industry are priced below \$1.00 / lb. As the relatively bulky nonwovens do not deliver the full extent of the most demanded property in the final

composite (modulus and strength to weight ratio) the future for nonwoven products using such expensive raw materials may have to await some breakthrough in production and raw material costs.

Production, we observed earlier, is getting a choice of new secondary processes which may alter the multiplication factor of some 4 or 5 to 1 between web cost and raw material costs for very short runs. For nonwoven products exploiting "secondary" properties such as electrical conductivity, chemical resistance, thermal resistance, etc., a true breakthrough and prospects for large volume production will also depend on reduced fiber costs.

Ashland has developed a \$9.00 / lb pitch carbon fiber tradenamed "Carboflex", tried by some nonwoven manufacturers. If it has not been found to deliver some properties as an equal to the more usual PAN fibers, it is nevertheless another option and may be a component in blends. The encouraging sign for the nonwovens manufacturers is observing research towards less expensive fibers in industries accustomed by their aerospace customers to focus on top of the line performance at ever higher prices.

PRODUCTS

For specific product information it is best in this field to refer directly to the manufacturers. Nomex[®] paper has been marketed for many years, and spread to very different end uses from electrical winding to honeycomb structures with many applications (such as airplane flooring). Spunlaced Kevlar[®]/Nomex[®] is of more recent entry, available in various mixes, 15%/85%, 30%/70%, or others. One market for these nonwoven fabrics is fireblocking seat covers in airplanes.

Both International Paper and James Cropper offer a range of 100% carbon sheets, starting below 10 gsm. International Paper also has a range of nickel coated carbon grades aimed at improving EMI/RFI shielding, a field also pioneered by International Paper. Both companies also have light weight 100% Kevlar[®] grades and various fiber blend webs.

This is still a business for the custom designed product. It is also a business where the application engineering expertise of the web manufacturers can be of immense help to someone creative or curious enough to suggest an application.

"REICOFIL, REICOTEX AND MELT BLOWN - THE NEW WAY FOR PRODUCING COMPOSITE FABRICS"

Hermann Balk - Reifenhauser GmbH & Co.
Dale A. Raymond - Fi-Tech, Inc.

ABSTRACT

The traditional Spunbonded Processes have made their success over the past twenty years. By completely rethinking all established theories in order to find easier solutions for the technical problems of a new Spunbonded Process, the "Reicofil" Process has been developed so that it now can be called "a simple process for a simple product". Polypropylene and Polyethylene Nonwoven fabrics can now be produced in a wide range of fabric weights with numerous advantages in the process. New products can now be made from a combination of Spunbonded and Melt Blown Nonwovens in conjunction with multi-layer composite structures.

INTRODUCTION

Another technology has become popular for producing textile fabrics; namely the production of Nonwovens. The guideline for the development of the Reicofil Nonwoven Process has always been "a simple process for a simple product". This Spunbonded Production Line, for manufacturing Polypropylene and Polyethylene Nonwovens, was introduced to the public in 1984. In April of 1988, a Licensing Agreement was entered with Exxon and Reifenhauser is now offering turnkey Melt-Blown Systems.

This unique, compact Spinbonding System offers significant advantages, special features, and has rewritten the conventional standards for making Spunbonded products and multi-layer composite fabrics.

PROCESS STAGES

The pre-mixed polymer with master batch is fed into the spin extruder and processed into a homogeneous, spinnable melt. Through the heated melt flow channel and melt filter, this spinnable melt is conveyed to the heated spin beam with its integrated spinneret. A spin pump uniformly feeds the melt into the spinning unit. In the spin beam the melt is uniformly distributed over the width and emerges under pressure from the spinning system. The filament bundle, consisting of several thousand filaments and cooling air is drawn downward by underpressure created by a ventilator. The drawn filaments are randomly entangled by turbulences in the distributing section and laid down on the conveyor belt screen. The calender bonds the nonwoven web by means of temperature and pressure. The bonded nonwoven is cooled by cooling rolls and conveyed to the winder.

In the winder, the nonwoven can be cut into selectable fabric widths and is wound onto reels. The edge trim waste is fed into the recycling extruder. After replastification, this extruder feeds the edge trim waste directly back into the spin extruder. Thus the line operates practically waste free.

Referring to Figure 1, this process is composed of the following parts:

- raw material storage and feeding
- dosing and mixing unit for polymer and additives
- spin extruder with edge trim recycling extruder
- melt flow channel with melt filter
- one spin pump only
- spin beam with integrated spinneret, electrically heated
- air blowing device with cooling aggregate
- drawing and distributing section
- screened conveyor belt with air-outlet conduit
- embossing calender
- edge trim recycling unit
- winder with cutting unit

This system is based on the principle of underpressure. Over the line's entire working width, ventilator-generated underpressure sucks filaments and cooling air down from the spinnerets and cooling chambers. As a result, filaments are properly drawn and oriented prior to being deposited onto the conveyor belt.

This process requires relatively little energy. For example, the total energy consumption for the conversion of polyolefin pellets into a finished nonwoven web is approximately 1.5 kWh/kg. of production or approximately one-third less than that which is required by other processes. In addition, this is a compact production line that can be set up and made operational quickly and easily. Automatic control of fabric weight distribution is currently being tested and should be available in the near future.

The line's Spinning Unit has a polymer manifold that was developed specifically for this application. Also, colors can be changed in minutes because the unit has an extremely small melt distribution channel. Changing from one polymer to another (e.g., from polypropylene, which has a spinning temperature of 240°C, to polyethylene, with a spinning temperature of 180°C) can also be carried out quickly because the Spinning Unit is electrically heated, and any requested temperature can be obtained without difficulty. Dowtherm is not used in this system.

Another important feature is the previously-mentioned Edge Trim Recycling Unit. During production, it is necessary to continuously trim the edge of the nonwoven web. In fact, edge trims of up to 20% of the web's width can be recycled in some cases. All waste trim is returned to the spinning process by the Edge Trim Recycling Unit.

Reifenhauser also offers a unique procedure for handling the testing and installation of ordered equipment. As a part of this process, each Line is assembled in the factory and tested under production conditions in the presence of the customer's staff, using the customer's choice of resin. Training also takes place at this time. After a successful test run, the Line is disassembled, and subsequently reassembled in the customer's plant, and is operational within a short period of time. The average assembly time is only five weeks.

Start-up procedures, acceptance runs and tests involving different polymers, web weights and pigments take approximately two weeks. In contrast, assembly and testing of competitive Spinbonding machines can take as long as one year.

Twenty Reicofil Lines have been ordered since 1984 by the U.S.A., the U.K., Japan, Israel, West Germany, Canada, People's Republic of China, Sweden, Brazil and Venezuela.

The first production plants were delivered having a final fabric width of 1.6 m. The next generation lines were made to produce 2.4 m Spunbonded fabrics. Reifenhauser is now building Spinbonding Systems which are 3.2 m wide and can offer line widths which are 4.2 m, 4.8 m and up to 5.0 m.

Not only line widths have increased; line speeds have increased from 50 m/min. to 75 m/min. and the next generation Lines are targeted to produce Spunbonded fabrics up to 125 m/min. having a maximum throughput of 500 kg/hr. for a 5.0 m wide line.

FABRICS

One of the process elements that provides this system a competitive advantage is its applicability to several resin types. Figure 2 illustrates the virgin polymer viscosity as a function of shear rate when using polypropylene pellets 3 mm dia. x 3 mm long, having a specific weight of 0.91 g/cm³ and a melt index of: MFI 230/2.16 which equals 25-35 g/10 min. at 230°C. The residual moisture of the virgin resin should be below 0.1%, dust content less than 0.5% and the ash content should be between 0.015 and 0.025%.

As seen in Figure 3, the recommended polypropylene has a narrow molecular weight distribution and relatively low molecular weight and viscosity. This is advantageous relative to forming the nonwoven web specifically concerning the thermal bonding characteristics of the fabric. Figure 3 also shows the molecular weight mass average (MW), molecular weight number average (MN), the viscosity average of the recommended molecular weight (MV), and the sedimentation average (MZ). The ratios of averages represent the range of molecular weight distribution.

FABRIC PROPERTIES

The tensile strength properties of Reicofil fabrics are nearly equivalent in the machine direction and cross direction as seen in Figure 4. This data has been plotted, given a range of fabric weights from 30 g/m² to 150 g/m². In addition to having nearly equivalent tensile strength values in both the machine and cross direction, Figure 5 shows that the percentages of maximum elongation and shrinkage are as well nearly the same in the two directions.

High tensile strength which is accompanied by fabric toughness, good tear strength durability, high resistance to surface abrasion, as well as other enhanced physical qualities such as softness and opaqueness tell us that these fabrics result in good product performances at lower fabric weights, and therefore can be produced at lower cost given a wide range of fabric weights (from 15 g/m² to several hundred g/m² in fabric widths from 1.0 m to 5.0 m).

FABRIC APPLICATIONS

This Spinbonding Process offers a variety of end product applications given the wide range of fabric weights. Figure 6 tabulates many of the products currently being made using Reicofil Nonwoven fabrics.

Throughput (kg/h)

<u>g/m²</u>	<u>3.2 m</u>	<u>4.2 m</u>	<u>5.0 m</u>
15	235	310	360
30	250	330	390
50	260	370	420
75	300	400	460
100	320	420	500

Production Speed (m/min)

<u>g/m²</u>	<u>3.2 m</u>	<u>4.2 m</u>	<u>5.0 m</u>
15	80	82	80
30	42	44	43
50	26	29	28
60	23	24	24
75	20	21	20
100	17	16	15

CONCLUSION

As a result of the simplification of a Spunbonded Process, a Spinbonding Line was designed and built at relatively low capital equipment cost. While the whole world is running Spunbonded Processes faster and faster, the Reicofil Line is able to produce Nonwoven fabrics at a comfortable speed, and still produce a highly-profitable product. This also means that a wide range of polymers (e.g., polypropylene resin having an MFI range from

20 to 60, recycled polypropylene, and polypropylene powdered resins) can be used.

Another advantage of the lower line speed leads finally to a new name:

REICOTEX

Reicotex is the name of both an extended Reicofil Process and the products made by this new Process. With a simple system any other fabric, woven, nonwoven, or film, can be guided on top of the unbonded filaments and finally bonded together with the Reicofil fabric at the calender unit. This offers a wide area of new products which are currently in the developmental stage. Initial trial production runs are looking very promising.

Polyolefin sealing can result in products that are not permeable to water, but that can "breathe". This offers a number of new product applications or improvements, including protective clothing, house wrappings, and shower curtains. In addition, sealing increases web strength--especially of polyethylene nonwovens--improves printability, as well as the printed image, and also has a positive influence on the textile hand.

Figure 7 shows that the average strength values of Reicotex Nonwovens are approximately 30% greater than those of Reicofil materials.

In the Reicotex Process, various materials have been combined with polypropylene and polyethylene nonwovens. For example, polypropylene-based fabrics have been produced by combining high-density polyethylene, low-density polyethylene, linear low-density polyethylene, polypropylene film and staple fibers. Polyethylene-based fabrics have been produced from a combination of polyethylene resins and polypropylene fibers. Both spunbonded (PP and PE) have been bonded to chalk-filled films making a very attractive composite product. Using these kinds of combinations, textile hand can be made stiffer, softer, more paperlike, or even silky. Appearance can as well be made shiny. Even the use of a second color is possible. This variety of characteristics undoubtedly will create a number of new applications, especially as more fabric producers and converters become familiar with the Reicofil and Reicotex processes.

REICOFIL + REICOTEX + MELT-BLOWN + LAMINATION SYSTEM

A Licensing Agreement was entered with Exxon for their very well-known Melt-Blown Process. This agreement includes worldwide marketing rights and an exclusive European right to manufacture melt-blown lines using Exxon's technology. Referring to Figure 8, one will see this new Melt-Blown-System incorporated into the Reicofil system including laminating capabilities for making multiple layer composite fabrics.

Melt-Blowing is a patented one-step process in which high-velocity air blows molten thermoplastic resin from an extruder die tip

onto a conveyor or take-up screen to form a fine-fibered web. These webs, or Melt-Blown Nonwoven fabrics as they are popularly known, have excellent hand, moderate strength, and a wide variety of end uses.

Products made from Melt-Blown Nonwovens, or composite structures including Melt-Blown webs, continue to grow in both number and diversity. Figure 6 tabulates many of the Melt-Blown products currently available on the world market.

The Exxon Melt-Blowing Process uses high-velocity air to form small diameter fibers, typically 3 microns. Resin is fed into an extruder where it is melted and then heated to the temperature required for Melt-Blowing. The molten polymer then enters the Melt-Blowing die, which is the heart of the Process. As it emerges from the die, polymer is drawn by high-temperature, high-velocity air. A large volume of cooling air is drawn into the primary jet as it leaves the die so the fibers reach their final diameter and solidity within about 1/4" of the die tip. Polypropylene continues to be a major resin used in Melt-Blowing. More recently, however, a number of other resin systems have been used in Melt-Blowing, significantly broadening the market for products made by this Process. Among these are low and high density polyethylene, ethylene copolymers, nylon, polyamides, polyesters, polystyrene, poly-4-methylpentene-1, polymethylemethacrylate, polytrifluorochloro-ethylene, polycarbonates, polyurethanes, silicones, pitch and a blend of these before-mentioned resins.

Reifenhauser has combined their Reicofil and Reicotex Processes with Exxon's Melt-Blowing Process to form a multi-composite system. This allows us to produce a minimum of 3-layer composites having a theoretically non-ending number of variations of fabric types.

CONCLUSION

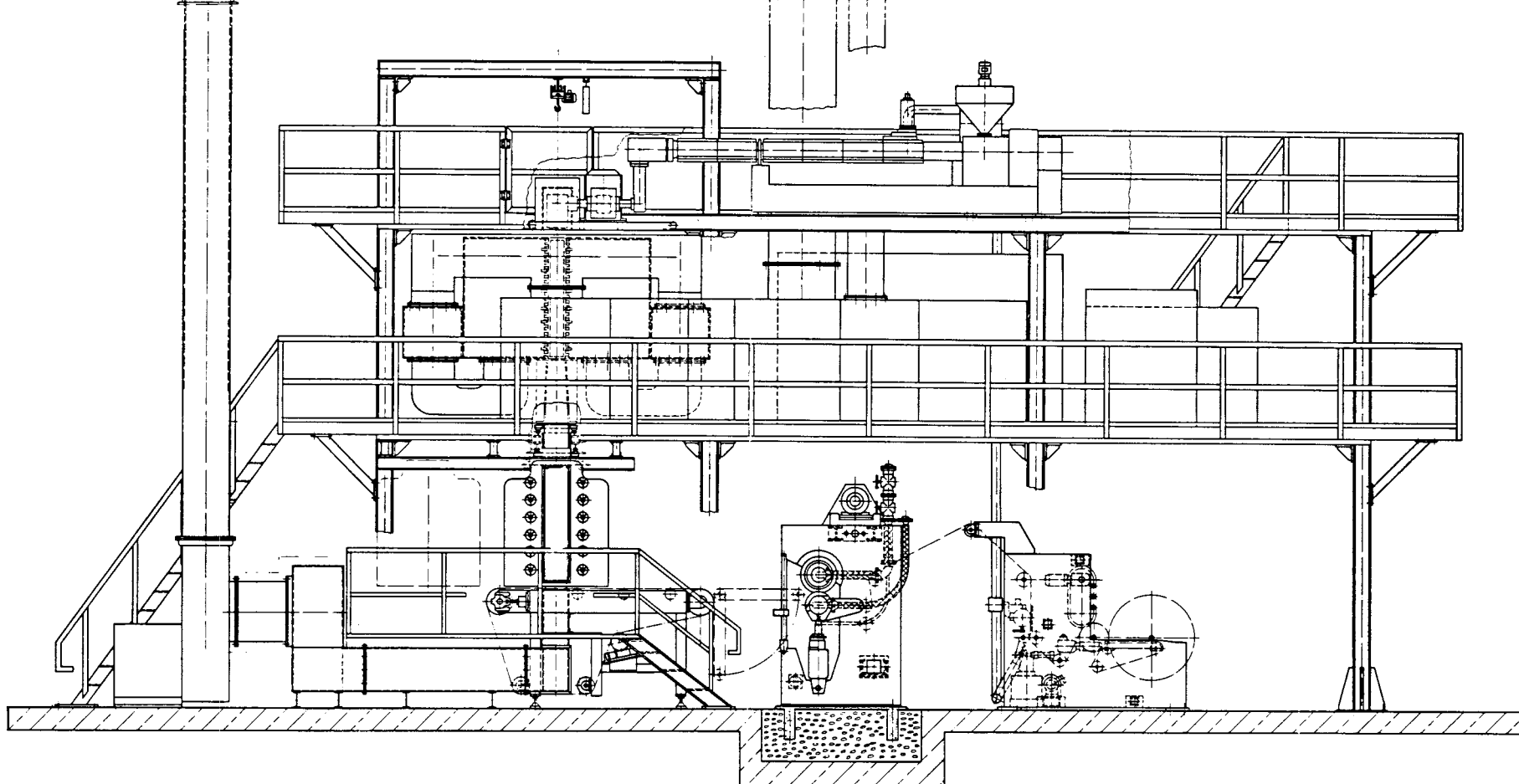
Spunbonded products and multi-layer composite fabrics will expand into totally new and different markets. All indications are Spunbonded fabrics will continue to expand significantly in existing markets. This includes both disposable and durable markets.

Given Reifenhauser's engineering capabilities and machine development program that was used to successfully introduce this new Spunbonding System around the world within only 3 and 1/2 years, yet higher goals will be achieved in the future.

Reifenhauser has already begun to work on the Reicofil System in order to convert it to a Polyester Nonwoven Producing System. A name is not yet given but the new head-line is still the same:

"Another simple process for another simple product."

Figure 1



1 3-Component dosing and mixing unit	8 Adapter between dosing pump bloc/ spin beam	16 Stretching device	24 Edge trim unit with refeed unit
2 Extruder	9 Spin beam	17 Exhaust channel	25 Operating and working platform
3 Refeeding extruder	10 Spinneret pack with spinneret plate	18 Suction fan	26 Travelling crap
4 Measuring head	11 Spinning pump drive with frame	19 Non-woven collecting machine with screen-belt conveyor	27 Storage and peeding device
5 Adapter between extruder and measuring head	12 Filament cooling device	20 Endless-Screen belt unit	28 Temperature control cabinet
6 Adapter between measuring head and dosing pump bloc	13 Monomeric suction	21 Embossing machine and calander	29 Thyristor cabinet
7 Dosing pump bloc	14 Cooled air compressor	22 Heating unit	30 AC-Power Supply
	15 Gasket	23 Non-woven web-winder	31 Operating panel for auxiliary machine

Viscosity of the Resin as a Function of Shear Rate

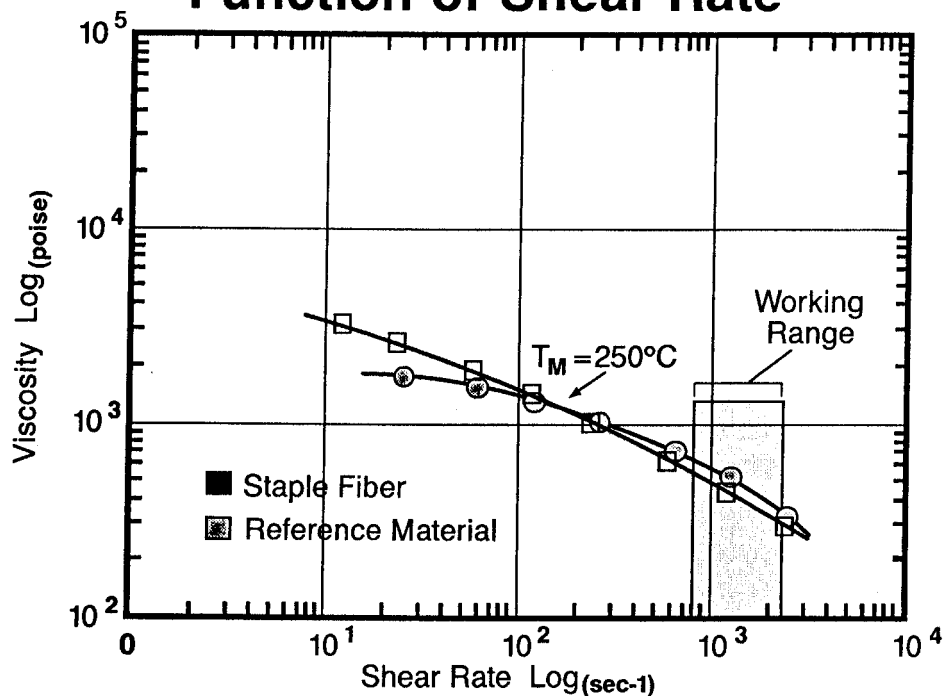


FIGURE 2

Molecular Weight Distribution Recommended Range

$M_W = 209460$
 $M_N = 56055.7$
 $M_V = 179234$
 $M_Z = 492552$
 $\frac{M_W}{M_N} = 3.74$
 $\frac{M_Z}{M_W} = 2.35$
 $\frac{M_Z}{M_N} = 8.79$
 $M(90) = 49436$
 $M(10) = 24625.4$
 $M_{PEAK} = 151433$

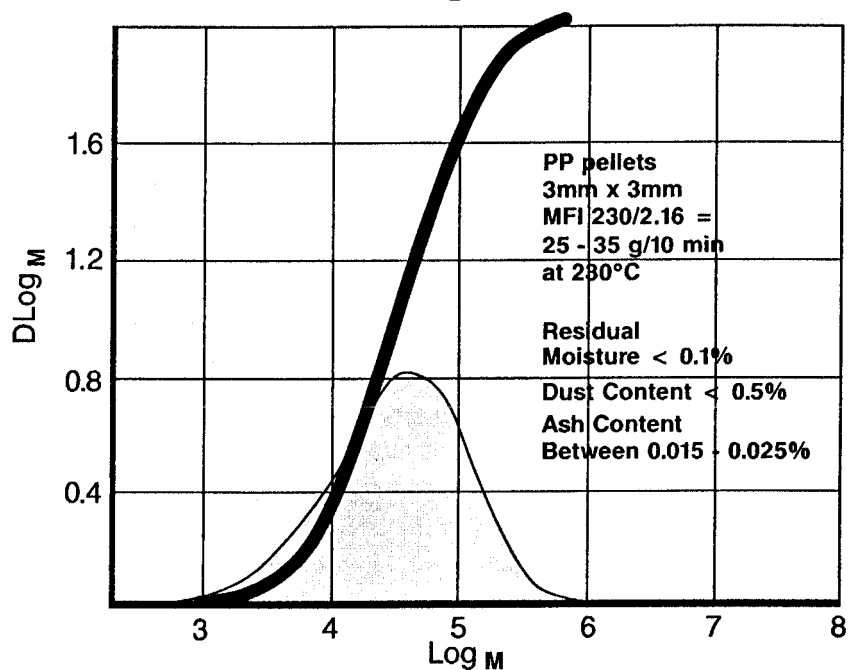


FIGURE 3

Tensile Strength of Reicofil Fabrics

Machine Direction vs. Cross Direction

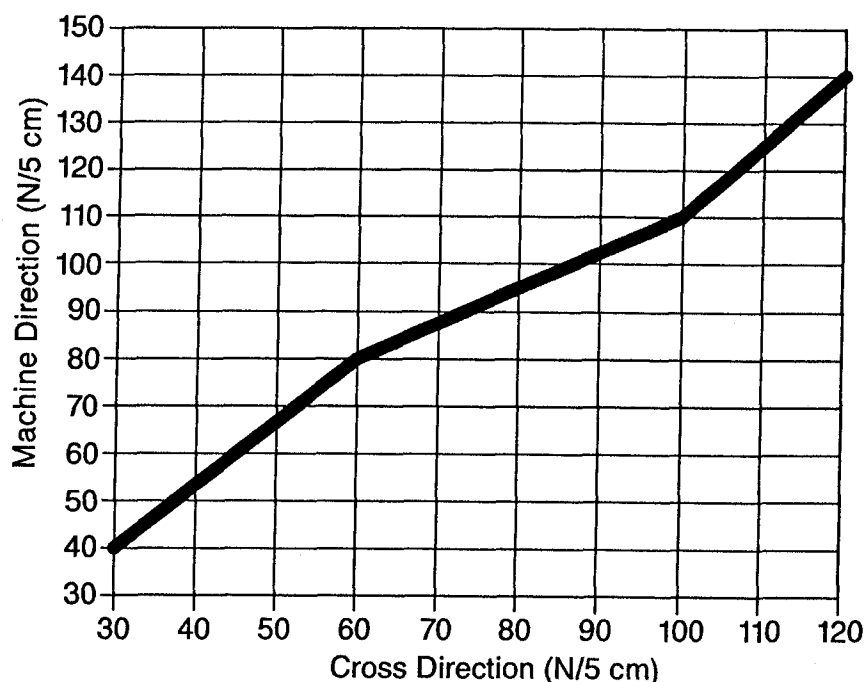
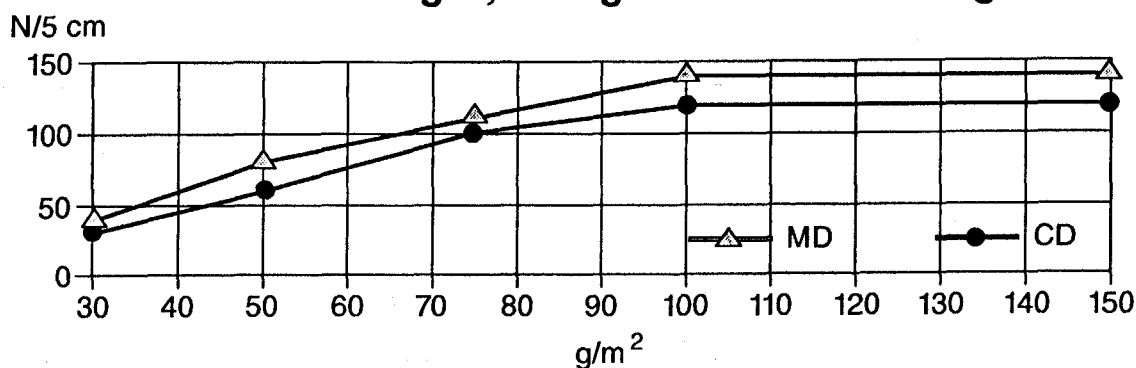


FIGURE 4

Properties of Reicofil Fabrics

Tensile Strength, Elongation and Shrinkage



Weight (g/m ²)	30	50	75	100	150
Max. Elongation MD (%)	30-60	50-100	80-120	80-120	60-90
Max. Elongation CD (%)	30-60	50-100	80-120	80-120	60-90
Shrinkage S (%) (130°C, 20 sec., hot air oven)	<2	<1	<1	<1	<1

FIGURE 5

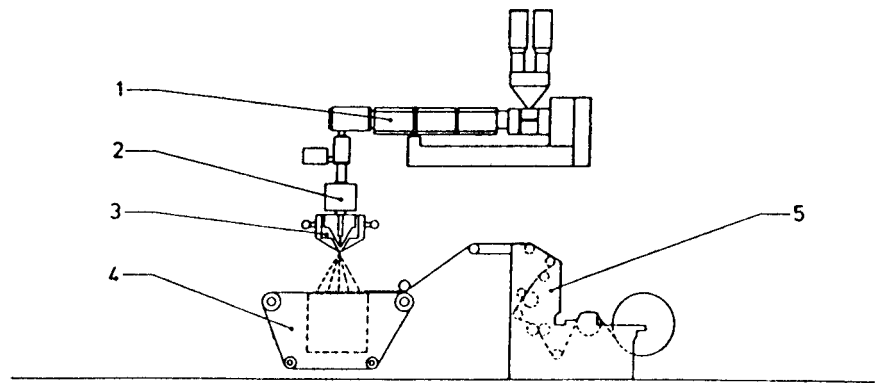
	g/m^2		g/m^2
1. Furniture industry garden/leisure sector/ upholstered furniture	17-110	12. Timber industry (veneers/armorings)	40-110
2. Horticultural non-wovens/ shadings	17-110	13. Home textiles (curtains, pillows, window curtains)	17-100
3. Automotive industry	30-100	14. Flockings	17-60
4. Insulation (pipes/cables)	17-100	15. Building industry	90-150
5. Filters (dry/wet)	17-80	16. Geotextiles/ shore protection	100-250
6. Luggage industry	50-100	17. Carpet backing/floor	90-140
7. Substrates PU/PVC	60-110	18. Packaging sacks	30-90
table cloths	42-60	19. Disposables/ disposable laundry	30-70
bathroom mats	42-110	20. Road repairing/ rapid curing cutback	80-150
8. Sanitary articles	15-42	21. Wall coverings/ tapestry	40-70
9. Medical articles for clinics	30-70		
10. Protective clothes	40-100		
11. Shoeware industry	30-90		

Average Product and Strength Values of Reicotex and Reicofil Non-Woven



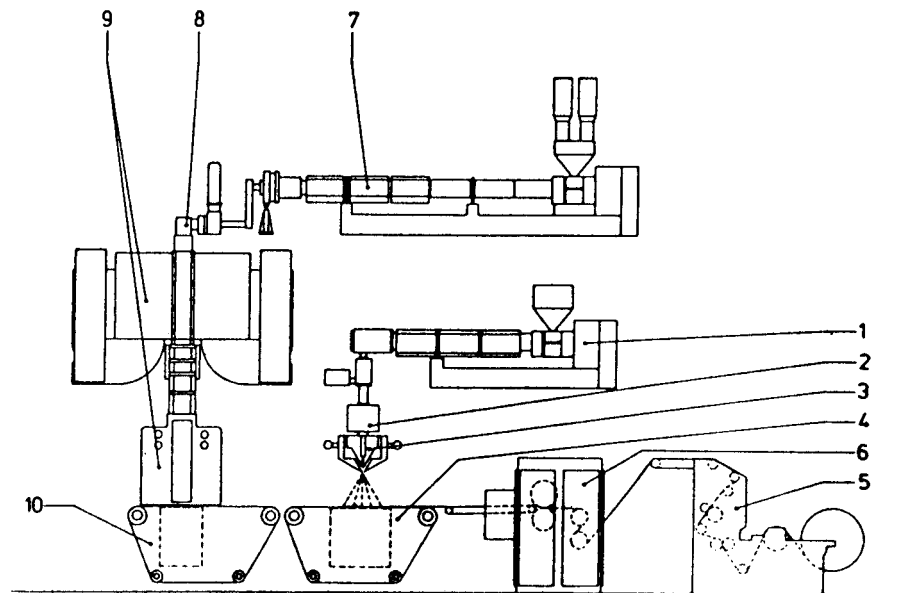
Melt-Blown-System

- 1 Extrusion for Melt-Blown
- 2 Melt-meetering
- 3 Melt-blowing die
- 4 Collector machine
- 5 Slitting and winding



REICOFIL plus Melt-Blown-System

- 1 Extrusion for Melt-Blown
- 2 Melt-meetering
- 3 Melt-blowing die
- 4 Collector machine
- 5 Slitting and winding
- 6 Heat-embossing
- 7 Extrusion for REICOFIL-Spinbonding
- 8 Melt-meetering
- 9 REICOFIL Process
- 10 Collector machine



REICOFIL plus Melt-Blown plus Lamination-System

- 1 Extrusion for Melt-Blown
- 2 Melt-meetering
- 3 Melt-blowing die
- 4 Collector machine
- 5 Slitting and winding
- 6 Heat-embossing
- 7 Extrusion for REICOFIL-Spinbonding
- 8 Melt-meetering
- 9 REICOFIL Process
- 10 Collector machine
- 11 Unwinder for lamination

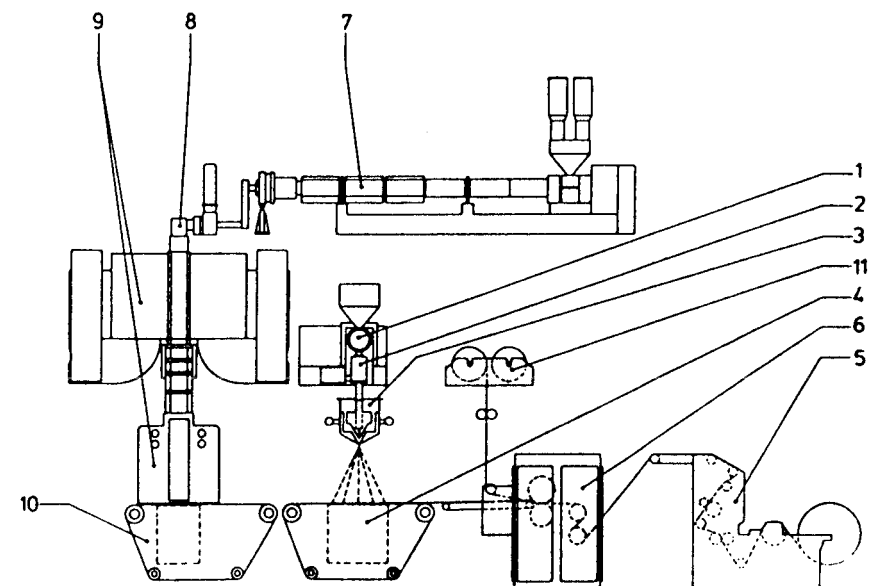


Figure 8

MELT BLOWING AIR FLOW SYSTEM DESIGN CONSIDERATIONS

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ABSTRACT

Since the major energy cost associated with melt blowing is the hot compressed air it is important to minimize these costs with good engineering design. This paper details several areas of design consideration which impact directly on air cost. Pressure losses associated with air piping and the air heater are one of the principal energy cost areas and several design rules are presented to minimize these cost. Compressor sizing or matching is the other design area considered. Use of the design rules presented in this paper should result in minimizing the hot-compressed air costs.

INTRODUCTION

The melt-blown fabric process has been advanced and refined greatly in the past few years. It has become apparent that the cost associated with the hot air streams will be one of the major operational cost for this process. Typically, more than 90 percent of the energy required at the die will be associated with the hot air required to draw the fibers.

The energy costs associated with the air delivery to the die are determined primarily by the compression and heating requirements. Typically a melt blown facility will consist of an air compressor, an air heater, and the connecting piping. Figure 1 shows schematically a typical air system.

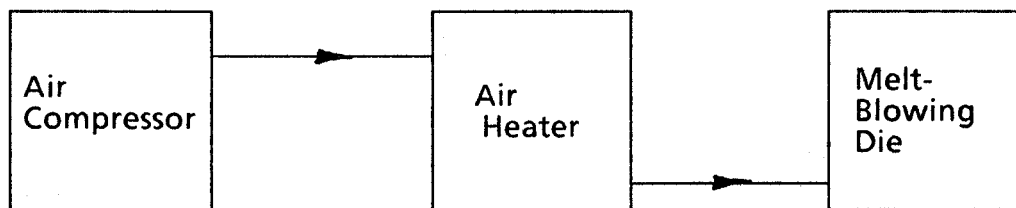


Figure 1 - Typical Air Flow System

The air compressor must compress the air to a discharge pressure which is sufficient to overcome the flow resistance between the compressor and the die. Normally, the required pressure at the die will be between 15 and 35 psi. Thus the compressor must provide air at this required die pressure plus the pressure loss associated with the piping and heater. With this in mind a portion of this paper is devoted to design considerations to minimize piping pressure loss between the compressor and die. Another portion of the paper is concerned with air compressor considerations.

PIPING PRESSURE LOSS

There will always be a pressure loss associated with moving or pushing air through pipes and fittings. To illustrate the importance of the choice of pipe size let us consider an application where we are using 300 SCFM (Standard Cubic Feet Per Minute) of air. One word of caution, there are several definitions of "standard air" so care must be exercised in "standard air" interpretations. For the purpose of this paper we will take 300 SCFM to mean 300 cubic feet of air having a density of 0.075 pounds mass per cubic foot.

If we consider this SCFM flow rate and take the air to be at say 95°F and 40 psig we can determine the pressure loss per foot of length for different pipe sizes. For illustration we will take our pipe to be galvanized, schedule 40 pipe. This calculation is the standard one found in any introductory fluid mechanics book (1). The calculation process is as follows:

1. Knowing the pressure and temperature use the ideal gas equation of state to calculate actual air density.
2. Knowing air flow rate, air density, and flow area of the pipe calculate the average velocity, V , in the pipe.
3. Knowing the type pipe and ID calculate the relative roughness of the pipe, ϵ/D .
4. Knowing the velocity, actual air density, air temperature (viscosity), and pipe diameter calculate the Reynolds number, R .
5. Use the Reynolds number and relative roughness in conjunction with a Moody friction factor chart to obtain a pipe friction factor, f .
6. Use the friction factor, air velocity, and pipe diameter in the Darcy pressure drop formula to calculate the pressure drop.

Using this calculation process for our 300 SCFM of air at 95°F and 40 psig results in the values shown in Table I below. The pressure drop is given in psi per foot of pipe length.

Table I
PRESSURE LOSS FOR 300 SCFM OF AIR FLOW
(Air at 95°F and 40 psig)

<u>Nominal Pipe Diameter</u>	<u>Average Air Velocity (fps)</u>	<u>Reynolds No.</u>	<u>Friction Factor</u>	<u>Pressure Drop psi per foot</u>
1"	234.8	4.52×10^5	0.022	0.400
2"	60.5	2.29×10^5	0.020	0.012
3"	27.5	1.55×10^5	0.020	0.0017

The significance of pipe diameter is obvious. To quantify this consideration in general terms we can look at the Darcy pressure drop relationship as shown in Equation 1.

$$\frac{\Delta P}{L} = \rho \frac{f}{D} \frac{V^2}{2} \quad (1)$$

Using the fact that the velocity depends on the mass flow rate, $\dot{m}$; the air density, ρ ; and the pipe diameter, D , as shown in Equation 2, we can

$$V = \frac{\dot{m}}{\rho \frac{\pi}{4} D^2} \quad (2)$$

develop the following relationship for a fixed flow rate, friction factor and air density (pressure and temperature).

$$\frac{\Delta P_1}{\Delta P_2} = \frac{D_2}{D_1} \frac{\left(\frac{1}{D_1}\right)^4}{\left(\frac{1}{D_2}\right)^4} = \left(\frac{D_2}{D_1}\right)^5 \quad (3)$$

Thus we see that for a given flow rate of standard air, air density, and pipe friction factor the pressure drop per unit length varies inversely with the diameter raised to the fifth power, with this in mind Design Rule No. 1 can be stated as follows:

RULE 1: KEEP PIPING RUNS BETWEEN THE AIR COMPRESSOR AND DIE AS SHORT AS POSSIBLE AND USE LARGE DIAMETER PIPE.

Another important pressure drop consideration has to do with the effect of air density. Using Equation 1 for a fixed mass flow rate and pipe diameter the pressure drop per foot of length depends on the air density as shown below.

$$\frac{\Delta P_1}{\Delta P_2} = \frac{\rho_1 \left(\frac{1}{\rho_1}\right)^2}{\rho_2 \left(\frac{1}{\rho_2}\right)^2} = \frac{\rho_2}{\rho_1} \quad (4)$$

If the air behaves close to an ideal gas we can rewrite Equation 4 to give

$$\frac{\Delta P_1}{\Delta P_2} = \frac{\rho_2}{\rho_1} = \frac{T_1}{T_2} \frac{P_2}{P_1} \quad (5)$$

The conclusion is that for a given flow rate of standard air and pipe size the greater the air density the lower will be the pressure drop. Since the air heater associated with the melt blowing process results in a lower air density, due to higher temperature, Design Rule No. 2 can be stated as follows

RULE 2: LOCATE THE AIR HEATER AS CLOSE AS POSSIBLE TO THE DIE. (This will save thermal energy due to reduced heat transfer area and compression energy due to reduced pipe pressure loss).

The third major area associated with piping pressure loss had to do with fittings. For the purpose of illustrating these losses we will again use our 300 SCFM, 95°F and 40 psig, air flow conditions. Most fluid mechanics references (1), (2) express the pressure loss for fittings in terms of a loss coefficient, k as shown in Equation 6. As

an example of fitting losses

$$\Delta P = \rho k \frac{V^2}{2} \quad (6)$$

let us consider regular screwed 90° Elbows on 1" , 2" and 3" pipe. Table II shows the results.

Table II
90° Regular (Screwed) Elbow Pressure Drop

Size	Loss Coefficient, k	Pressure Drop
1"	1.50	2.4 psi
2"	0.95	0.1 psi
3"	0.72	0.016 psi

To further illustrate the importance of fittings let us consider our same flow conditions and pipe sizes for gate valves. The results are shown in Table III

Table III
Gate Valve Pressure Drop

Size	Position	Loss Coefficient, k	Pressure Drop
1"	open	0.24	0.38 psi
2"	open	0.16	0.017 psi
3"	open	0.12	0.0027 psi
1"	1/2 closed	4.08	6.46 psi
2"	1/2 closed	2.72	0.286 psi
3"	1/2 closed	2.04	0.045 psi

A third design rule can be stated with regard to fittings.

RULE 3: MINIMIZE THE NUMBER OF PIPE FITTINGS, USE LARGE FITTINGS ON LARGE PIPE, AND MINIMIZE FITTINGS WHICH THROTTLE (Partially open valve) or HAVE LARGE PRESSURE LOSS (Air Heater with large pressure drop).

There are obviously other design consideration with regard to air flow system pressure drop but these three rules cover the major energy loss areas associated with piping. Since any pressure loss in the piping must be supplied by the compressor we will now look at some air compressor considerations.

AIR COMPRESSOR CONSIDERATIONS

No attempt will be made here to discuss the relative merits of particular brands or types of compressors. The focus will be on illustrating the importance of the correct sizing of a compressor intended to serve a melt blowing process. Once appropriate values of the capacity and discharge pressure have been established normal purchasing procedures should result in a compressor to meet the specifications.

Let us first consider compressor capacity. Often we think the bigger the better but for a compressor operating at less than its design or rated capacity the energy cost per unit of air compressed is increased. This increased cost is due to the fact that when the compressor is unloaded it still requires a significant fraction of the rated power. To illustrate this point let us consider a single screw rotary compressor rated at 650 SCFM and requiring 171 bhp at the design or rated capacity. Typically this compressor will require about 18 percent of the rated power when it is unloaded. Let us consider an application where we only need 400 SCFM capacity. Equation 7 given below can be used to estimate the energy cost for below capacity operation.

$$\frac{\text{cost}}{\text{cfm}} = \frac{P_{\text{rated}}}{Q_{\text{actual}}} \left[\frac{Q_{\text{actual}}}{Q_{\text{rated}}} + P_{\text{fraction unloaded}} \left(1 - \frac{Q_{\text{actual}}}{Q_{\text{rated}}} \right) \right] \quad (7)$$

Inserting the numbers for the compressor described above gives the results below

$$\frac{\text{cost}}{\text{cfm}} = \frac{171}{400} \left[\frac{400}{650} + \left(\frac{18}{100} \right) \left(1 - \frac{400}{650} \right) \right]$$

$$\frac{\text{cost}}{\text{cfm}} = 0.293 \frac{\text{hp}}{\text{cfm}} = 0.218 \frac{\text{kw}}{\text{cfm}}$$

Another common way of quoting compression energy cost is cost per 1000 ft³ of air. Table IV is a summary of compression energy cost for this example.

Table IV
Compression Energy Cost for Typical Rotary Compressor
 (Single Screw Rated at 650 SCFM)

<u>Air Flow Rate</u>	<u>kw</u> <u>cfm</u>	<u>kw-hr</u> <u>1000 ft³</u>
650 SCFM (Rated capacity)	0.196	3.27
400	0.218	3.63
200	0.276	4.60

The trend shown in Table IV is typical but reciprocating compressors have smaller power requirements for unloaded operation. Maybe 10 percent of the rated power as opposed to 18 percent for the rotary compressor example given above.

The other major consideration in sizing a compressor is the required discharge pressure. This discharge pressure will be very sensitive to the piping system as illustrated in the first part of the paper. In order to illustrate the energy cost associated with various discharge pressures the following formula (3) may be used to give the energy cost in KW per 100 cfm.

$$\frac{\text{cost}}{100 \text{ cfm}} = 20 \left[\left(1.13 R_c \right)^{0.286} - 1 \right] \quad (8)$$

R_c is the ratio of the discharge pressure in psia to the intake or ambient pressure in psia. To illustrate the use of Equation 8 let us consider a compressor having a discharge pressure of 100 psig. Using an ambient pressure of 14.6 psia gives

$$\frac{\text{kw}}{100 \text{ cfm}} = 20 \left[\left(1.13 \frac{114.6}{14.6} \right)^{0.286} - 1 \right] = 17.4 \frac{\text{kw}}{100 \text{ cfm}}$$

Equation 8 is intended only as a "ball park" estimating formula since the actual performance is dependent on the type of compressor, size or capacity, and other parameters. Equation 8 can be used to reliably predict the effect of compression ratio. Suppose we only needed air at 45 psig discharge rather than the 100 psig used in the illustration above. We will use Equation 8 to estimate the energy savings if we only compressed to 45 psig.

$$\frac{\text{Power}_{0 \rightarrow 45 \text{ psig}}}{\text{Power}_{0 \rightarrow 100 \text{ psig}}} = \frac{\left[\left(1.13 \frac{59.6}{14.6} \right)^{0.286} - 1 \right]}{\left[\left(1.13 \frac{114.6}{14.6} \right)^{0.286} - 1 \right]} = 0.63 \quad (9)$$

Thus we would save about 37 percent on compression cost by using a compressor with the required 45 psig discharge pressure rather than one with a 100 psig discharge pressure. We will state an additional design rule.

RULE 4: ESTABLISH THE REQUIRED FLOW RATE AND DISCHARGE PRESSURE CAREFULLY BEFORE PREPARING SPECIFICATIONS FOR THE AIR COMPRESSOR.

Generally, the specifications might call for about 20 percent excess capacity and discharge pressure capability in a well designed and analyzed air system.

As stated previously it is not the intent of this paper to discuss the relative merits of different brands or types of compressor. However, two observations may be of value in planning air systems. For a given capacity and discharge pressure a reciprocating compressor will require between 10 and 20 percent less energy per unit of air but the initial cost will be greater than for a rotary machine. Also the penalty for off design operation will be 5 to 15 percent greater for a rotary machines. With these and other consideration in mind it is apparent that any air compressor specifications should require bidders to furnish performance data on hp/cfm at both loaded and unloaded operation. With this information and the initial costs it will be possible to minimize not just operating cost but total cost as well.

SUMMARY

Since the energy cost for air is such a major portion of the operating cost of melt blowing it is important to make good design decisions initially. The design rules presented in this paper should help to ensure an air system which has minimal energy costs during operation.

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Process-Structure-Property Relationships In Melt Blowing of Different Melt Flow Rate Polypropylene Resins

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ABSTRACT

The process-structure-property relationships in melt blowing of ultrafine fibers are complex and involve many variables. On-going research utilizing the 20-inch melt blown pilot line at the University of Tennessee is providing a greater understanding of these relationships at both macroscopic and microscopic levels. In this research, the effects of polymer throughput on the mechanical and morphological properties of polypropylene melt blown webs were studied in terms of stress-strain properties, bending rigidity, bursting strength, heat of fusion, wide angle x-ray diffraction (WAXD) patterns, gel permeation chromatography (GPC) analysis, and single fiber strength. The 300 and 1400 Melt Flow Rate (MFR) polypropylene resins were melt blown using two throughput rates: 160 g/min and 320 g/min. As the throughput rate increased, the breaking strain value decreased, possibly due to more fiber entanglements and the presence of a greater number of large diameter fibers. However, the breaking strength remained relatively constant with increasing throughput rate. The heat of fusion values and WAXD patterns indicated that there were no morphological changes, especially the degree of crystallinity between low and high throughput webs. The GPC analysis showed that 300 MFR resin experienced more molecular degradation when processed at high throughput rate.

INTRODUCTION

In recent years, there has been a considerable interest in the melt blowing process from both a commercial and a scientific point of view. The melt blowing process falls under the general classification of spunbonded nonwoven textiles. It is a process in which usually a thermoplastic, fiber forming polymer is extruded through a horizontal die containing several hundred small orifices. Convergent streams of hot air (exiting from top and bottom sides of the die tip) rapidly attenuate the extruded polymer streams to form extremely fine diameter fibers (1 -5 μm). The attenuated fine fibers subsequently get blown by high velocity air onto a collector screen -- thus forming a nonwoven melt blown web. Figure 1 shows the schematic of the Exxon melt blowing pilot line at The University of Tennessee (UTK). The fibers in the melt blown web are held together by a combination of entanglement and cohesive sticking. Since the fibers are drawn to their final diameters while still in the semi-molten state, there is no downstream method of drawing the fibers in this semi-molten state before deposition onto the collector, and hence the webs exhibit low to moderate strength.

Researchers at UTK have studied the process-structure-property relationships of melt blown fibers and fabrics in which different melt flow rate (MFR) polypropylene resins have been melt blown under different conditions. The process-structure-property relationships in melt blown fabrics of ultrafine fibers are complex and involve many variables. In this research, effects of polymer throughput on the mechanical and morphological properties of polypropylene melt blown webs were studied in order to provide a greater understanding of process-structure-property relationships at both macroscopic and microscopic levels. The physical and morphological effects of the melt blown process on fiber and fabric properties were characterized using different melt flow rate (MFR) resins as a function of throughput rates.

The effects were evaluated in terms of stress-strain properties, bending rigidity, bursting strength, heat of fusion measurement, wide angle x-ray diffraction (WAXD) patterns, gel permeation chromatography (GPC) analysis, and single fiber strength.

EXPERIMENTAL METHODS AND PROCEDURES

Materials

The polymers used in this study were commercial Exxon 300 and 1400 MFR homopolypropylenes whose characteristics are given in Table I. Polypropylene is the most commonly used polymer for melt blown nonwoven products. This is because of its economic edge over raw materials and ease of processing.

Web Preparation

Melt blown webs for this study were made on the 20-inch (50.8 cm) melt blown line at The University of Tennessee. The line features a 20-inch state-of-the-art die, an electric furnace for heating the air, and an Ingersoll-Rand 650 scfm (18.4 m³/min) air compressor. A 2-inch (5.08 cm) extruder (30/1 L/D) was utilized for this study. The process conditions were monitored and controlled through a microprocessor.

An experimental flow chart is shown in Figure 2. The 300 and 1400 Melt Flow Rate (MFR) PP resins were melt blown using two throughput rates: 160 g/min and 320 g/min. The throughput rate of 160 g/min is typically used in our 20-inch melt blown laboratory trials, while throughput rates of 320 g/min or greater may be utilized in commercial processing. The process conditions used for this study are given in Table II. There are a number of

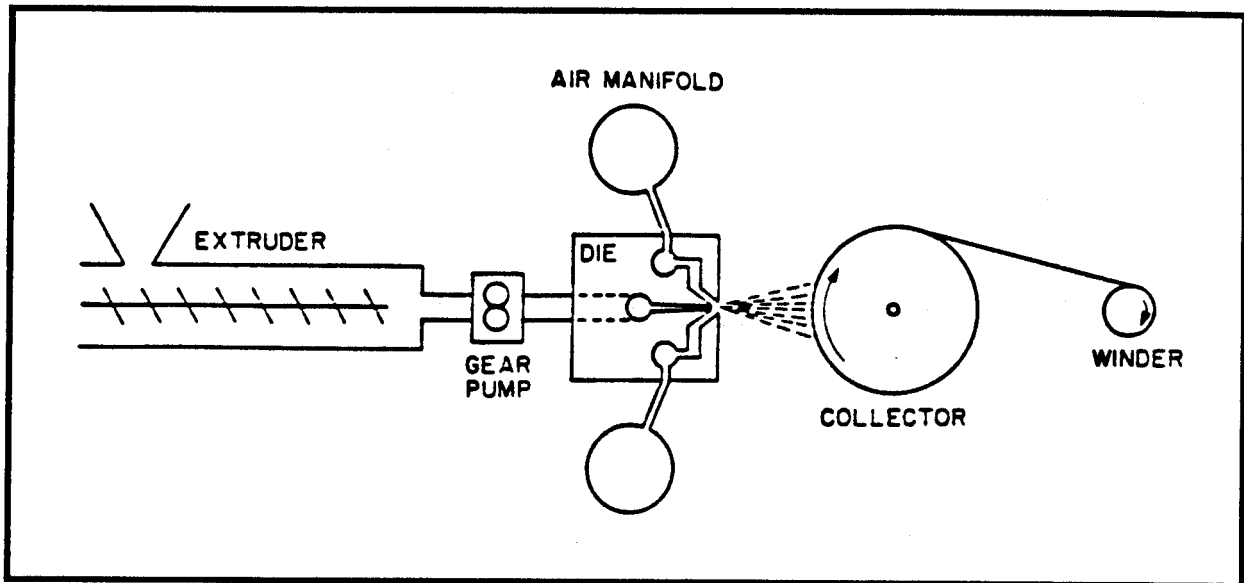


Figure 1. Melt Blown Pilot Line at The University of Tennessee.

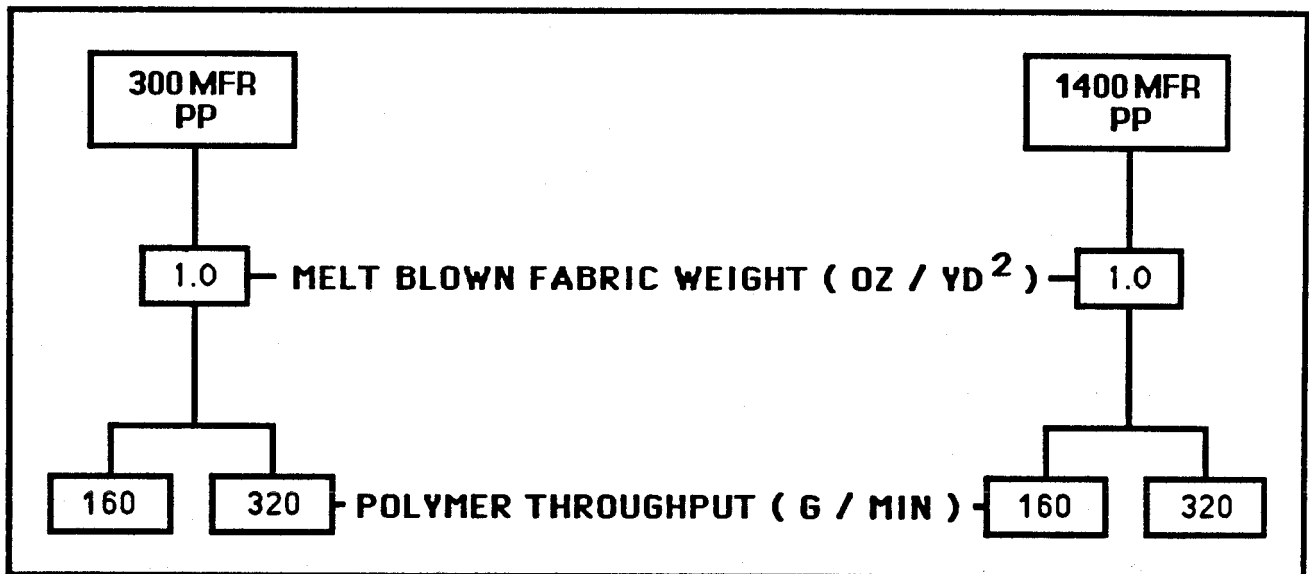


Figure 2. Experimental Design.

Table I
Characteristics of 300 and 1400 MFR Resins

Properties Exxon Code	Melt Flow Rate (g/10 min)	Intrinsic Viscosity (dL/g)	Weight Average Molecular Weight M _w	Number Average Molecular Weight M _n	Polydispersity Index $\bar{M}_w/\bar{M}_n$	Molecular Weight at Peak Max. (by GPC)
PP 3145-44290	300	1.06	145486	55593	2.62	105729
PP 3776-44212	1400	0.76	94552	45505	2.08	77239

Table II
Operating Conditions for melt blown webs

MFR VARIABLES	300		1400	
	160 g/min	320 g/min	160 g/min	320 g/min
Melt Temperature (F)	483	512	440	470
Die Temperature (F)	488	520	450	470
Air Temperature At Die (F)	540	572	462	480
Air Valve Opening (%)	75	75	75	75
Screw Speed (RPM)	20	40	20	46
Collector Speed (FPM)	29	58	29	58
DCD (inches)	12	12	12	12
Fabric Weight (oz/yd ²)	0.99	1.00	1.00	1.00

operations variables which affect fabric properties. Since the main purpose of this work was to study the effects of throughput rates on fibers and fabrics properties, the following conditions were held constant: die-to-collector distance, air rate, and fabric weight of 1 oz/yd² (33.91 g/m²). The other key process variables -- polymer and air temperatures -- were changed as needed to produce optimal conditions in order to achieve similar tactile hand. For example, the 300 MFR resin has been shown in previous trials to result in much stiffer fabric and in large fiber diameters when processed at the same temperatures as 1400 MFR resin.

Physical Properties Measurement

The physical properties of the webs were characterized in terms of the tensile stress-strain properties, bending rigidity, and bursting strength. Two sets of twenty-five one inch (2.54 cm) width strips were cut for tensile and bending rigidity measurements. One set had the long dimension parallel to the machine direction (MD), and the other had its long dimension parallel to the cross direction (CD). For bursting strength measurement, 25 5 x 5 inch (2.54 x 2.54 cm) square samples were cut. The samples for testing were selected randomly over a length of 50 meters.

Tensile stress-strain properties were measured in accordance with ASTM standard D-1682. Both MD and CD tensile properties were measured using the Instron Tensile Tester. The load-extension curves of each sample type were averaged to generate a composite stress-strain curve. A technique for averaging the curves was developed in which, for each curve, the extensions were measured at different load levels. After obtaining the extension data from all 25 curves, they were averaged to obtain average extensions at different load levels. The load and extension data were then normalized to obtain results in terms of specific stress and engineering strain.

The averaged stress-strain curves were plotted using DISSPLA graphic software; a simple cubic spline function was used to interpolate the curves.

The stress-strain properties of single fibers were measured using a floor model Instron tensile tester. The method for mounting and testing single fibers as described by Choi et al. [6], was employed for this study with appropriate changes. A gauge length of 2 millimeters, a cross-head speed of 2 mm/min, and a full scale load corresponding to 10 grams were used. The single fibers chosen for testing had diameters ranging from 3 to 25 μm .

The bending rigidity of the webs, as described in ASTM standard D-1388, was measured by means of the Cantilever Bending Tester. The bursting strength was measured using the Mullen Bursting Tester in accordance with ASTM method D-3387.

Heat of Fusion Measurement

A model DSC-7 Perkin Elmer Differential Scanning Calorimeter was used to measure the experimental heat of fusion ΔH_f^* . A temperature range of 50 to 200 $^{\circ}\text{C}$ was used with a heating rate of 10 $^{\circ}\text{C}/\text{min}$. Indium was selected as the standard to calibrate the measurements. The exact weight of samples ranging from 3.5 mg to 4 mg was determined with an accuracy of 0.001 mg in an aluminum pan. The DSC curves were normalized and evaluated using TAS7 Perkin Elmer software. The percent crystallinity was calculated from the heat of fusion data by the use of following equation:

$$X_c = (\Delta H_f^* / \Delta H_f) \times 100 \%$$

where ΔH_f^* is the heat of fusion of the sample and ΔH_f is the heat of fusion of one hundred percent crystalline material. The value of heat of fusion of 100% crystalline polypropylene used in this work as 146.5 J/g [8].

Wide Angle X-Ray Diffraction

Wide angle x-ray diffraction (WAXD) patterns for the webs were obtained using a Philips Norelco x-ray generator and flat plate camera. Nickel filtered, copper radiation (wavelength 1.542 Å) was used. The x-ray films were exposed for 20 hours with a generator setting of 35 kV and 20 mA. X-ray diffractograms were obtained using a Rigaku diffractometer with copper radiation in reflection mode. Samples were prepared by cutting webs into small square pieces and pressing them on a glass disk to form a mat of 2 mm thickness. The diffractograms (two-theta scans) were obtained over the angular range of 5° to 35°.

Gel Permeation Chromatography

A gel permeation chromatography (GPC) analysis of web and resin samples was carried out at the Exxon Chemical Company, Baytown, Texas. GPC analysis was performed to calculate the polydispersity index of resins and webs, and also to assess the molecular degradation of processed samples.

Scanning Electron Microscopy

Photomicrographs of the webs were taken using the ETEC Auto Scan electron microscope. Four photomicrographs at 50x and 500x magnifications were taken for each sample. Photomicrographs were taken to study the surface characteristics of fibers and variations in fiber diameter as a function of polymer throughput.

RESULTS AND DISCUSSION

The fundamental aim for this work was to study the structure-property-process relationships in terms of mechanical and morphological properties in the melt blowing of different MFR resins at low and high throughput rates. An objective of the study was also to generate a statistically reliable data base by testing a greater number of samples and thereby minimizing the possibility of making incorrect conclusions from a limited set of data.

Physical Properties

The physical properties of 300 and 1400 MFR polypropylene melt blown webs as a function of throughput rates are given in Table III. Figures 3 and 4 show that at the lower throughput of 160 g/min, tenacity decreased with increasing MFR in the MD, and increased slightly with MFR in the CD. Figures 5 and 6 show that the breaking elongation increased with MFR in both machine and cross directions. As shown in Figures 7 and 8, the flexural rigidity decreased with increasing MFR in the MD and remained relatively constant in the CD. These trends have also been observed by other researchers [16]. The shorter chain lengths in high MFR resins should result in fewer chain entanglements and less crystallinity. Thus, a decrease in tenacity and flexural rigidity, and an increase in breaking strain with increasing MFR would have been expected. Figure 9 shows that the bursting strength was inversely proportional to MFR, which was expected since polymer strength is generally proportional to molecular weight.

Overall, the higher throughput (320 g/min) samples appeared to have a harsher hand and more brittle failure behavior. It is believed that due to poor heat transfer at higher throughput rates, thermal fusing of fibers

Table III
Physical Properties of 300 and 1400 MFR melt blown webs

PROPERTIES		5 SAMPLES				25 SAMPLES				WEIGHT		FLEXURAL RIGIDITY (kg·cm)	BURSTING STRENGTH (kPa)	SINGLE FIBER	
SAMPLE ID		STRESS (mN/tex)	STRESS (g/den)	STRAIN (%)	STRESS (mN/tex)	STRESS (g/den)	STRAIN (%)	STRESS (mN/tex)	STRESS (g/den)	STRAIN (%)	oz/yd ²	g/m ²		STRESS (MPa)	STRAIN (%)
300	160 g/min-machine	x	14.96	0.169	4.45	15.39	0.174	9.09	19.89	11.76	0.96	32.54	654.88	33.94	26.07
		%cv	23.46	23.46	12.81	19.89	19.89	11.76							
	160 g/min-cross	x	6.17	0.069	12.84	5.98	0.068	10.95	8.45	14.01	0.99	33.57	162.85	30.14	25.18
		%cv	9.69	9.69	14.09	8.45	8.45	14.01							
1400	320 g/min-machine	x	11.65	0.132	2.19	11.42	0.129	2.76	17.76	27.32	0.95	32.08	733.21	28.47	39.42
		%cv	22.61	22.61	13.47	17.76	17.76	27.32							
	320 g/min-cross	x	5.48	0.062	2.60	5.97	0.068	3.31	18.43	40.96	1.00	33.91	269.50	26.65	40.01
		%cv	21.68	21.68	37.79	18.43	18.43	40.96							
300	160 g/min-machine	x	12.07	0.137	11.93	11.60	0.131	14.14	8.84	34.86	0.95	32.08	480.31	28.47	39.42
		%cv	7.11	7.11	30.19	8.84	8.84	34.86							
	160 g/min-cross	x	6.35	0.072	44.06	6.56	0.074	47.64	6.29	12.79	1.00	33.91	201.24	26.65	40.01
		%cv	5.48	5.48	9.98	6.29	6.29	12.79							
1400	320 g/min-machine	x	11.86	0.134	2.81	12.09	0.137	2.93	8.14	16.69	1.00	33.91	739.36	26.65	40.01
		%cv	10.02	10.02	15.69	8.14	8.14	16.69							
	320 g/min-cross	x	4.11	0.047	2.91	4.22	0.048	3.57	13.17	20.24	1.00	33.91	235.83	26.65	40.01
		%cv	25.05	25.05	20.54	13.17	13.17	20.24							

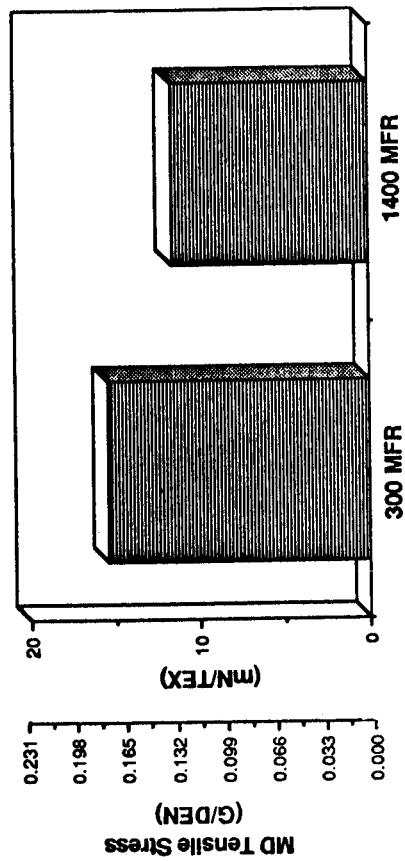


Figure 3. Effect of MFR on MD tenacity at 160 g/min throughput.

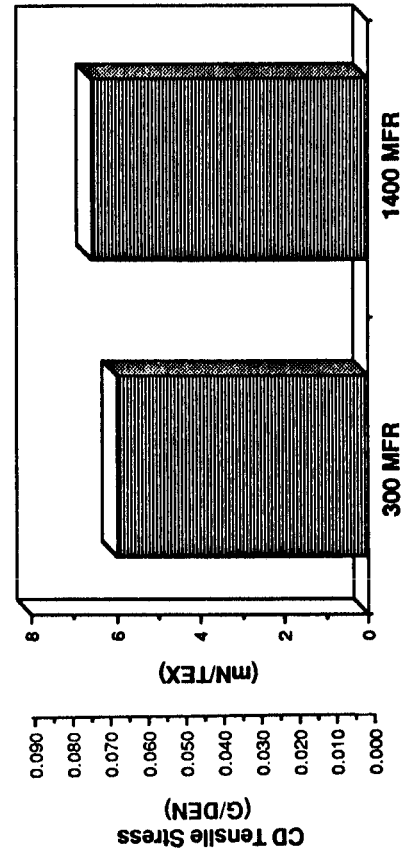


Figure 4. Effect of MFR on CD tenacity at 160 g/min throughput.

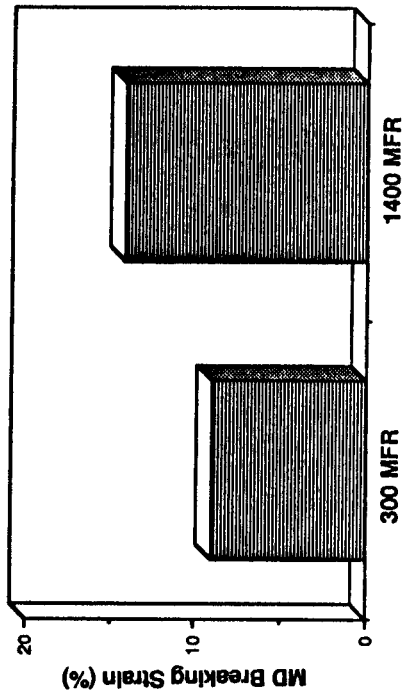


Figure 5. Effect of MFR on MD breaking elongation at 160 g/min throughput.

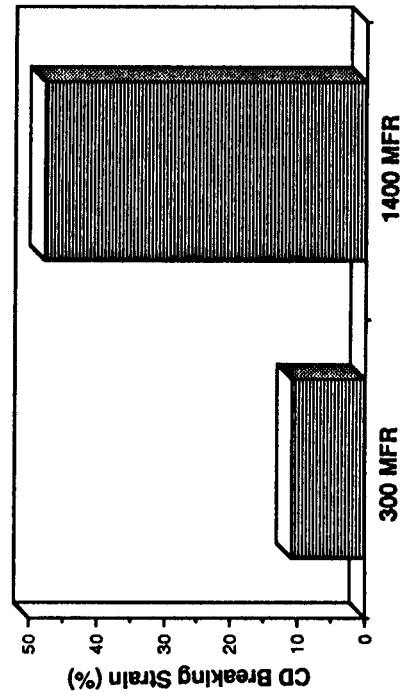


Figure 6. Effect of MFR on CD breaking elongation at 160 g/min throughput.

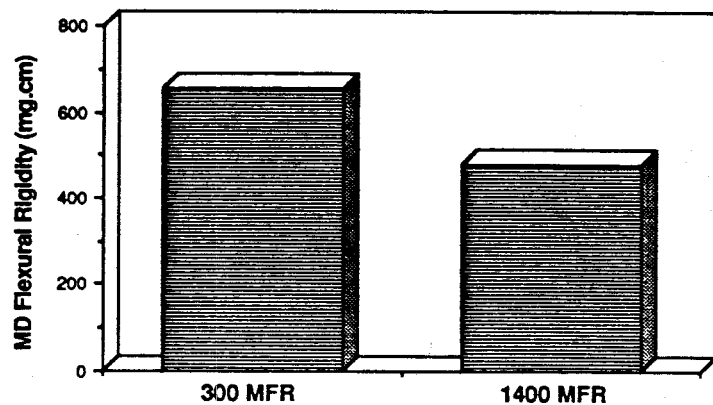


Figure 7. Effect of MFR on MD flexural rigidity at 160 g/min throughput.

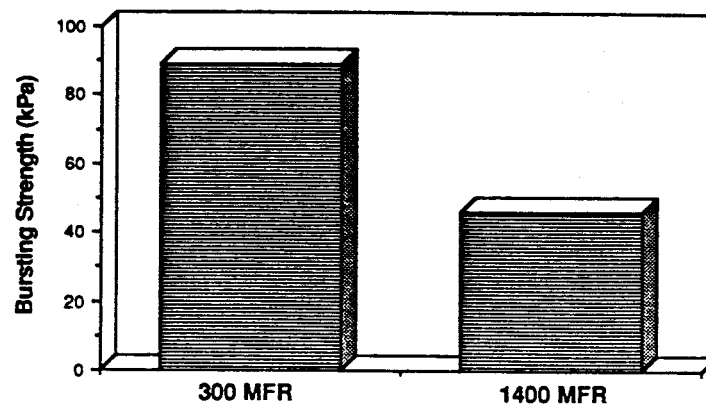


Figure 9. Effect of MFR on bursting strength at 160 g/min throughput.

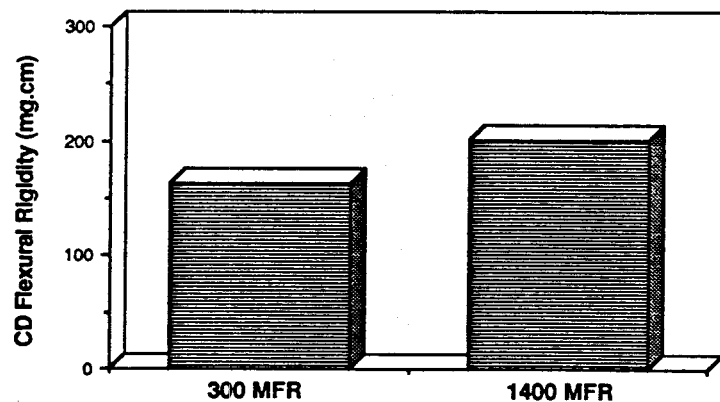


Figure 8. Effect of MFR on CD flexural rigidity at 160 g/min throughput.

increases [2]. This increased thermal bonding restricts the fiber shear movements resulting in more harsh and brittle characteristics. Figures 10 through 13 show that at higher throughput rate, the tenacity and breaking strain in both the directions remained relatively constant with increasing MFR. However, the breaking strain values were considerably lower than the values of 160 g/min throughput samples. This trend was also observed by Eaton et al [16], and may be due to more fiber entanglements (which these researchers believe occurs at higher throughput rate) and hence the strength will depend to a larger degree on mechanical bonding and on single fiber strength rather than thermal sticking. The flexural rigidity, as shown in Figures 14 and 15, remained relatively constant in both directions with increasing MFR. However, the flexural rigidity values were considerably higher than that of 160 g/min throughput samples. As shown in Figure 16, the bursting strength decreased dramatically at higher polymer throughput. The sharp drop in bursting strength values was probably due to the brittle characteristics of the higher throughput webs.

Initially a set of 5 samples was analysed for each web type. In order to generate a statistically reliable data base by testing a greater number of samples and thereby minimizing the possibility of making incorrect conclusions from a small sample size, an additional set of 25 samples was analysed for each web type. Figures 17 through 20 show the average stress-strain curves of both sets of 5 and 25 samples for each web type. Overall, there appeared to be a little difference in stress-strain properties of 5 samples versus 25 samples. However, the data for the set of 25 samples can be considered more reliable because of a slightly lower percentage coefficient of variation.

Table III shows that among the melt blown samples tested, the tensile stress values at which failure occurred ranged between 4.11 and 15.39 mN/Tex (0.047 and 0.18 g/den) and tensile strain at failure stress ranged

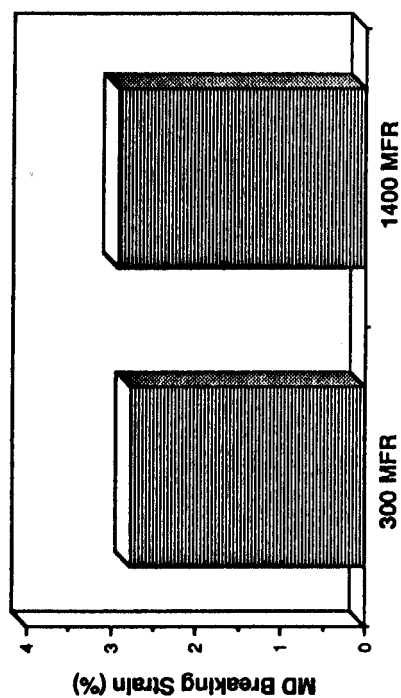


Figure 12. Effect of MFR on MD breaking elongation at 320 g/min throughput.

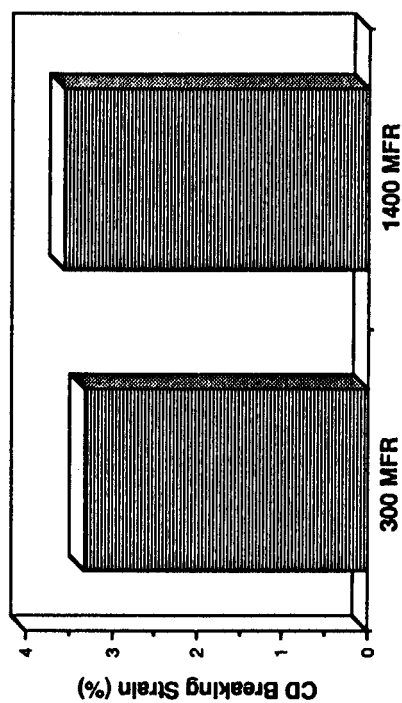


Figure 13. Effect of MFR on CD breaking elongation at 320 g/min throughput.

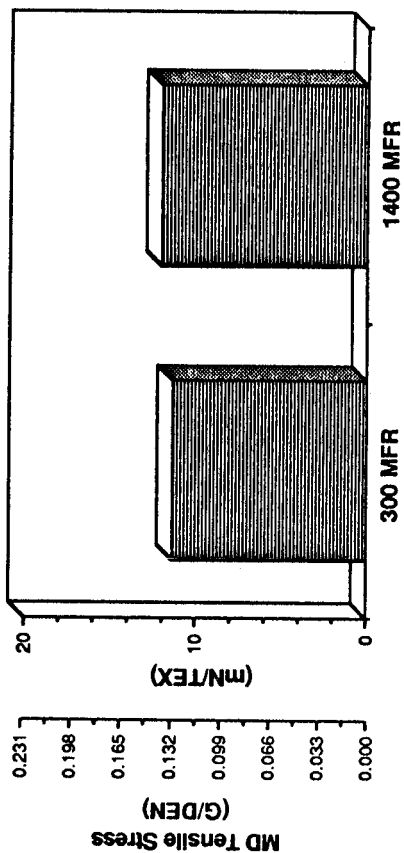


Figure 10. Effect of MFR on MD tenacity at 320 g/min throughput.

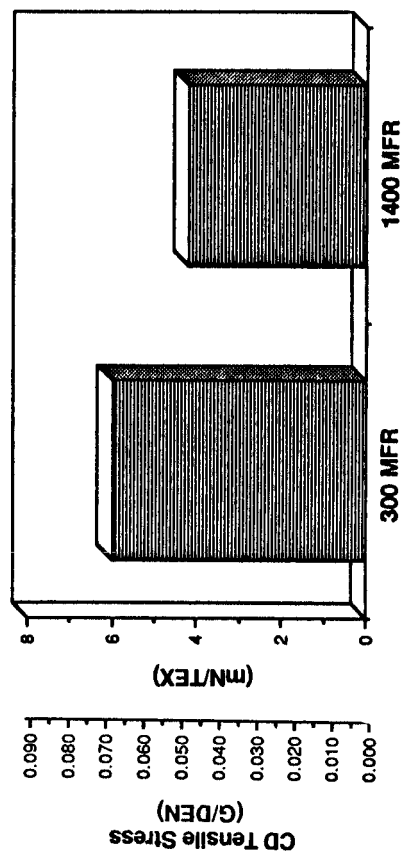


Figure 11. Effect of MFR on CD tenacity at 320 g/min throughput.

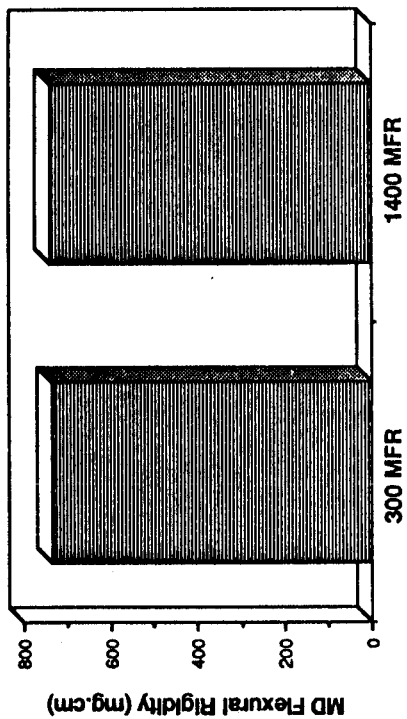


Figure 14. Effect of MFR on MD flexural rigidity at 320 g/min throughput.

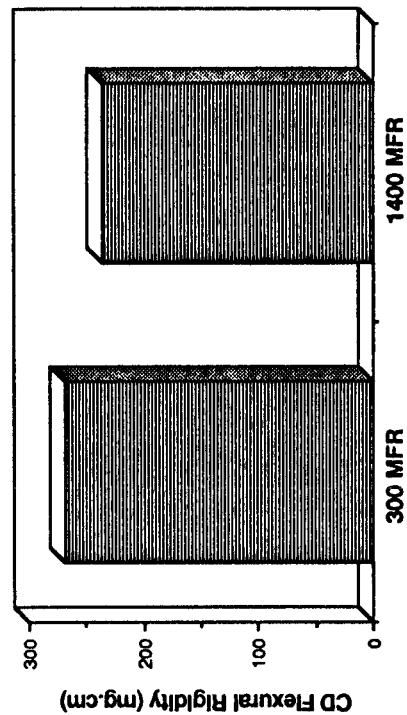


Figure 15. Effect of MFR on CD flexural rigidity at 320 g/min throughput.

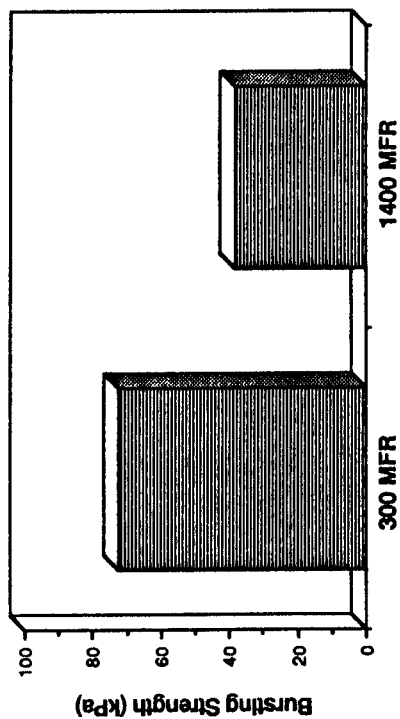


Figure 16. Effect of MFR on bursting strength at 320 g/min throughput.

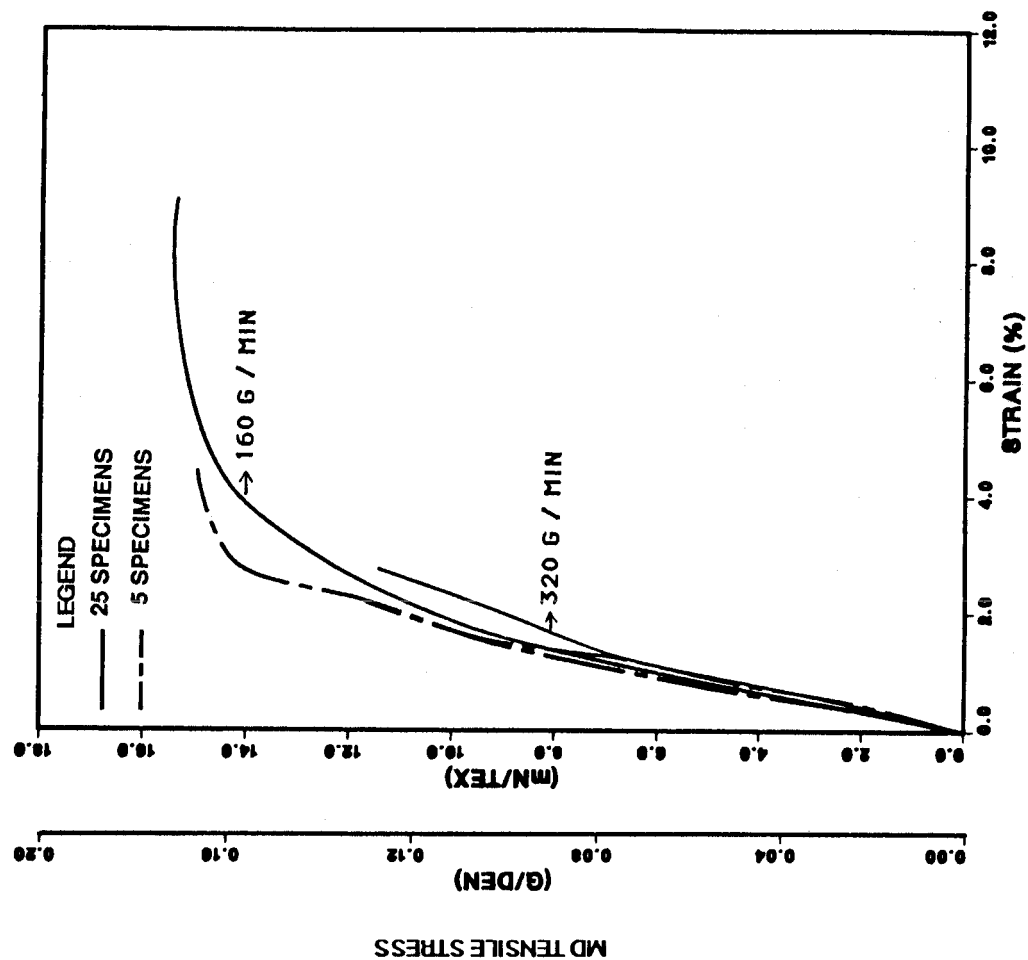


Figure 17. Average stress-strain curves of 300 MFR Polypropylene webs in MD at 160 g/min and 320 g/min throughput rates.

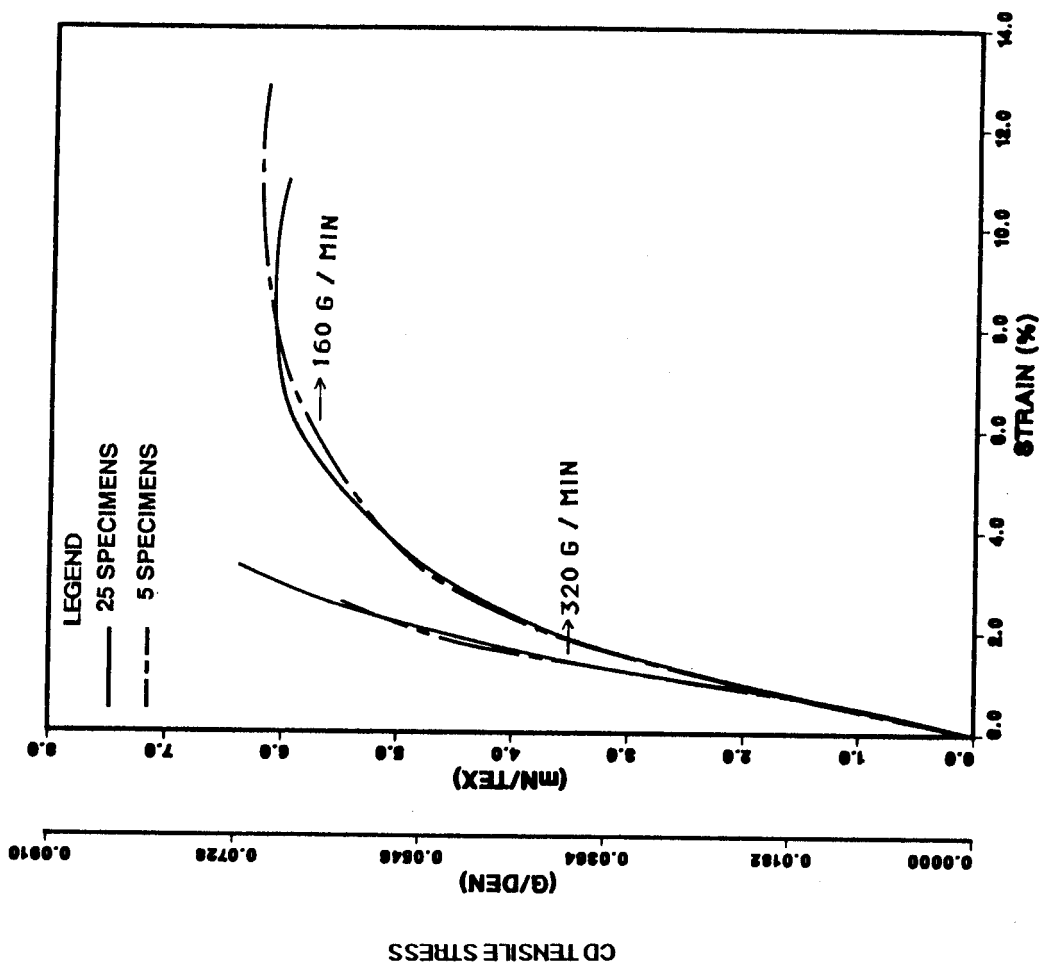


Figure 18. Average stress-strain curves of 300 MFR Polypropylene webs in CD at 160 g/min and 320 g/min throughput rates.

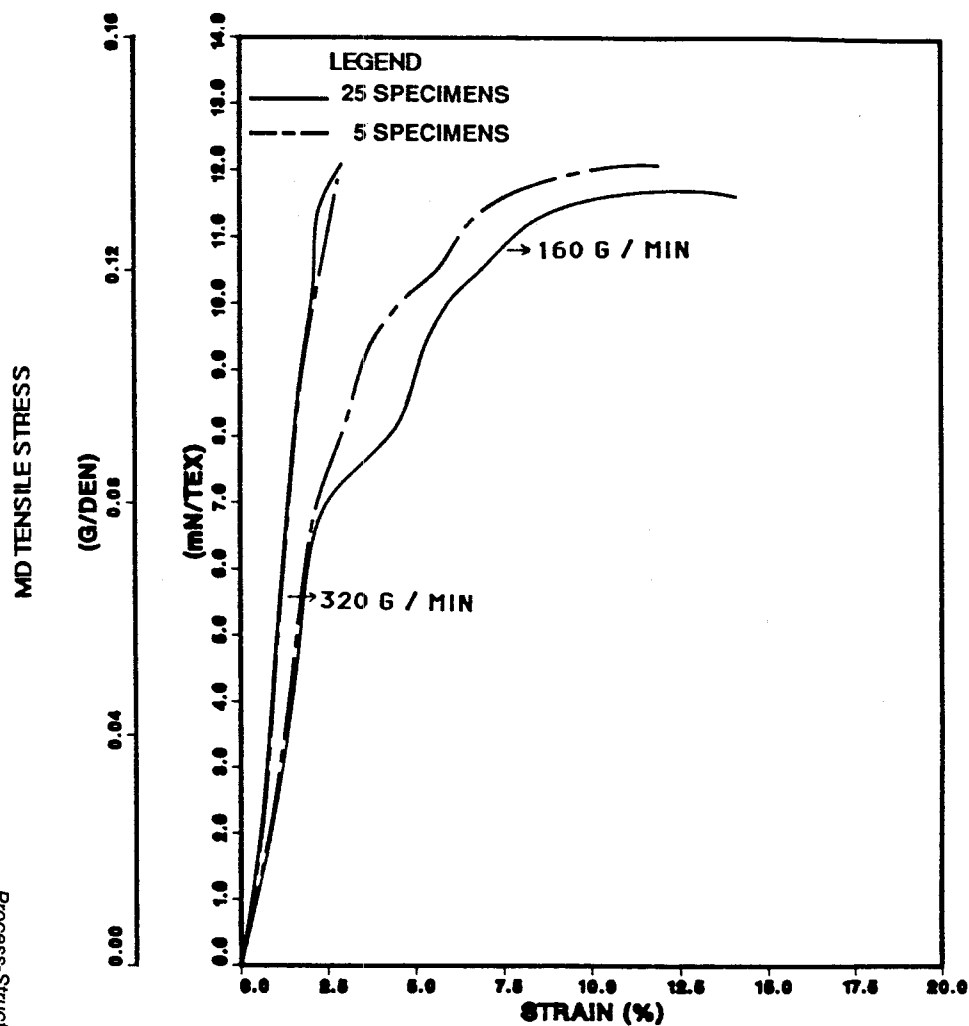


Figure 19. Average stress-strain curves of 1400 MFR Polypropylene webs in MD at 160 g/min and 320 g/min throughput rates.

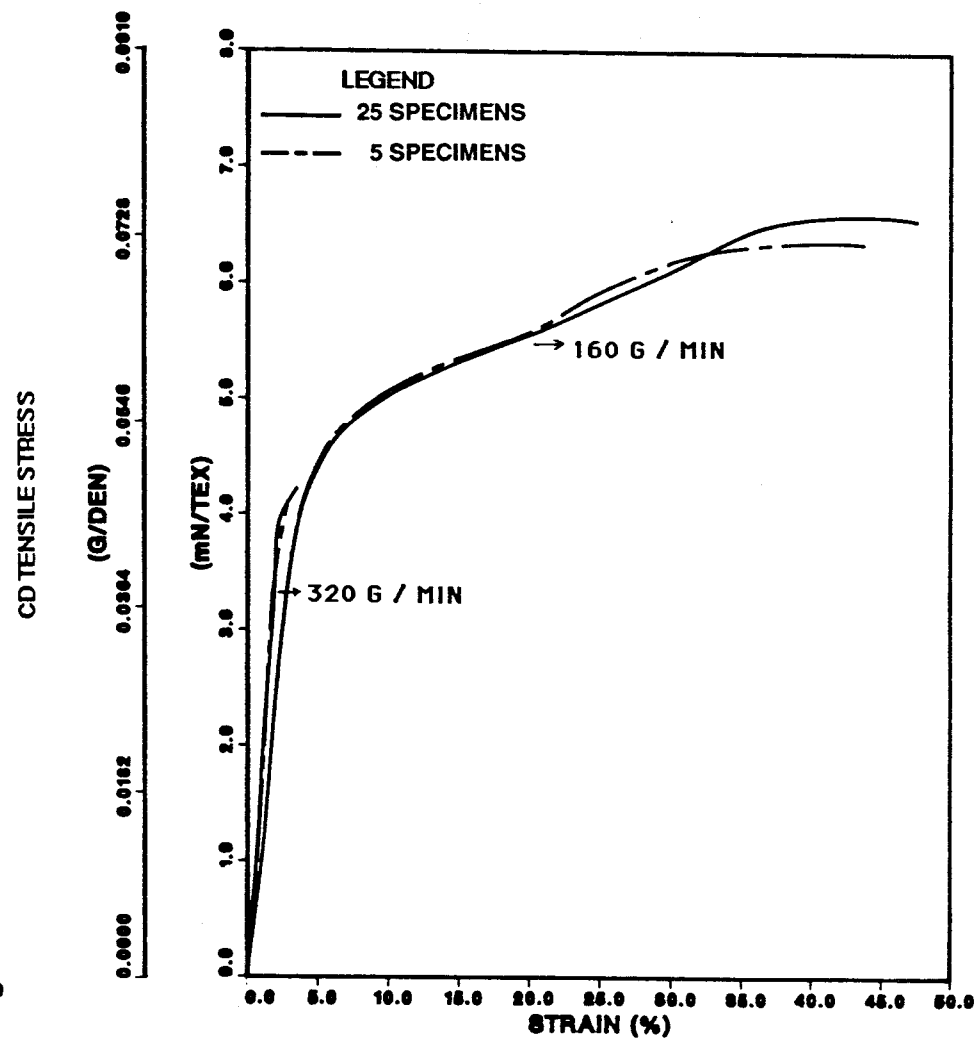


Figure 20. Average stress-strain curves of 1400 MFR Polypropylene webs in CD at 160 g/min and 320 g/min throughput rates.

between 2.17 and 46.61 %. The stress-strain curves were smooth, except in one or two cases where data discrepancy caused irregularity. The ductile behavior of webs resulting from low polymer throughput and brittle behavior of the high throughput melt blown webs were readily evident from the stress-strain curves.

The ductile behavior of low throughput web is believed to be due to the presence of a greater number of small diameter fibers and fewer fiber entanglements. On the other hand, the brittle behavior of high throughput web is believed to be due to the presence of a greater number of large diameter fibers, more fiber entanglements (effects of fiber entanglements are also being studied by UTK researchers), and geometric roughness of the fibers. At this point, it is indicated by the changes in MD/CD tensile strength ratio that the nature of random planar fibrous assemblies in the melt blown webs is dependent on throughput conditions.

Figure 21 shows the SEM micrographs of low and high throughput webs. It is evident from the micrographs that the low throughput rate results in a smaller diameter fibers and high throughput rate results in a larger diameter fibers. It is believed that the difference between the amount of polymer melt and the amount of air may be a critical factor for the final average fiber diameters in the webs. The large difference between the amount of polymer melt and air at low throughput conditions means more air and drag force per amount of polymer melt, which may have caused excessive draw-down of the fibers, resulting in a greater number of small diameter fibers in the web. While, the small difference between the amount of polymer melt and air at high throughput conditions means less air and drag force per amount of polymer melt, therefore less draw-down of the fibers, resulting in a greater number of large diameter fibers in the web.

The single fiber strength of 300 and 1400 MFR resin samples varied between 27 MPa and 34 MPa and the strain varied between 25% and 40%.

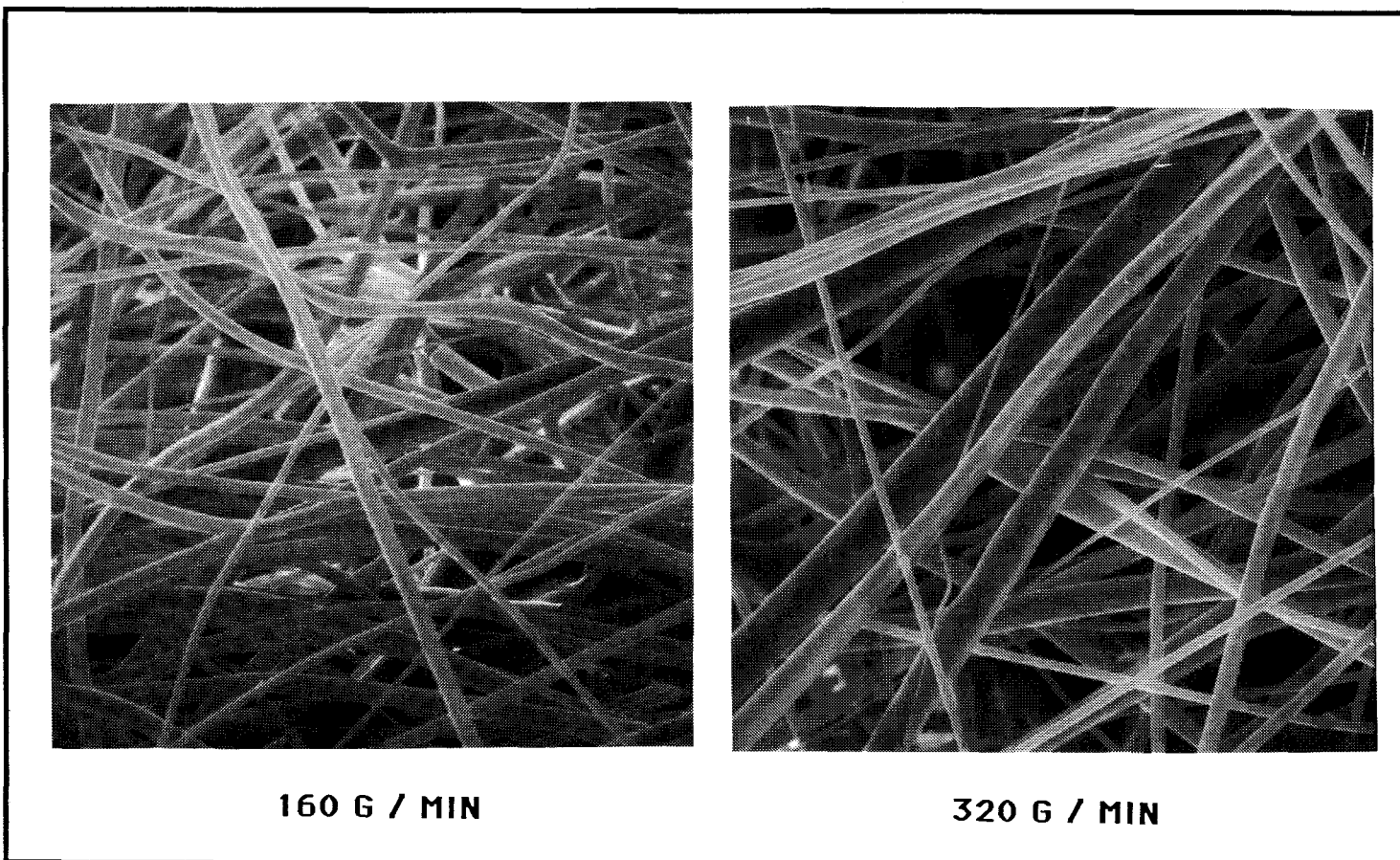


Figure 21. SEM photomicrographs of 300 MFR PP melt blown webs.

The single fiber strength was substantially higher than melt blown web strength and lower than high speed melt spun filaments [6]. This indicated that the lower melt blown web strength was possibly due to the fiber slippage in the web rather than individual fiber breakage.

Heat of Fusion

In order to study the structure-property-process relationship of the melt blowing process in terms of morphological properties, Differential Scanning Calorimetry (DSC) analysis was carried out. Table IV shows ΔH and percent crystallinity values for different MFR polypropylene resins and webs as a function of throughput rates. The DSC fusion curves for resins and webs are shown in Figures 22 and 23. There are no characteristic differences in ΔH values between low and high throughput webs. However, the difference in ΔH values between resin and web is noticeable, but only in 300 MFR samples. This was obvious because the higher molecular weight resin (300 MFR) degrades more than the lower molecular resin (1400 MFR) during processing. The ΔH values also indicated that there were no morphological changes, especially in percent crystallinity, between low and high throughput webs.

In higher throughput web samples the occurrence of two fusion endotherms was observed. The appearance of two endothermic maxima in the DSC fusion curves in some polymers is a point of interest for many investigators [9]. The double endothermic peaks exhibited by high throughput webs may be due to the existence of different unit cell dimensions or the presence of more than one morphological form of a single crystal structure.

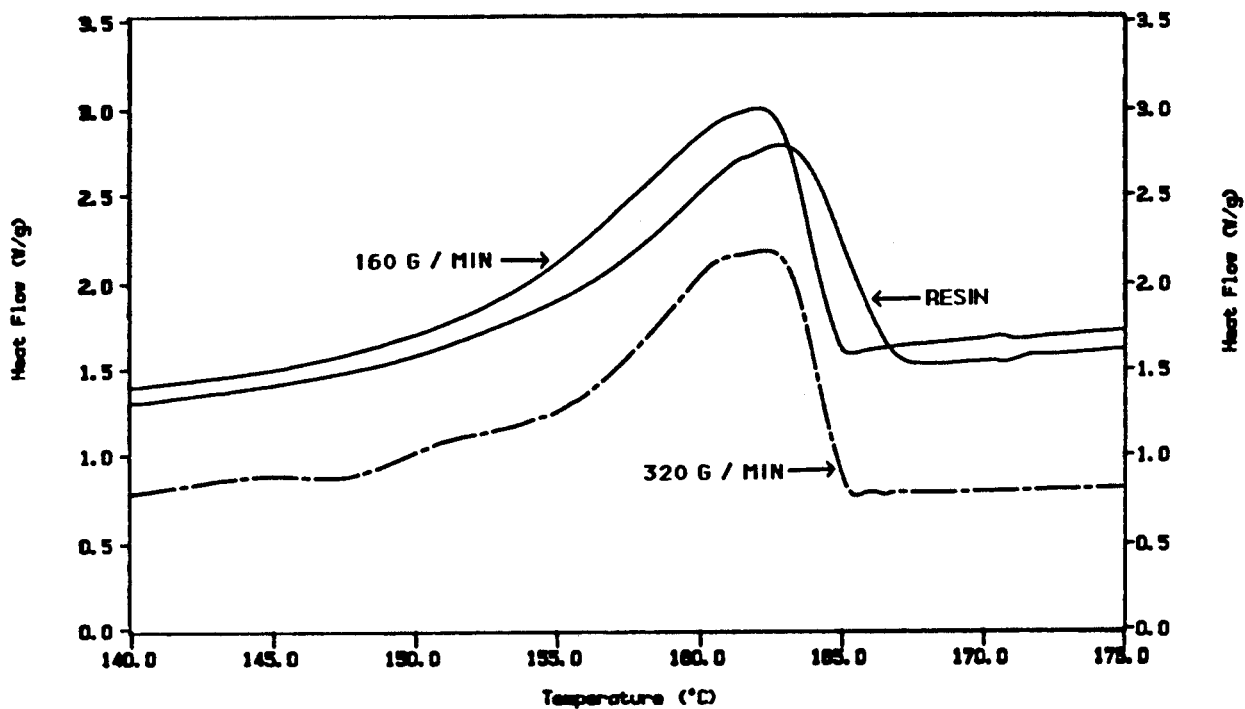


Figure 22. DSC curve of 300 MFR PP resin and webs at two throughput rates.

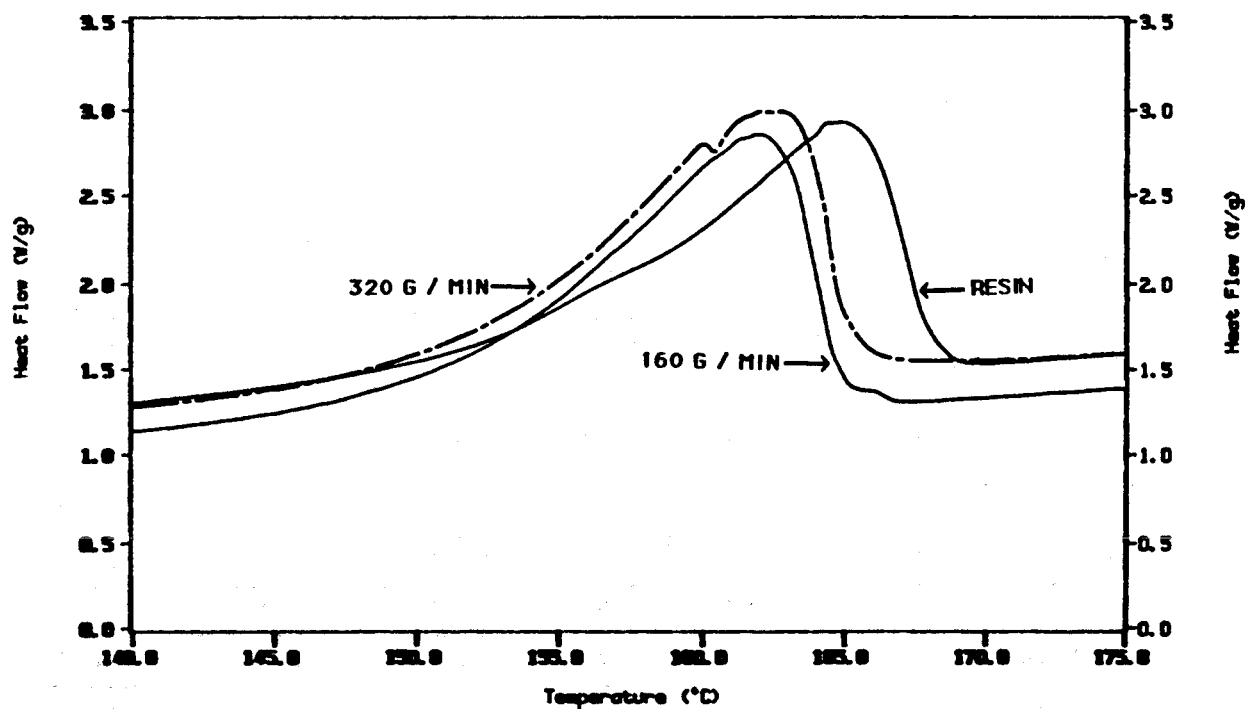


Figure 23. DSC curve of 1400 MFR PP resin and webs at two throughput rates.

Table IV
 ΔH and % Crystallinity values of different MFR PP resins and webs as a function of throughput rates

SAMPLES	300 MFR			1400 MFR		
Properties	Resin	160 g/min.	320 g/min.	Resin	160 g/min.	320 g/min.
Heat of Fusion (ΔH) J/g	78.15	83.58	84.85	85.20	87.81	85.86
Crystallinity (%)	53.34	57.05	57.92	58.16	59.94	58.61
Peak Temperature °C	162.38	161.56	162.01	165.70	161.76	162.67

Table V
Intrinsic Viscosity and Molecular Weight of Resins and Webs (through GPC Analysis)

Properties Sample Type	Melt Flow Rate (g/10 min)	Intrinsic Viscosity (dL/g)	Weight Average Molecular Weight M_w	Number Average Molecular Weight M_n	Polydispersity Index $\alpha = M_w/M_n$	Molecular Weight at Peak Max. (by GPC)
Resin	300	1.06	145486	55593	2.62	105729
160 g/min Web	300	0.92	123247	37696	3.27	83540
320 g/min Web	300	0.79	99733	46007	2.17	80332
Resin	1400	0.76	94552	45505	2.08	77239
160 g/min Web	1400	0.63	74882	31071	2.41	63471
320 g/min Web	1400	0.62	73044	29898	2.44	63471

Wide Angle X-Ray Diffraction

Wide angle X-ray scattering techniques were used to assess the morphological changes in the melt blown webs. Two-theta scans were taken over an angular range of 5° to 35° . Figure 24 shows the diffractograms of web samples. In the 300 MFR web samples the peaks of 110, 040, and 130 planes are sharper than the 1400 MFR web samples, thus indicating the possibility of having higher degree of crystallinity and larger crystallite size in the 300 MFR webs. The relative peak intensities were same among low and high throughput webs for each MFR resin, and therefore, the crystallinity in low and high throughput webs may be the same. The DSC results have shown the same trend. The high throughput web samples of 300 MFR resin showed a peak at 16.3° which is between 110 and 040 peaks. The occurrence of this peak may have been due to the presence of two types of morphological forms [9]. At this point of the research this finding is not conclusive and further investigation will be carried out.

Figure 25 shows WAXD flat film patterns of 300 MFR and 1400 MFR samples. There is no significant difference in any of the four patterns. Four or more relatively broad ring patterns with dark background around the origin indicated that the fibers are oriented in all directions in the webs with a moderate degree of crystalline monoclinic structure and possibly a low degree of amorphous smectic structure [6].

Gel Permeation Chromatography

Figures 26 and 27 show the GPC chromatograms of 300 and 1400 MFR resins and webs. Table V lists the properties measured through the GPC technique. It is evident from the data that the 300 MFR resin when processed at high throughput rate showed a noticeable decrease in

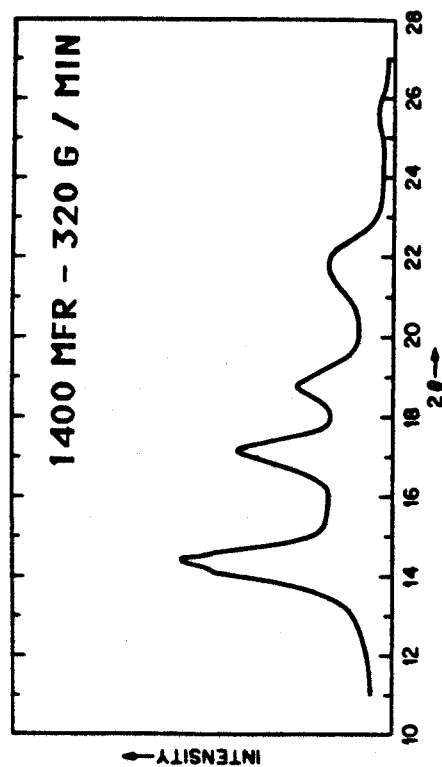
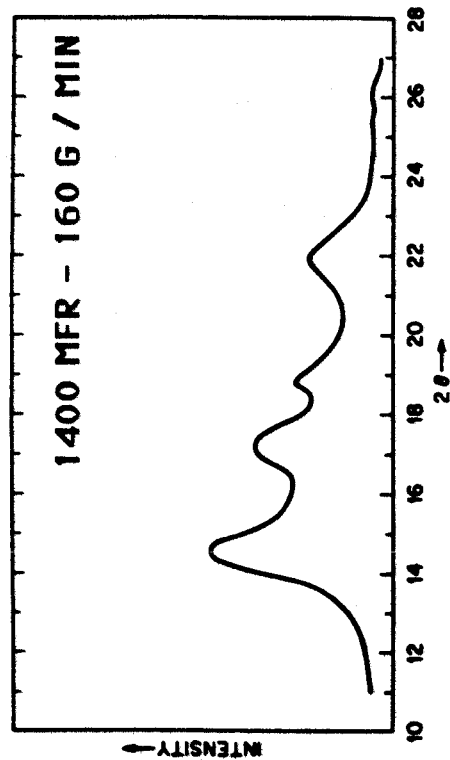
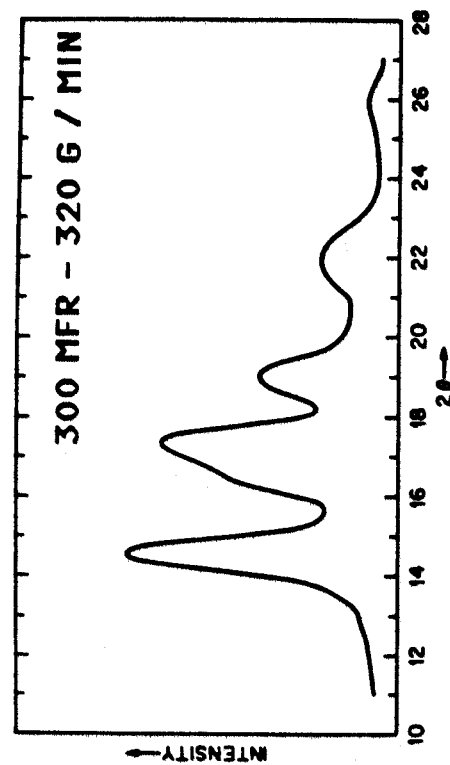
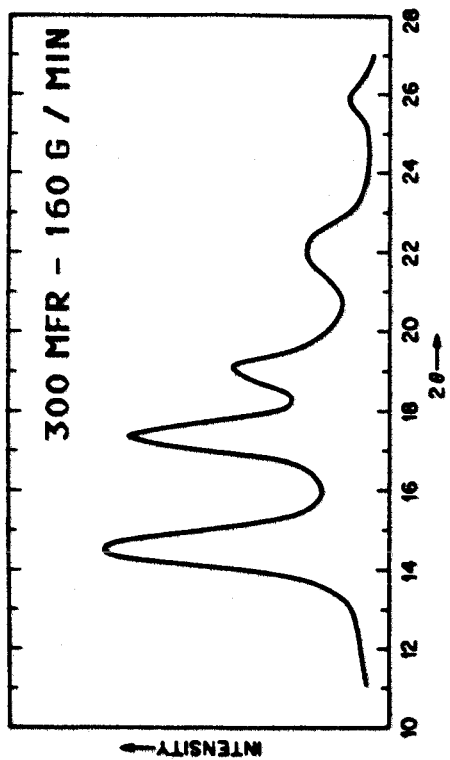


Figure 24. Wide Angle X-ray Diffractograms of polypropylene melt blown webs.

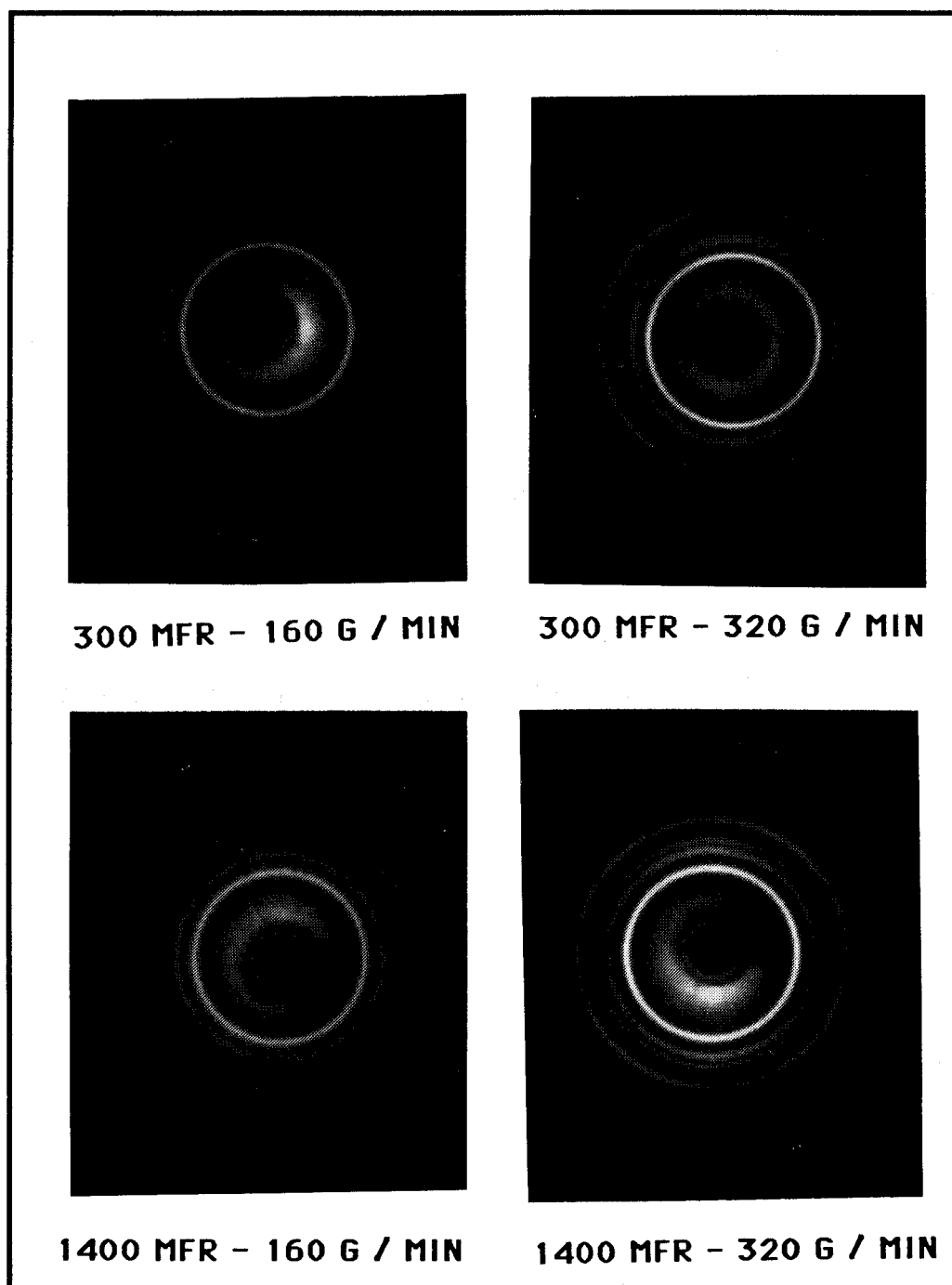


Figure 25. Wide Angle X-ray Diffraction film patterns of polypropylene melt blown webs.

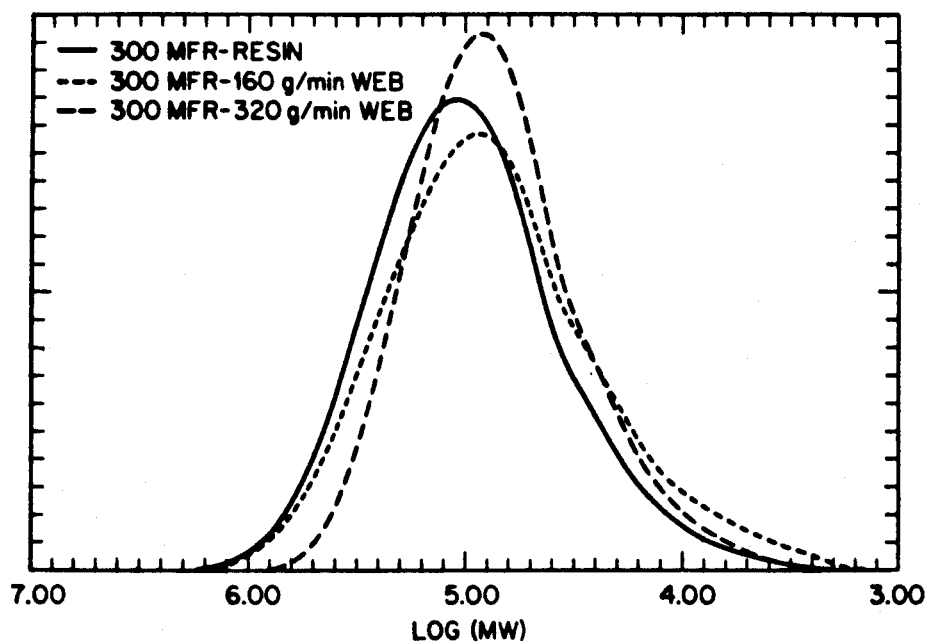


Figure 26. GPC chromatogram of 300 MFR polypropylene resin and webs at two throughput rates.

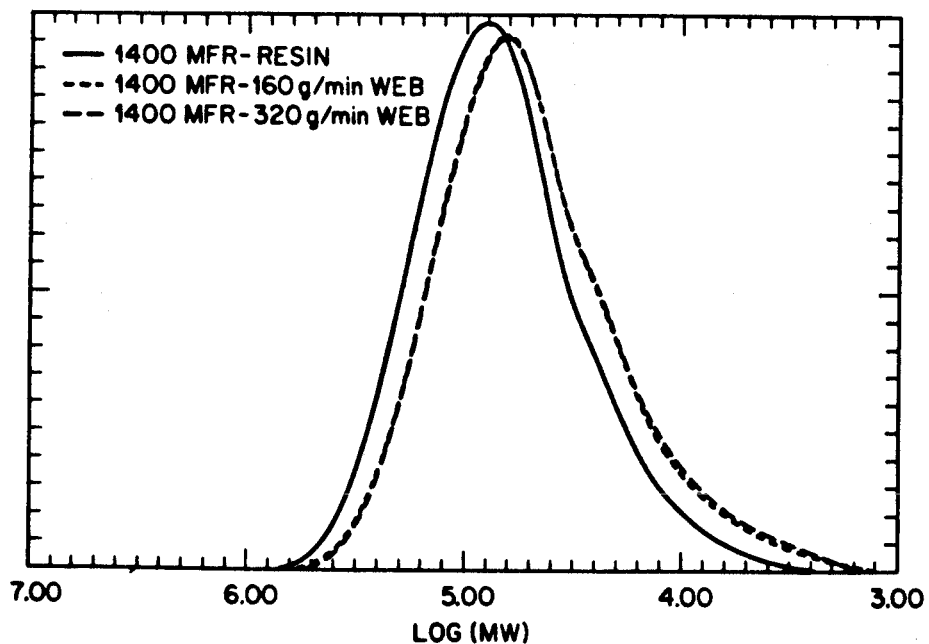


Figure 27. GPC chromatogram of 1400 MFR polypropylene resin and webs at two throughput rates.

molecular weight, intrinsic viscosity, and polydispersity index. On the other hand, the 1400 MFR resin when processed at high throughput rate showed a little change in molecular weight, intrinsic viscosity, and polydispersity index. The more molecular degradation in the 300 MFR resin samples than the 1400 MFR resin samples at high throughput conditions, may be attributed to the presence of long polymer chains, high shear rate, and high processing temperatures.

CONCLUSIONS

1. The effects of polymer throughput on the mechanical properties of polypropylene webs were significant, especially in terms of breaking strain. However, the effects on morphological properties were not as notable.
2. As the throughput rate increased, the web breaking strain decreased, possibly due to more thermal fusing of fibers, the presence of a greater number of large diameter fibers, and more fiber entanglement. The high throughput rate samples appeared to have a harsher hand and more brittle behavior.
3. The heat of fusion values indicated that there were small morphological changes, especially in percent crystallinity, between low and high throughput webs. In higher throughput web samples the occurrence of two fusion endotherms was observed, suggesting the possibility of having two different unit cell dimensions or more than one morphological form of a single crystal structure.
4. WAXD film patterns showed that the gross fiber orientation in the webs was random. WAXD diffractograms showed that the degree of crystallinity in the 300 MFR samples was higher than the 1400 MFR

samples, and the crystallinity was same among low and high throughput webs for each MFR resin.

5. At high throughput rate the molecular weight of the 300 MFR resin decreased notably, while that of the 1400 MFR resin remained relatively constant but still at a lower value than the 300 MFR resin.

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USE OF MONOFILAMENT MELT SPINNING AND ON-LINE MEASUREMENT TECHNIQUES FOR EVALUATION OF RESINS FOR SPUNBONDING AND MELT-BLOWING

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ABSTRACT

A method is described for evaluation of polymer resins for application in the spunbonding and melt blown nonwoven web processes. The method involves melt spinning of monofilaments, on-spinline measurements of diameter, temperature and birefringence profiles and characterization of the structure and properties of as-spun filaments. The on-line measurements provide a method for evaluating the resin's apparent elongational viscosity and crystallization kinetics in the presence of molecular orientation.

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INTRODUCTION

Spunbonding and melt-blowing are important commercial processes for producing nonwoven webs. The spunbonding process produces webs with good strength and tear resistance while the melt-blown process makes soft, breathable and drapable webs and fabrics whose properties are suitable for applications such as filter media, thermal insulation and sterile household, personal and medical supplies. Webs produced by both processes are often laminated into a composite structure to gain the advantages of each.

In both processes a thermoplastic polymer is extruded through a die containing small diameter spinneret holes. In the spunbonding process the filaments are attenuated by air drag applied at a distance from the spinneret holes so that the development of filament structure is quite similar to that occurring in multifilament melt spinning (1). This allows a reasonably high level of filament orientation to be developed and incorporated into the web after lay-down and thermal bonding.

In the melt-blown process a stream of high velocity hot air is directed on the filaments as they emerge from the spinneret. This air produces filament attenuation, entanglement and transport to a collection device where the web is laid down (2,3).

The webs produced by the melt-blown process typically contain filaments whose diameters are in the range 1-15 micrometers (4,5). The webs owe much of their softness and filtration capacity to the presence of these fine denier filaments and the way in which they entangle and bond to each other. Larger average filament diameters and/or too much interfilament bonding leads to stiffer fabrics (resembling films) with lower specific surface area and poorer filtration capacity. Other properties such as web strength depend on the internal structure (orientation and crystallinity) of the filaments as well as the interfilament bonding (5).

Although the spunbonding and melt-blowing processes both have several variables that can be used to adjust the properties of the resulting webs, the nature and

properties of the resin influence both the processing window and the final web properties. In addition to basic spinnability of the resin, two of the most significant of these resin properties are elongational viscosity and crystallization kinetics under molecular orientation. The elongational viscosity influences the spinnability of the polymer and controls the level of stress developed in the molten filaments for a given set of kinematic processing conditions. For a given force applied by air drag or other means, the elongational viscosity of the polymer controls the rate of filament drawdown and fiber deniers that can be achieved. The level of stress developed controls the molecular orientation developed in the melt and ultimately the level of orientation frozen into the filament after stress-induced crystallization occurs. Stress-induced crystallization kinetics determine where and under what conditions crystallization occurs in the process and the morphology and properties of the resulting filaments.

Elongational viscosity is difficult to measure by established techniques, especially for typical melt blowing resins due to their relatively low molecular weights and resulting low viscosities. At the present time there are no established laboratory methods for measuring stress-induced crystallization kinetics. These problems have lead us to investigate the potential of monofilament melt spinning combined with on-line measurement techniques for evaluating filament diameter, temperature and birefringence profiles as a method to obtain comparisons between differing resins. An advantage of this approach is that melt spinning is a relatively simple, well understood process that can be carried out under controlled conditions in the laboratory. It also requires relatively small amounts of resin compared to the spunbonding or melt blowing process.

We outline here the basics of the approach and give some results for a series of three polypropylenes.

EVALUATION OF APPARENT ELONGATIONAL VISCOSITY

An "apparent elongational viscosity" of the resin can be evaluated as a function of temperature from the on-line measurements made on the running spinline. The required data are measurements of filament diameter and temperature as a function of distance from the spinneret coupled with a measurement of the spinline tension carried out at a position near the filament take-up device. The procedure discussed here was first described by George and Deeg (6); the first successful application to experimental data was carried out in our laboratory for the case of nylon-6 by J. H. Bheda (7). The procedure is outlined in Figure 1. The experimental diameter and temperature profiles and the tension at the bottom of the spinline are first used to compute the rheological force as a function of position along the spinline. The required calculation is an integration of the form

$$F_{rheo}(z) = F_L - \int_z^L \rho_{air} C_d V^2 \pi D dz - W[V(L) - V(z)] + \int_z^L \rho g \pi (D^2/4) dz \quad (1)$$

Here W is mass throughput, V is velocity, F_L is the take-up tension for a spinline of length L . The density of the polymer melt, ρ , must be known as a function of temperature, and the calculation also requires a knowledge of the air drag coefficient, C_d , that determines the magnitude of the force component due to the air drag on the rapidly moving filament. The latter quantity has been evaluated by various investigators (8-12) and is known sufficiently accurately for take-up velocities less than 2000 m/min, where the air drag contribution is relatively small, so that little error is introduced by errors in the air drag coefficient (7).

Next the diameter and temperature profiles are fitted to smooth functions through regression models and the velocity profile is calculated using the continuity

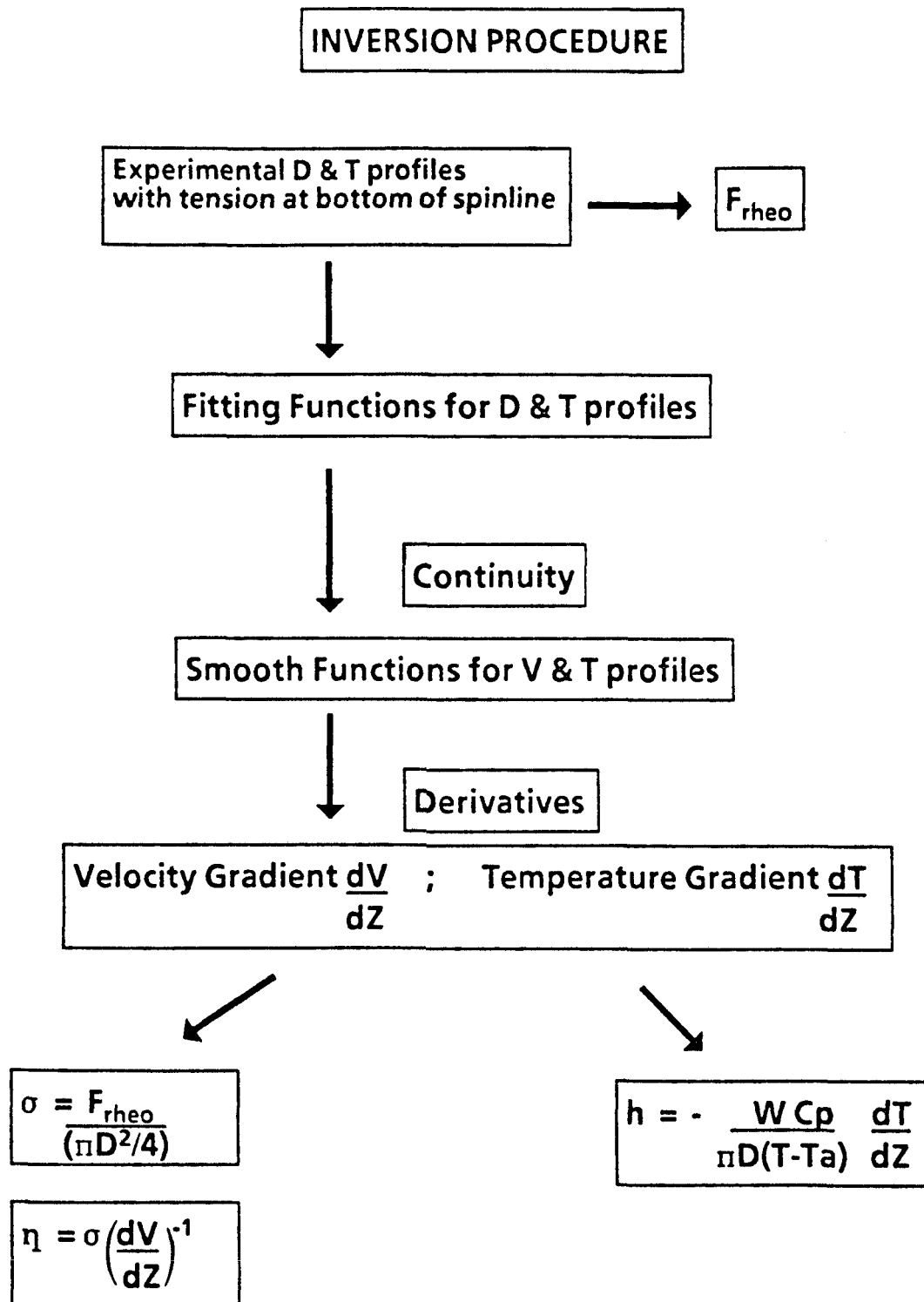


Figure 1. Schematic of the inversion procedure for evaluation of apparent elongational viscosity and heat transfer coefficient.

equation. The smooth velocity and temperature profiles are differentiated to obtain the velocity and temperature gradient profiles along the spinline. Combination of the rheological force and velocity gradient allows a calculation of the apparent elongational viscosity at each point along the spinline.

This approach would give the actual elongational viscosity if the polymer exhibited a Newtonian viscosity over the entire temperature range of measurement. In practice, most polymers exhibit non-Newtonian viscoelastic behavior and their viscosities depend on their deformation histories as well as their instantaneous deformation rates. However, for the case of high melt flow rate, low viscosity polymers, the Newtonian approximation can be used, with caution, to obtain apparent elongational viscosities that can be used for comparison of various resins.

As noted in Figure 1 the temperature gradient profiles can be used to evaluate the heat transfer coefficient for the running spinline.

In general, this approach can only be applied in the absence of crystallization occurring in the spinline or to portions of the profiles that are not complicated by crystallization. In the absence of crystallization we expect that the apparent elongational viscosity and heat transfer coefficient should follow equations of the form

$$\eta = \text{const} \exp \left(\frac{E_n}{T + 273} \right) \quad (2)$$

$$h = \text{const} (V/A)^p \quad (3)$$

Equation 2 is the Arrhenius temperature dependence expected for viscosity and Equation 3 is of the form suggested by expressing the heat transfer relation of Kase and Matsuo (13) in terms of spinning variables. Here V is the local filament velocity and A is its cross sectional area.

STRESS-INDUCED CRYSTALLIZATION KINETICS

During processing of semicrystalline polymers, crystallization almost always takes place in the presence of molecular orientation and under nonisothermal conditions. Evaluation of the relative behavior of different resins for their crystallization behavior under these conditions is one of the most difficult problems of polymer science. In the melt spinning process it is possible to crystallize resins under a range of molecular orientations. The level of molecular orientation in the melt just prior to crystallization can be controlled by the level of stress developed in the molten filament. Because of this we have found that the melt spinning process provides an answer to the problem of evaluation of the stress-induced crystallization behavior of different resins.

Our approach requires the use of a mathematical model of the melt spinning process which includes a detailed description of the development of crystallization under molecular orientation. The details of this model and the fundamental arguments leading to the development of the required crystallization kinetics equations are given elsewhere (14,15).

The relation of crystallization kinetics under stress to quiescent crystallization and an approach for their evaluation are illustrated in Figure 2. The quiescent crystallization kinetics are first evaluated by the usual techniques (eg. differential scanning calorimetry, dilatometry, etc.) or from information in the literature. This will involve evaluation of the constants C_1 , C_2 and C_3 in the expression for quiescent crystallization rate. The stress-induced crystallization rate is a function of both temperature, T , and the level of molecular orientation in the melt, f_a . The equation for stress-induced crystallization involves an additional constant C that determines the magnitude of the effect of molecular orientation in the melt, f_a , on the crystallization rate. The value of C can be determined from on-spinline measurements of diameter, temperature and birefringence profiles. This is done by adjusting the

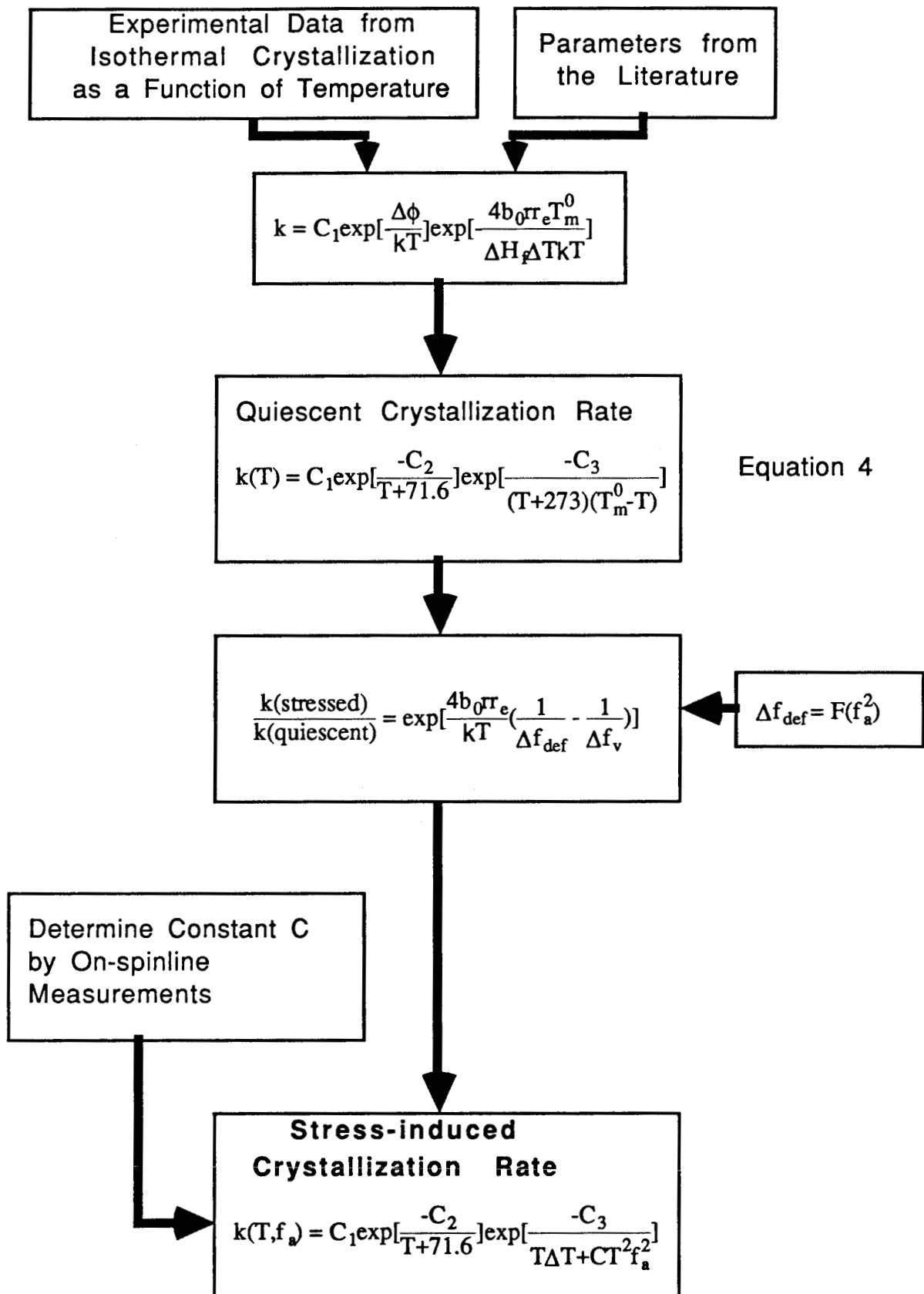


Figure 2. Scheme for evaluating stress-induced crystallization rate from quiescent crystallization and melt spinning data.

parameter C until the mathematical model provides computed profiles in agreement with experiment.

The development of crystallinity during processing can be expressed in terms of the modified Avrami equation (16-18) shown in Figure 3. This shows clearly that the crystallinity, X_t , is determined by the interaction of stress-induced crystallization rate and cooling rate. High cooling rates tend to reduce the fraction of the sample crystallized by reducing the amount of time available for crystallization in the temperature range where crystallization rate is significant. Increasing molecular orientation increases the crystallinity by increasing the crystallization rate, $K(T, f_a)$. The maximum value of the product $K(T, f_a) \times (dT/dt)^{-1}$ is a measure of the tendency of the resin to develop crystallinity during specific processing. We shall call this product the "dynamic crystallization kinetic factor." It can also be evaluated from the melt spinning results and used for adjustment of process conditions, resin selection or process improvement, Figure 3.

EXPERIMENTAL

Materials

Although the techniques described in this paper can be applied to a wide range of materials, example results are described for three polypropylenes supplied by Exxon Chemical Company. The characteristics supplied by Exxon for these resins are reported in Table 1. The molecular weight averages were measured by gel permeation chromatography (GPC) and the intrinsic viscosities were measured in decalin at 135°C.

Melting Spinning

A Fourné Associates single screw extruder and spinning head were used for melt spinning. Details are described elsewhere (7,14,15). A gear pump controlled the flow rate through a single hole spinneret. The extruder and spinning head is mounted on a large column so that it can be readily positioned for easy access to the spinline during

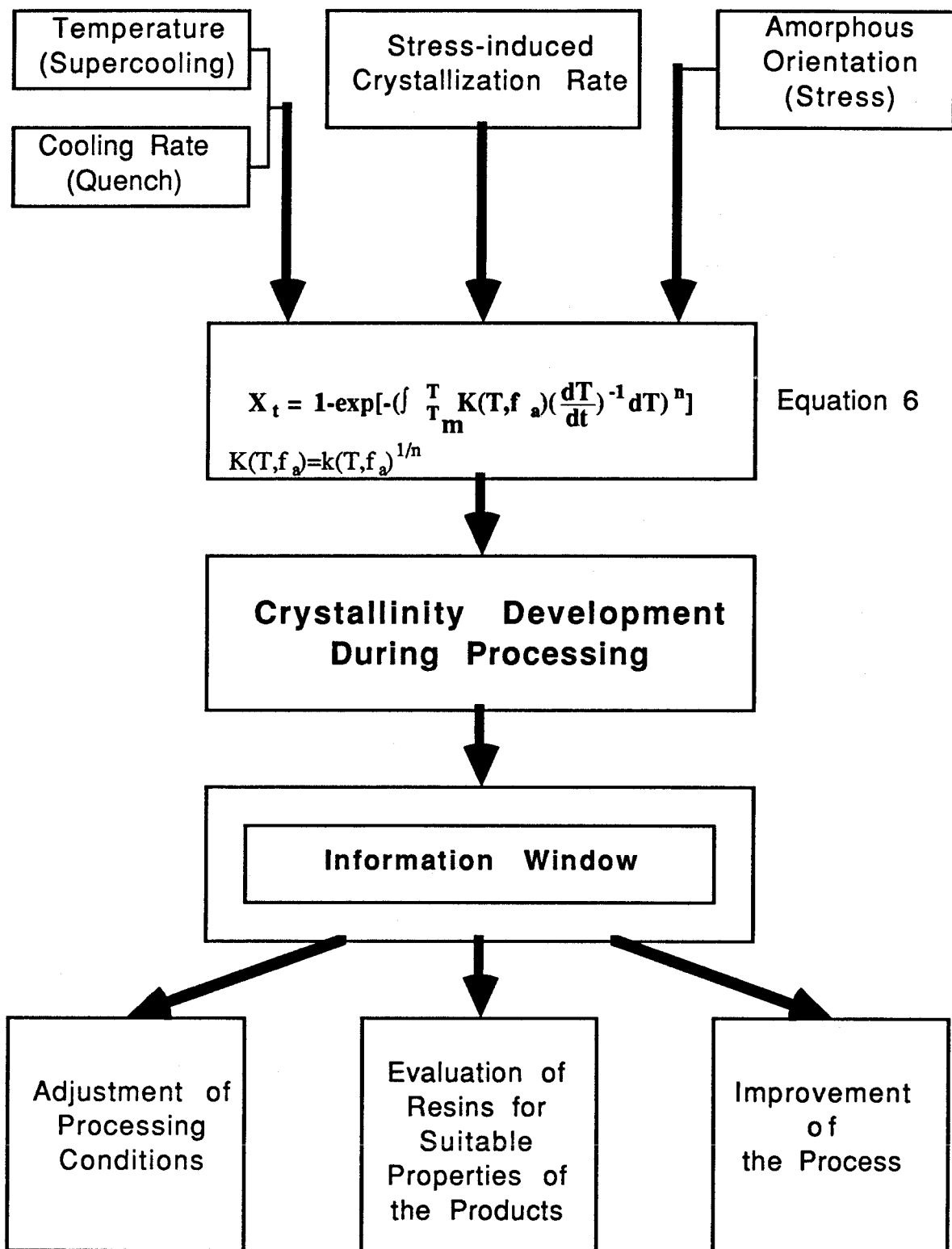


Figure 3. Factors involved in computation of crystallinity during processing and its use for for process improvement.

TABLE 1
As-Received Polymer Properties

Exxon code	Melt index (g/10 min)	Our code name	Intrinsic viscosity (dL/g)	Weight average molecular weight M_w (by GPC)	Number average molecular weight M_n (by GPC)	Poly-dispersity $\alpha = M_w/M_n$	Molecular weight at peak max (by GPC)
3014, lot 40435	12	E-012	1.511	238,000	50,010	4.76	141,180
11852-011-001 (control)	35	E-035	1.213	170,450	78,940	2.16	147,180
3145, lot 40611	300	E-300	0.929	124,400	43,590	2.85	89,770

TABLE 2
Melt Spinning Processing Conditions

Extrusion Temperature	230°C (PP-E-012) 210°C (PP-E-035) 190°C (PP-E-300)
Spinline Length	2.5 meters
Mass Throughput	1.68 g/min
Die Diameter	0.762 mm
Take-up Velocity	1000-5000 m/min

on-line measurements. Filament drawdown can be carried out with a winder or an air drag device. The latter uses pressurized air to produce a suction force on the filament at the entrance to the device. The take-up velocity can be varied by adjusting the pressure of the air supply.

The process conditions used in the present research are summarized in Table 2.

On-line Measurements

1. Diameter Measurement

The on-line diameter measurement was carried out by using a Zimmer system supplied by Zimmer OHG, West Germany. The instrument is based on a non-contact electro-optic back illumination principle permitting measurements on both opaque and semitransparent materials. An IBM-XT microcomputer was interfaced with the Zimmer system to collect and store data. Two thousand diameter values were collected by the computer within twenty seconds at each position at which diameter was measured. A statistical program was used to process the data collected.

2. Birefringence Measurement

On-line birefringence of filament was determined using an Olympus polarizing microscope and an Olympus 4 order Berek compensator to measure the optical retardation. The microscope was mounted on a movable stand which can be moved parallel to the spinline. The retardation was calculated from the compensator reading and the birefringence was calculated from the following equation

$$\Delta N = R/D \quad (7)$$

where ΔN is fiber birefringence, R is retardation reading, D is fiber diameter which was measured by the Zimmer instrument at the same spinline position as the birefringence measurement.

3. Temperature Measurement

The on-line temperature of the filament was measured using a Barnes Infrared Microscope model RM-2B. A null-balance technique was used wherein the temperature of the spinline was compared to the temperature of a small heater at a known temperature. The details have been previously described (7,19,20).

4. Tension Measurement

The tension in the spinline was determined using a Rothchild Electric Tensionmeter model INTEG R-1198 measuring head. The instrument was connected to an IBM-XT personal computer. Two thousand readings were collected by the computer within 20 seconds at each measurement condition and an average value of tension was calculated.

RESULTS AND DISCUSSION

Structure of As-Spun Filaments

The density of the as-spun filaments of each resin as a function of spinning conditions is presented in Figure 4. It is observed that the density varies widely with resin and spinning conditions. These variations can be interpreted as resulting from differences in crystallinity or molecular order. X-ray diffraction patterns for selected samples are shown in Figure 5. The birefringence, which is a measure of average molecular orientation, is shown in Figure 6 and the crystalline orientation measured by x-ray diffractometry is shown in Figure 7.

The results shown in Figures 4 through 7 indicate large differences of behavior between the three resins studied. The data in Figures 4 and 5 show that the higher molecular weight, lower MFR resin develops a high level of crystallinity and a monoclinic crystal structure at all spinning speeds studied. The 300 MFR resin develops a relatively low level of "crystallinity" and a structure that contains

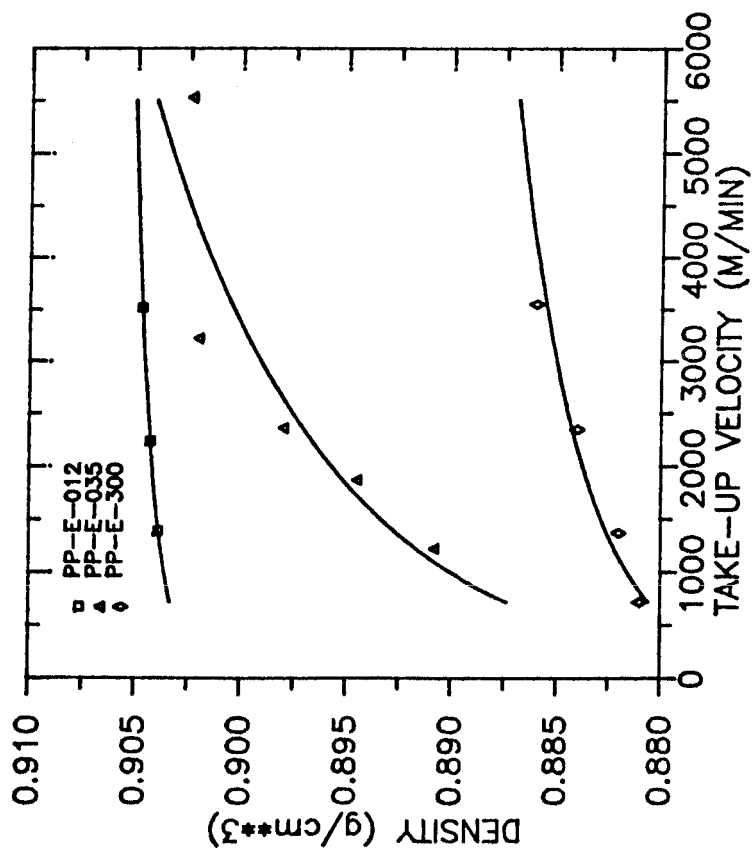
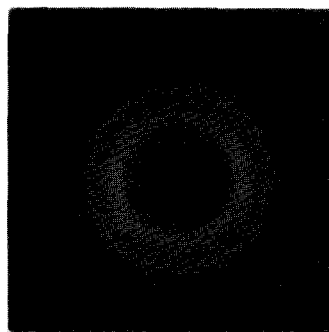
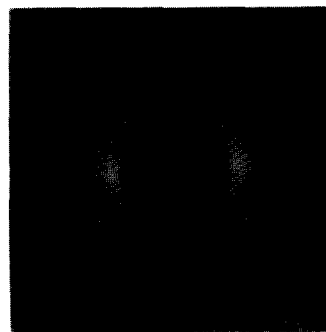


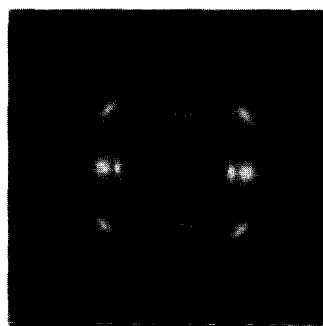
Figure 4. Density of as-spun fiber as a function of take-up velocity.



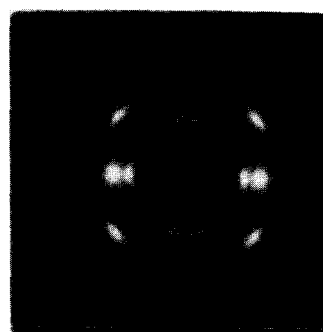
a



b



c



d

Figure 5. X-ray diffraction patterns of selected as-spun filaments. Take-up velocities were (a) 1220 m/min, (b) 2360 m/min, (c) 3220 m/min, (d) 5530 m/min.

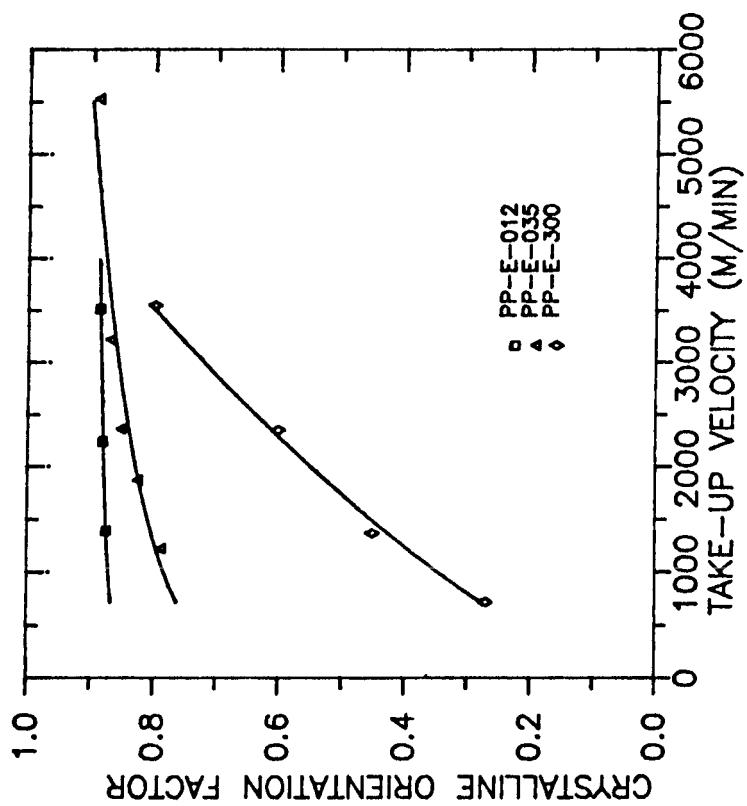


Figure 7. C-axis crystalline orientation factor as a function of take-up velocity.

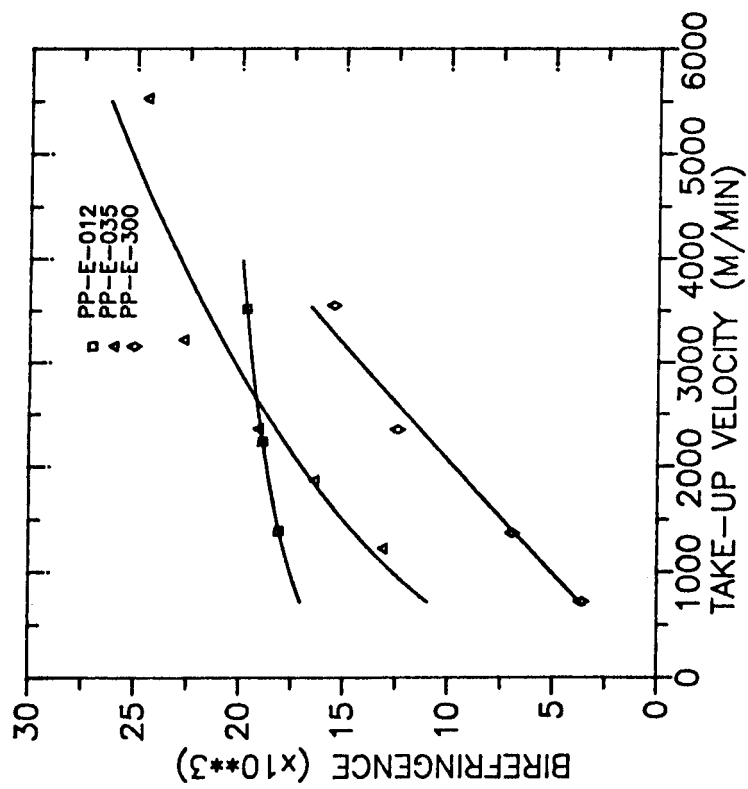


Figure 6. Birefringence of as-spun fiber as a function of take-up velocity.

primarily smectic phase. The structure of the 35 MFR resin varies widely with spinning conditions.

The results to be presented in subsequent sections show that the differences of molecular orientation can largely be traced to the major differences in elongational viscosities of the resins with resulting differences in stress and molecular orientation developed at the beginning of and during crystallization. The differences in crystalline order result largely from differences in crystallization kinetics caused by the molecular orientation and consequent differences in the temperature at which the structure is developed during spinning.

On-line Measurements

Examples of diameter, temperature and birefringence profiles measured by on-line techniques are shown in Figure 8 for the 35 MFR resin. The diameter draws down rapidly in the upper part of the spinline and then levels off at its final value as the filament solidifies. Drawdown is more rapid with increased take-up velocity and solidification occurs closer to the spinneret. The temperature profiles show that temperature drops smoothly in the upper part of the spinline, but as crystallization occurs the profile exhibits a "plateau" due to the heat of crystallization being given up. Further, as take-up velocity increases, crystallization occurs at higher temperature. This effect is clearly due to the increased crystallization kinetics resulting from the increased spinline stress and associated molecular orientation. The birefringence profiles rise slowly prior to crystallization due to increased molecular orientation in the amorphous melt. As crystallization occurs the birefringence rises more rapidly. This is caused by oriented nucleation and growth of the crystalline phase (21). Since the crystals are nucleated by molecules having the highest orientations, the crystals tend to grow with much higher average orientations than the amorphous melt. The distribution of crystal orientations and, hence, the final

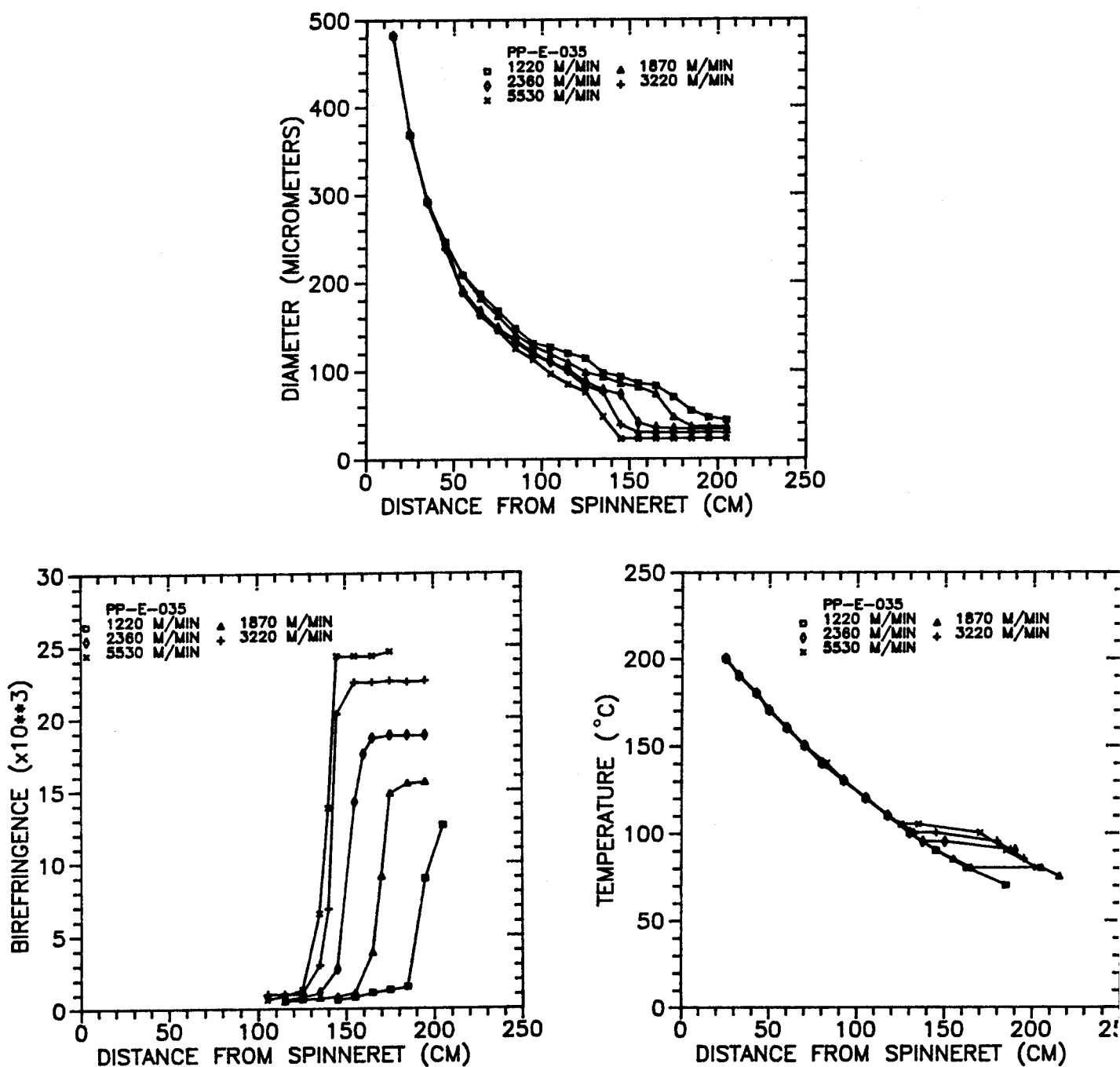


Figure 8. On-line measurements for the 35 MFR resin. (a) diameter profiles, (b) birefringence profiles, (c) temperature profiles.

birefringence of the filaments depends on the average level of orientation in the melt as crystallization occurs (22,23).

Figure 9 shows diameter and birefringence profiles for the other two resins studied. Comparisons among the profiles for the three resins show that drawdown and crystallization occur farther from the spinneret with increasing resin MFR. This also implies that for a given take-up velocity crystallization occurs at lower temperature with increasing resin MFR. This effect leads to the lower "crystallinity" and relative increase of smectic phase with increase of resin MFR.

Apparent Elongational Viscosities

The apparent elongational viscosities of the three polymers were measured using the inversion technique described above. In order to avoid complications from crystallization, the on-line diameter and temperature profiles used in the inversion were for spinning at take-up velocities in the range 800 to 1000 m/min where crystallization could be postponed until well down the spinline.

Figure 10 shows the resulting apparent elongational viscosities plotted on logarithmic coordinates as a function of reciprocal temperature. The straight lines through the results represent the best fit of the results to the Arrhenius temperature dependence expressed by Equation 2. The resulting expressions are:

For PP-E-300

$$\eta = 160 \exp\left(\frac{1960}{T + 273}\right) \quad (\text{units are poise})$$

For PP-E-035

$$\eta = 551 \exp\left(\frac{1950}{T + 273}\right)$$

For PP-E-012

$$\eta = 1478 \exp\left(\frac{1650}{T + 273}\right)$$

The activation energy for viscous flow is observed to be nearly the same for all three polypropylenes. The preexponential constant clearly shows molecular weight

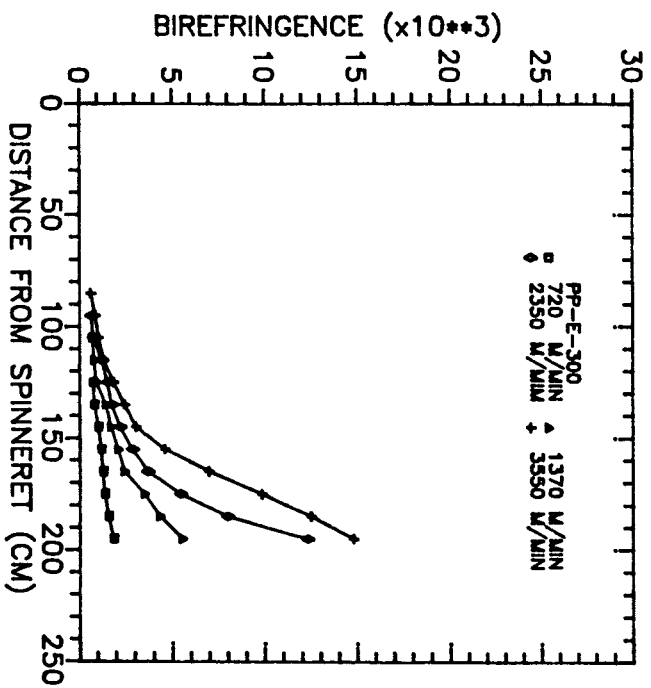
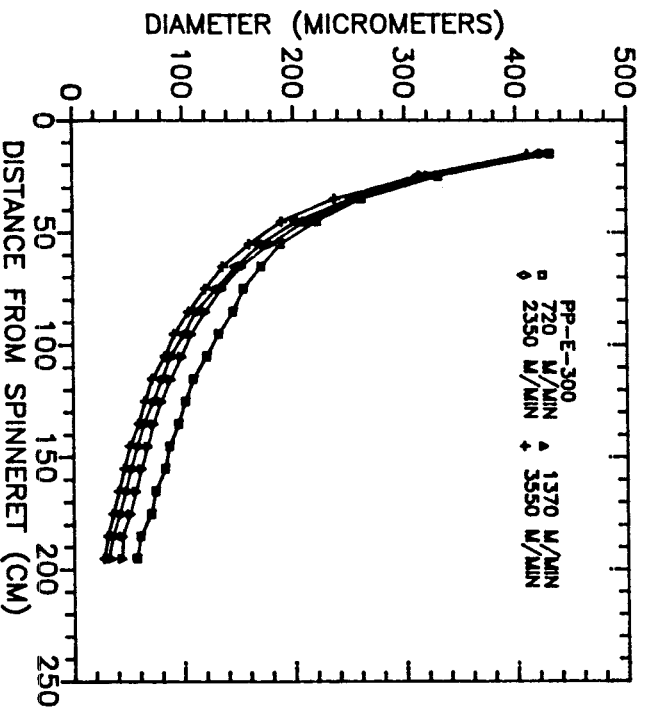
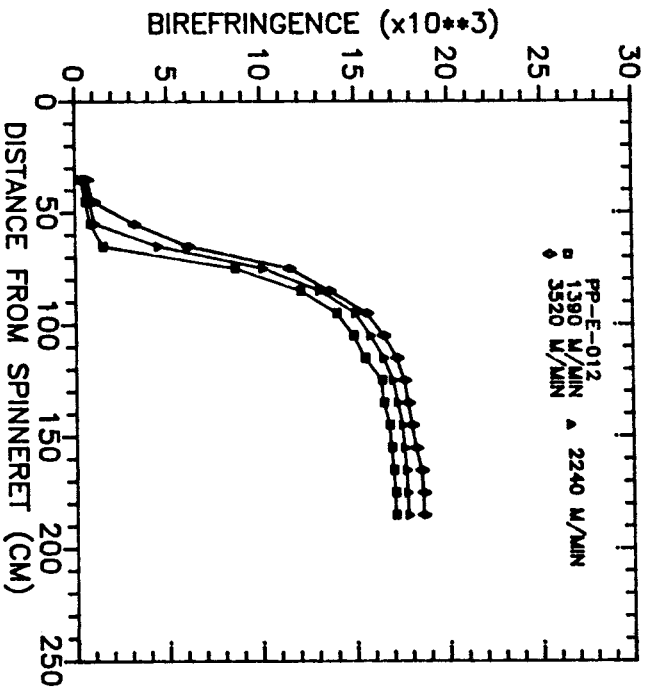
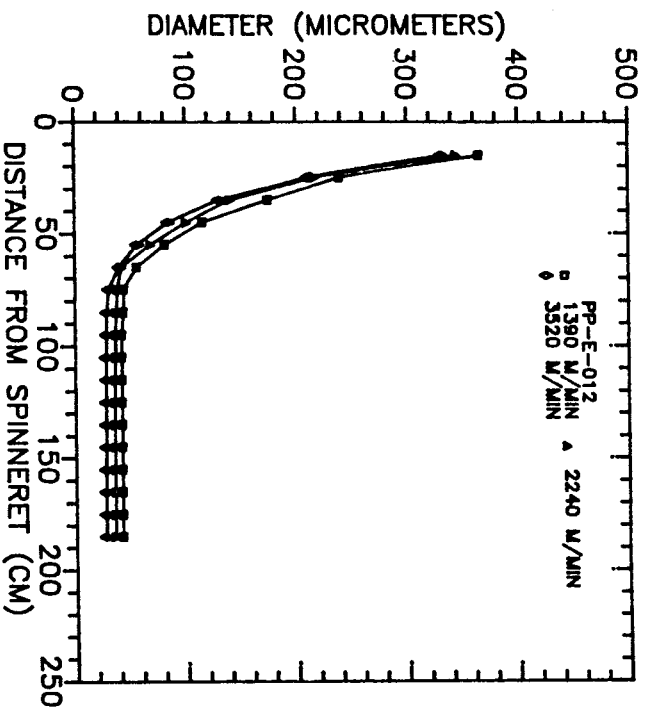


Figure 9. Diameter profiles, a and c, and birefringence profiles, b and d, for the 12 MFR resin, a and b, and the 300 MFR resin, c and d.

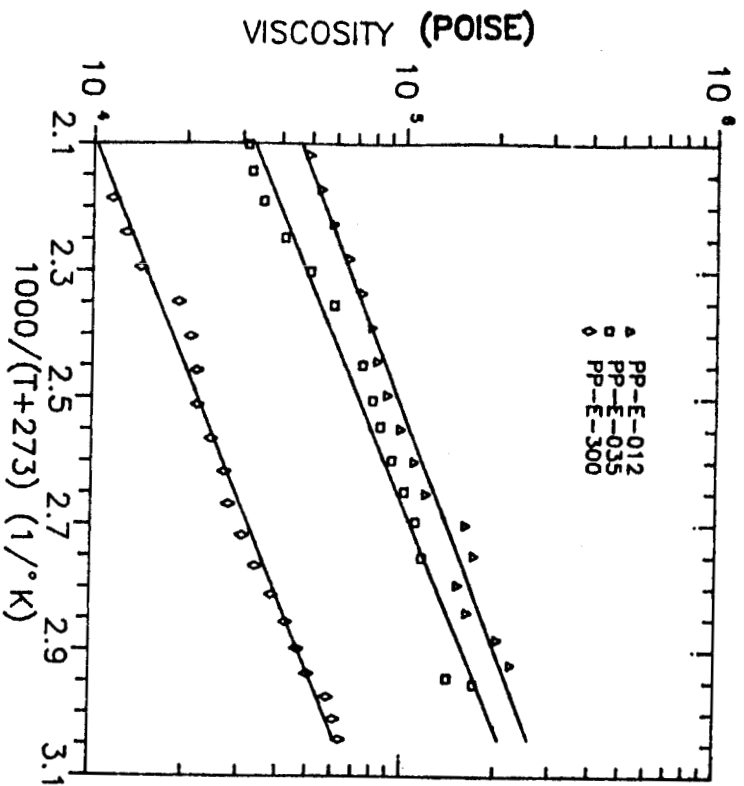


Figure 10. Apparent elongational viscosity as a function of temperature.

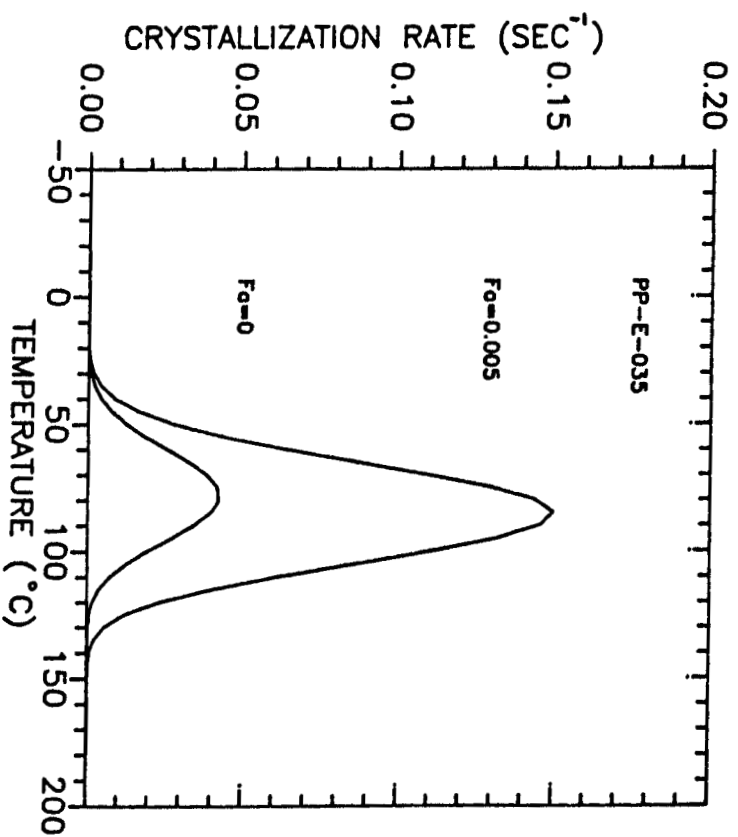


Figure 12. Crystallization rate $K(T, f_g)$ for the 35 MFR resin.

dependence and varies approximately as M_w to the 3.5 power. Comparison of the apparent elongational viscosities to isothermal measurements (24) of shear viscosity at two temperatures, 170°C and 180°C, showed that temperature dependence of apparent elongational viscosity is somewhat lower than observed for shear viscosities, while the magnitude of the apparent elongational viscosities are of order three times the zero shear viscosity in accord with the so-called Trouton law (25).

Crystallization Kinetics Under Molecular Orientation

The crystallization kinetics for the three resins were evaluated using the approach outlined above. The constants C_1 , C_2 and C_3 were first evaluated from earlier measurements (26,15) of quiescent crystallization rates. The on-line data for the three resins were then simulated using the mathematical model of Zieminski and Spruiell (14) modified by the use of Equation 5, Figure 2, for the crystallization kinetics with molecular orientation. The experimental values of apparent elongational viscosity derived above were used in the model and the parameter C of Equation 5 was adjusted until the simulation and on-line data were in agreement. It was found that this could be done with a high level of agreement as illustrated in Figure 11. It should be noted that C was only allowed to vary with resin and not with spinning conditions.

A plot of Equation 5 is shown for the 35 MFR resin in Figure 12 for two values of the amorphous orientation factor f_a . The curve for f_a equal zero corresponds to the crystallization rate $K(T)$ for quiescent crystallization, while the curve for f_a equal 0.005 illustrates the extremely important effect of molecular orientation. It is observed that molecular orientation not only increases the rate at any temperature but shifts the maximum rate to higher temperature. The value of C determined for each resin from the above analysis was 940, 1040 and 1160 for PP-E-012, PP-E-035 and PP-E-3001 respectively. It can be seen that the values of C do vary from resin to resin, but they only vary by a few percent, not by orders of magnitude.

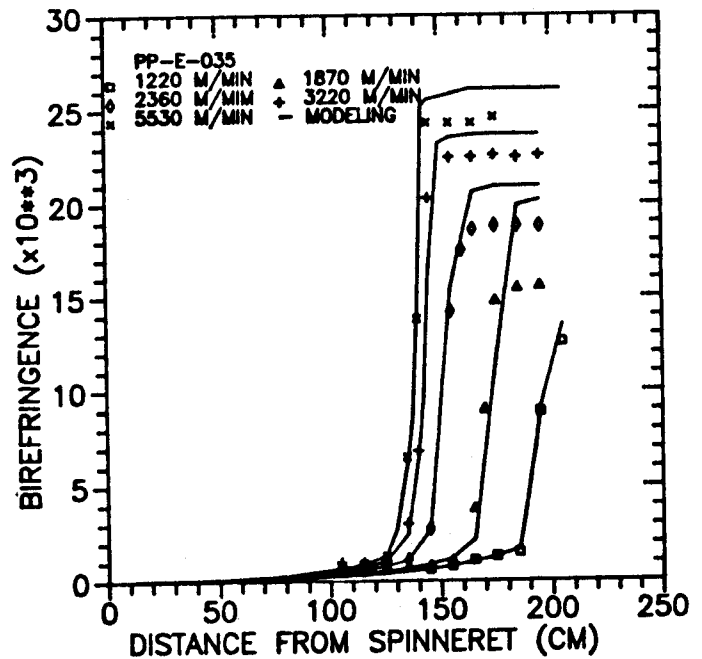
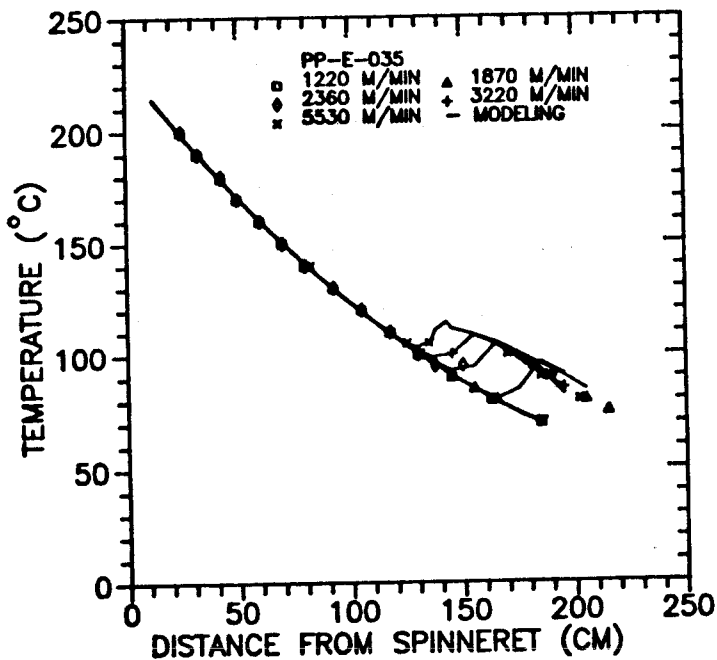
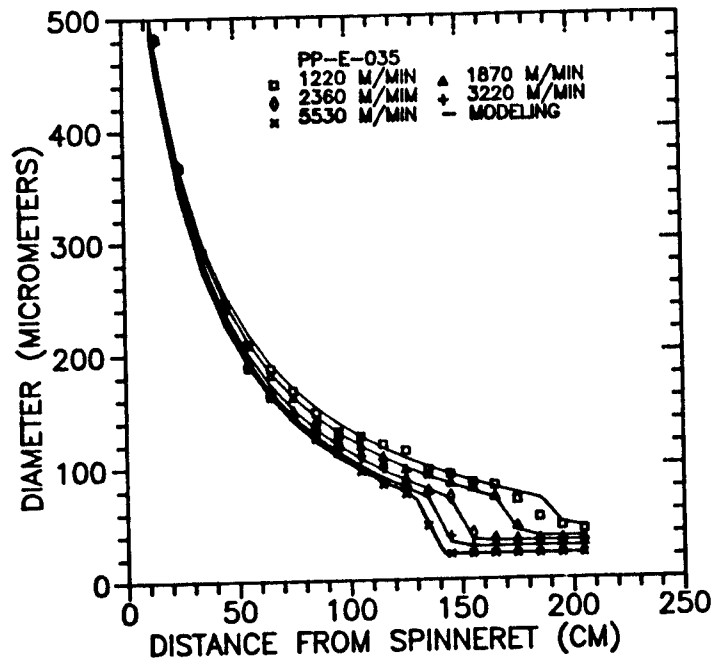


Figure 11. Comparison of simulated profiles (curves) to experimental profiles (points) for the 35 MFR resin. (a) Diameter, (b) birefringence, (c) temperature.

As shown in Figure 3 the overall development of crystallization during processing is best shown in terms of the "dynamic crystallization kinetic factor" $K(T, f_a) \times (dT/dt)^{-1}$. We show this evaluated at the conditions where crystallization starts for the three resins studied here in Figure 13 as a function of take-up velocity during spinning. The results of Figure 13 are shown after normalization by dividing by the quiescent crystallization rate $K(T)$ in Figure 14.

The increase of crystallization rate during processing compared to the quiescent case increases rapidly with decrease of resin MFR as shown in Figure 14. The dynamic crystallization kinetic factor for the 300 MFR resin is only of order a factor of ten greater than for the quiescent case under the spinning conditions studied, while the values for the 12 MFR resin are several orders of magnitude greater. These differences also explain why the crystalline order is greater the lower the resin MFR and the higher the take-up velocity.

It should be remembered that changing spinning conditions will result in changes in crystallization temperature, T , molecular orientation, f_a , and the cooling rate (dT/dt) . The primary reason for the very large differences of dynamic crystallization kinetic factors between the resins at given take-up velocity is due to differences of molecular orientation, f_a , developed for each resin. These differences in molecular orientation are due to the large differences in elongational viscosity between the resins.

Significance for Spunbonding or Melt Blowing

The results presented above give many insights into the effects of changing resin characteristics on their processability in the spunbonding and melt-blown processes and the resulting structure and properties of the webs. In addition to the expected ease and reduced cost of pumping higher MFR resins through the small spinneret holes, the lower elongational viscosities allow greater attenuation of the filament diameters and slower crystallization during processing. Generally, softer webs are produced by

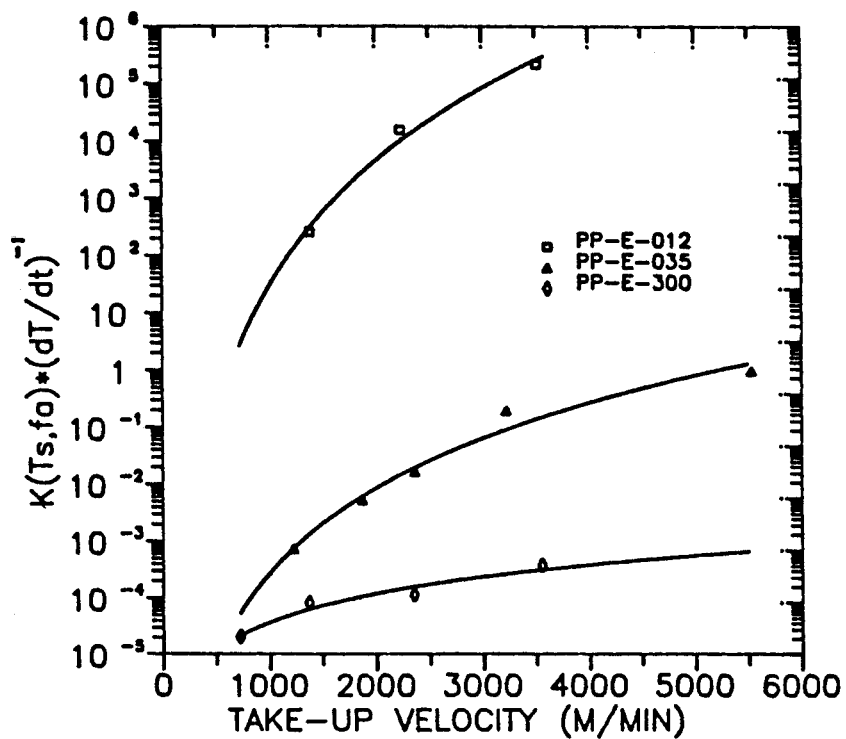


Figure 13. Comparison of dynamic crystallization kinetic factors for the three resins during melt spinning.

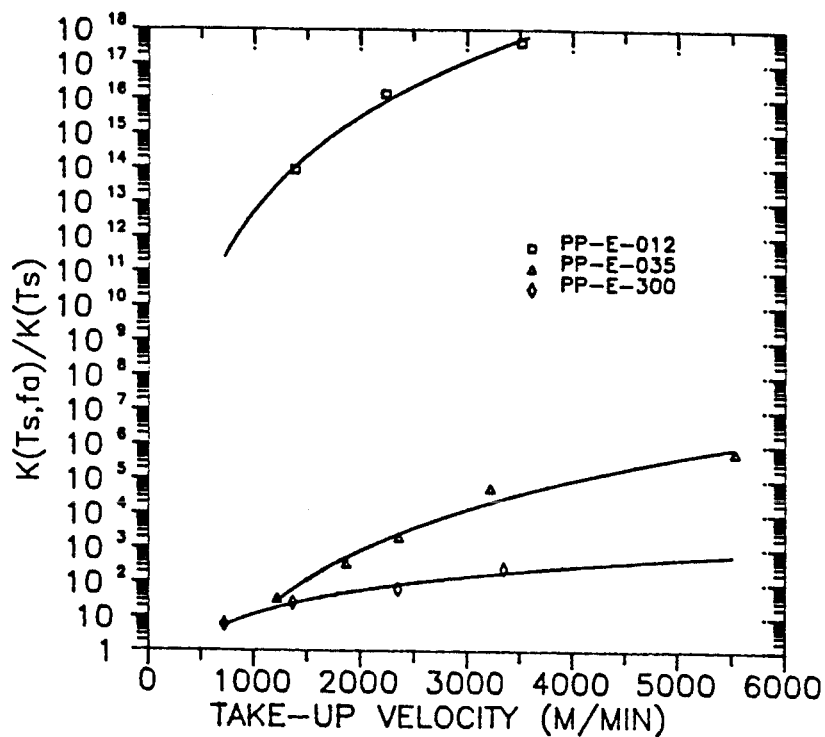


Figure 14. Normalized dynamic crystallization kinetic factors for the three resins.

reduction of fiber diameters and crystallinity of the filaments. Interfilament bonding and entanglements also play an important role in determining the web properties. This involves the thermal bonding step for spunbonding, but for the melt-blown process bonding must be controlled by changing the resin properties and such process parameters as extrusion temperature, air temperature and pressure, and die-to-collector distance. The point in the process at which crystallization of the filaments occurs should be key to effecting this control.

CONCLUSIONS

A simple monofilament melt spinning process combined with on-line measurement of filament diameter, temperature and birefringence provides an exceptionally powerful technique for evaluation of resins for application in related processes such as multifilament spinning, spunbonding and melt blowing. In addition to the basic spinnability of the resin, the technique can be used to evaluate filament structure-property relationships and to measure apparent elongational viscosities and relative crystallization behavior of selected resins. Through understanding of these basic properties of the resins and their interaction with process variables it should be possible to develop a detailed understanding of the role of resin characteristics on the filament and web structure and properties.

The present application of the technique to three polypropylene resins showed that crystallization kinetics are largely controlled by the molecular orientation developed in the melt in the temperature range where crystallization can occur. This molecular orientation is in turn controlled by the resin apparent elongational viscosity. Increasing the resin molecular weight tends to increase both its elongational viscosity and its crystallization rate during processing.

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RECENT DEVELOPMENTS IN ETHYLENE POLYMERS
FOR MELT BLOWN APPLICATIONS

PRESENTED AT THE 3RD CHEMICAL CONGRESS
OF NORTH AMERICA
JUNE 7, 1988
TORONTO, ONT. CANADA

by
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EXXON CHEMICAL CO.
BAYTOWN, TEXAS

ABSTRACT

Although ethylene based polymers have been available for film and molding applications for a number of years, they have only recently been investigated for their utility in the melt blown process. This paper reviews the types of ethylene polymers that have been developed by Exxon Chemical Co. for this process and the applications envisioned for each family of polymers.

INTRODUCTION

Over the past two years, Exxon Chemical has developed three families of ethylene based polymers for melt blown applications. These include: (1) Linear polyethylene for soft, non-elastic applications, (2) Ethylene Vinyl Acetate (EVA) copolymers for elastic applications, and (3) Ethylene Methyl Acrylate (E-MA) copolymers for elastic bonding applications. The polymers developed in each family for the melt blown application are listed in Table 1.

LLDPE: SOFT AND DRAPEABLE WEBS

Linear low density polyethylene(LLDPE) has been found to have several desirable characteristics that can be utilized in melt blown webs. Compared to polypropylene(PP), these polymers exhibit a much lower bending modulus which translates into a softer, more drapeable fabric. The lack of long chain branching in combination with a narrow molecular weight distribution(MWD) combine to contribute to the ability of this polymer to draw down into very fine microfibers. The ability of the polymer to resist gamma radiation also makes it an interesting candidate for medical products requiring gamma sterilization. These attributes are reviewed in Table 2.

Figure 1 provides an indication of the fiber size achievable with a 100 melt index LLDPE resin as a function of the air rate used to produce a melt blown web. At higher air rates, comparable to those used to manufacture PP melt blown filtration capable webs, the LLDPE is also able to be drawn to 2-3 micron fiber diameters.

Figure 2 compares the shear rheology of the 100 MI LLDPE grade to a typical 300 MFR PP resin. This data indicates that one should expect a higher die pressure at a given flow rate for the LLDPE since it is not as shear sensitive as the PP. This is in fact observed as shown in Table 3 where the processing characteristics for a PP web are compared to those required to produce an LLDPE web. In addition to the shear sensitivity difference between the polymers, LLDPE is also limited by the melt temperature which can be tolerated before the web begins to melt on the collector roll and form a stiff 'boardy' product. The conditions for web formation shown in Table 3 were found to provide the optimum balance between rate and web formation for this polymer on the pilot melt blown line at the University of Tennessee.

Table 4 provides an overview of the web properties for an LLDPE polymer relative to both a PP web and an LDPE web. The better drape of the LLDPE web is reflected in the lower modulus values reported. The lower tenacity of the LLDPE web is the price to be paid for this improvement in web hand. The primary difference between LLDPE and LDPE webs is the ability of the linear polymer to draw down into filtration quality fiber diameters. The branched LDPE is not capable of producing this fine a fiber diameter.

Figure 3 provides evidence of the gamma radiation resistance achievable with LLDPE melt blown webs. Although the bursting strength measured for the PP web is initially much higher than that measured with the LLDPE web, the loss in strength at 2.5 Mrads for the LLDPE web is negligible, while the loss for the PP web puts it at a lower value than that for the radiated LLDPE. The radiation resistance of the PP web can be improved marginally by incorporating gamma stabilizers, but the LLDPE web still shows higher strength even without the stabilizer.

EVA ELASTIC WEBS

Ethylene vinyl acetate copolymers (EVA) have been utilized extensively in the packaging field in areas that take advantage of their high degree of elasticity. These polymers are produced in a bulk polymerization process and incorporate vinyl acetate in the side chains as shown in Figure 4. The resulting polymer has a low modulus and high degree of extension capability. VA levels in the range of 18 to 28 % by weight are most commonly available. They are limited in their applications by thermal degradation occurring at melt temperatures higher than 230°C and the fact that they do not draw down into as fine a fiber as can be achieved with LLDPE or PP.

Table 5 lists the processing conditions found to produce the best possible webs on the melt blown pilot line at the University of Tennessee. As mentioned above, the melt temperature is constrained by the process degradation of EVA which occurs at temperatures higher than 230°C. Even with this lower melt temperature constraint, the die pressure measured for the EVA polymer tested is lower than that for the LLDPE owing to a combination of higher shear sensitivity and higher melt index achievable with the EVA. Higher air flow rates are possible since the elastic EVA is not as prone to fly formation as is the linear PE or PP. The branched nature of this polymer does not allow as fine a fiber to be formed however, with average fiber diameters on the order of 8 to 10 microns.

Table 6 compares the web properties of a 28% VA copolymer to those of LLDPE and LDPE. As mentioned above, the low modulus and high vinyl acetate incorporation contribute to a high degree of elasticity in the web as shown in this table. LDPE also shows some elasticity owing to its branched structure, but the effect of the vinyl acetate side chain on the elasticity is clearly differentiated.

Table 7 shows the elastic recovery of a 28% VA web as a function of the number of stretch cycles it is subjected to at various total stretch levels. As can be seen, the recovery is nearly complete at stretch levels less than 50%. Higher levels of stretch produce higher permanent set values as expected.

Figure 5 shows the stress vs strain plot of the EVA web at a 100 % extension level. The first and fifth cycle are shown for comparison. As shown, the majority of the permanent set in the fabric is seen after the first cycle, and subsequent cycles show little further deterioration in this property.

E-MA BONDING WEBS

Ethylene methyl acrylate copolymers (E-MA), like EVA copolymers are formed via a random placement of methyl acrylate in the ethylene backbone through a free radical bulk polymerization process. The level of MA available in commercial polymers of interest normally varies between 15 to 25 % by weight. Like EVA, the disruption of the ethylene backbone crystallinity by the presence of the MA side chains leads to a low modulus, elastic product. The structure of this family of polymers is shown schematically in Figure 6.

Table 8 compares the processing conditions for an E-MA web with those used for the EVA web. One of the advantages seen for E-MA relative to EVA is a higher threshold for thermal degradation. This polymer can be processed at melt temperatures up to 325°C. In practice, the melt temperature is limited by the softening point of the polymer on the collector roll. Cooling of the collector roll would be expected to allow for higher temperatures to be employed, and hence higher output rates to be generated. Since the E-MA polymers are also non-linear, branched materials, they also cannot be drawn down into very fine diameter fibers.

Table 9 compares the web properties of the other types of ethylene polymers to those of E-MA polymers. The modulus values measured are comparable to those for LDPE and EVA. The tenacity is comparable to that for EVA. The elongation measured is intermediate between that of LDPE and EVA. So why should one consider using E-MA? The answer is that the E-MA is a recognized tie resin in the packaging field, and this property is of interest in multiple layer structures combining dissimilar polymers.

Figure 7 shows a conceptual view of how an E-MA web might be utilized to provide an adhesive breathable tie layer between a PP web and an LLDPE web. The PP layer provides strength and abrasion resistance to the outside of the garment while the LLDPE provides skin contact softness. Figure 8 shows how such a construction might be fabricated. Table 10 details the results of a web produced according to this scheme. The composite web takes on the strength of it's strongest member, the PP melt blown web. Even with the much higher total basis weight of the composite structure, the drape of the web is only slightly reduced below that of the single ply PP web as reflected in the modulus data. The higher elongation measured for the composite structure reflects a slight increase in web toughness attributable to the elasticity of the E-MA bonding fabric layer.

Another construction conceptualized for E-MA utility is shown in Figure 9. This figure shows how E-MA can be utilized as a thermally activated adhesive layer between a non-woven PP web and an LLDPE film. This type of construction is typical of that employed in a diaper or feminine hygiene product.

The bond strength measured with and without the E-MA melt blown web present is shown in table 11. Although this data shows a marginal increase in bond strength between the LLDPE and PP layers, it should be pointed out that the failure mechanism with the E-MA layer present was distinctly different than that without the E-MA. With the E-MA layer in place, the composite construction failed through a combination of film yielding followed by PP web tensile failure. This is reflected by the much higher elongation values measured with the E-MA present. It is anticipated that a film with higher tensile strength and/or a spunbond PP web would give much higher bond strengths than those reported here.

CONCLUSIONS

In conclusion, it is interesting to note areas where ethylene based polymers might find utility in melt blown webs. LLDPE can produce very fine denier fibers comparable to those achieved for filtration applications within PP. The LLDPE webs have much better inherent gamma radiation resistance than PP which makes them of interest in medical applications requiring gamma sterilization. The applicability of the LLDPE webs is limited only by the lower tenacity seen as a tradeoff for the better softness and drape relative to PP.

EVA produces a melt blown web with a high degree of recoverable elasticity. This characteristic makes this polymer an interesting candidate as a stand-alone elastic web.

E-MA webs have been found to have utility as a breathable adhesive layer between dissimilar polymers such as PP and LLDPE. The inherent elasticity of this family of polymers also contributes to the toughness of a composite web where it is utilized.

TABLE 1**EXXON ESCORENE® POLYETHYLENE GRADES
FOR MELT BLOWN APPLICATION**

	Grade	Melt Index (g/10Min)	Other Specifications
Linear PE	LPX-61.69	100	.930 g/cc
EVA	LD-724.36	135	18% VA
	LD-727.16	450	18% VA
	LD-764.36	200	28% VA
	LD-765.36	500	28% VA
E-MA	XS-13.04	135	20% MA

TABLE 2**LINEAR LOW DENSITY POLYETHYLENE (LLDPE)**

Structure:

LDPELLDPE

Properties:

- Lower Modulus Than PP or HDPE
- No Long Chain Branching
- Narrow MWD
- Radiation Resistant

TABLE 3

TYPICAL PROCESSING CONDITIONS FOR LLDPE VS PP

	300 MFR PP	100 MI LLDPE
Melt Temperature (°C)	270	250
Die Pressure (psi)	200	400
Air Temperature (°C)	260	240
Air Flow (CFM)	250	250
Die To Collector Distance (in)	10-12	10-12
Output (g/hole/min)	0.42	0.40
Fiber Diameter (microns)	2-4	2-4

TABLE 4

LLDPE WEB PROPERTIES VS LDPE AND PP

Type Of Resin	PP Homopolymer	LLDPE	LDPE
Melt Index (dg/min) At 190°C	95	100	250
Density (g/cc)	.909	.930	.925
Comonomer	-	C ₄	-
Fabric Basis Wt (oz/sq yd)	.95	1.02	1.00
Modulus MD	7.1	0.9	1.5
MPA TD	2.6	0.5	0.9
Tenacity MD	.20	.04	.04
(g/denier) TD	.11	.02	.02
Elongation MD	37	28	54
(%) TD	80	42	69
Fiber Diameter (Microns)	2-4	2-4	10-12

TABLE 5

TYPICAL PROCESSING CONDITIONS FOR EVA VS LLDPE

	190 MI EVA	100 MI LLDPE
Melt Temperature (°C)	230	250
Die Pressure (psi)	350	400
Air Temperature (°C)	220	240
Air Flow (CFM)	300	250
Die To Collector Distance (in)	15-18	10-12
Output (g/hole/min)	0.38	0.40
Fiber Diameter (microns)	8-10	2-4

TABLE 6

EVA WEB PROPERTIES VS LDPE AND LDPE

Type Of Resin	LLDPE	LDPE	EVA
Melt Index (dg/min) At 190°C	100	250	190
Density (g/cc)	.930	.925	.945
Comonomer	C ₄	-	28% VA
Fabric Basis Wt (oz/sq yd)	1.02	1.00	.97
Modulus MD	0.9	1.5	1.8
MPA TD	0.5	0.9	1.4
Tenacity MD	.04	.04	.02
(g/denier) TD	.02	.02	.01
Elongation MD	28	54	204
(%) TD	42	69	216
Fiber Diameter (Microns)	2-4	10-12	8-10

TABLE 7

**ELASTIC RECOVERY AND PERMANENT SET
OF AN EVA WEB (2.8 OSY)**

Extension Level (%)		Maximum Force (LBS) On Cycle No.					Permanent Set (%) At 5 Cycles
		1	2	3	4	5	
50	MD	0.80	0.76	0.73	0.70	0.73	3
	TD	0.88	0.84	0.80	0.79	0.78	3
100	MD	1.14	1.06	1.02	1.00	0.98	18
	TD	0.54	0.52	0.50	0.49	0.48	18
150	MD	1.18	1.08	1.04	1.00	0.98	38
	TD	0.60	0.56	0.54	0.52	0.54	34
200	MD	1.06	0.96	0.90	0.88	0.86	69
	TD	0.54	0.50	0.48	0.46	0.44	50
250	MD	1.14	0.98	0.88	0.82	0.79	94
	TD	0.66	0.59	0.56	0.53	0.51	75

TABLE 8

**TYPICAL PROCESSING CONDITIONS
FOR E-MA VS EVA**

	135 MI E-MA	190 MI EVA
Melt Temperature (°C)	260	230
Die Pressure (psi)	350	350
Air Temperature (°C)	250	220
Air Flow (CFM)	225	300
Die To Collector Distance (in)	12-15	15-18
Output (g/hole/min)	0.40	0.38
Fiber Diameter (microns)	10-12	8-10

TABLE 9

E-MA WEB PROPERTIES VS OTHER ETHYLENE POLYMERS

Type Of Resin	LLDPE	LDPE	EVA	E-MA
Melt Index (dg/min) At 190°C	95	250	190	135
Density (g/cc)	.930	.925	.945	.940
Comonomer	C ₄	-	28% VA	20% MA
Fabric Basis Wt (oz/sq yd)	1.02	1.00	.97	1.04
Modulus MD	0.9	1.5	1.8	1.8
MPA TD	0.5	0.9	1.4	0.8
Tenacity MD	.04	.04	.02	.02
(g/denier) TD	.02	.02	.01	.01
Elongation MD	28	54	204	65
(%) TD	42	69	216	126
Fiber Diameter (Microns)	2-4	10-12	8-10	10-12

TABLE 10

TENSILE STRENGTH/ELONGATION BALANCE FOR PP/E-MA/LLDPE LAMINATED WEB VS. PP WEB

Product Structure	1 OSY PP Web	1 OSY PP Web 0.7 OSY E-MA Web 1 OSY LLDPE Web
Tenacity (g/denier)		
MD	0.11	0.10
TD	0.05	0.06
Elongation (%)		
MD	19	41
TD	40	57
Secant Modulus (M Pa)		
MD	97	117
TD	31	54

TABLE 11

**BOND STRENGTH PP/E-MA LAMINATE VS.
PP BONDED TO LLDPE FILM**

Product	PP Bonded Directly To LLDPE Film	PP/E-MA Composite Bonded To LLDPE Film
Bond Temperature (°F)	225	225
Bond Pressure (psi)	40	40
Bond Time (Sec)	5	5
Bond Strength At Fail (Lbs)	2.0	3.1
Elongation At Failure (%)	14	157

FIGURE 1

**FINE DENIER FIBERS CAN BE ACHIEVED
WITH LLDPE**

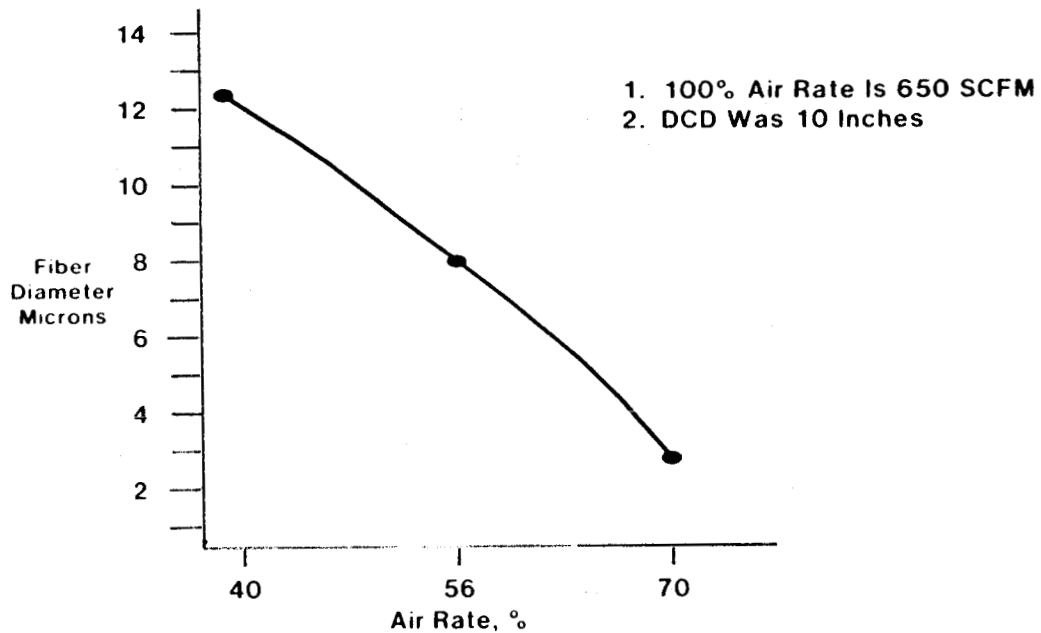


FIGURE 2

LINEAR PE's LESS SHEAR SENSITIVE THAN PP

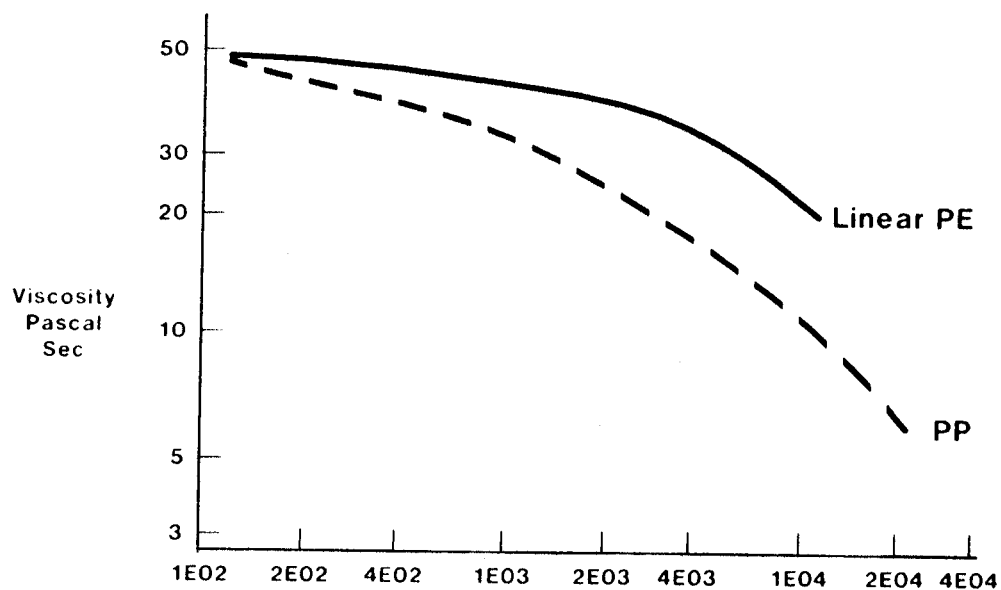


FIGURE 3

**STRENGTH RETENTION OF LINEAR PE
VS PP WEB EXPOSED TO GAMMA RADIATION**

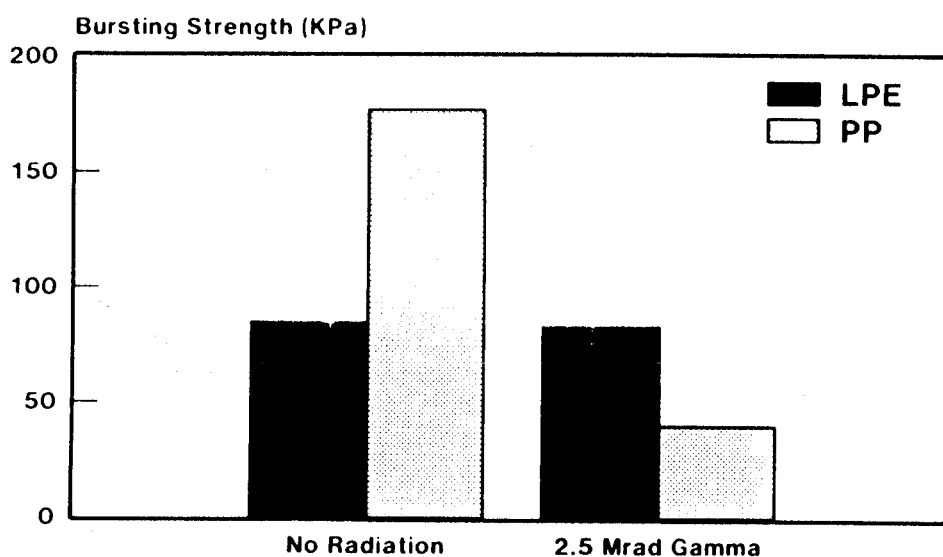
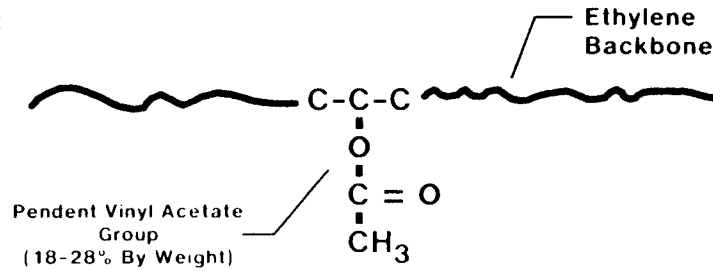


FIGURE 4

ETHYLENE VINYL ACETATE COPOLYMER (EVA)

Structure:



Properties:

- Low Modulus
- High Elongation To Break
- Low Temperature Thermal Bonding

Limitations:

- Process Degredation At Temperatures Over 230°C
- Branching Comparable To LDPE

FIGURE 5

ELASTIC RECOVERY OF EVA WEB (2.8 OSY) OVER 5 CYCLES AT 100% EXTENSION/CYCLE

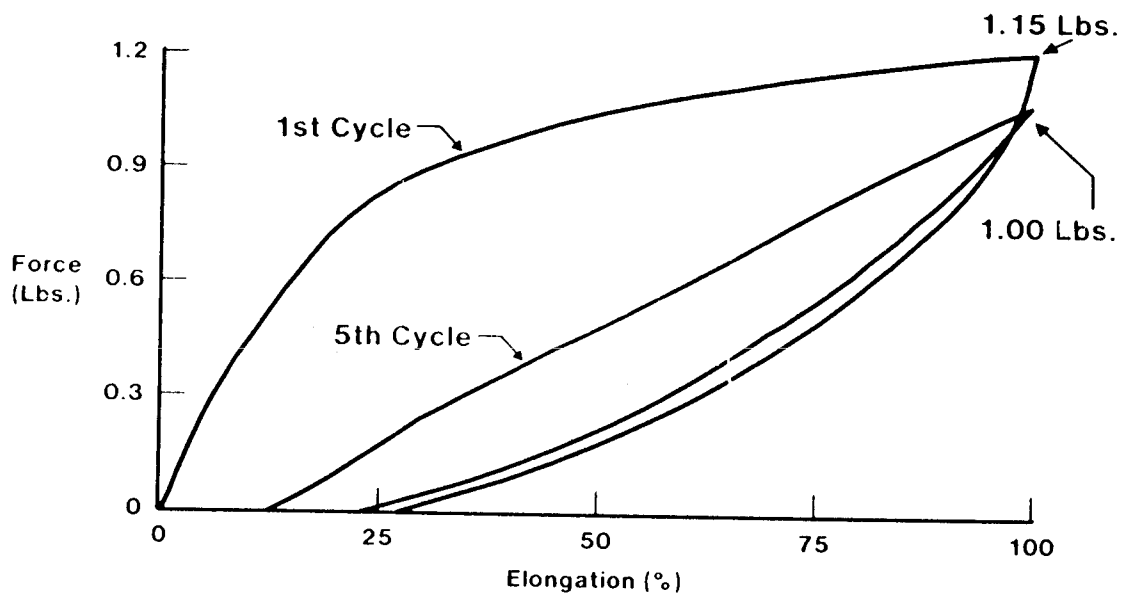
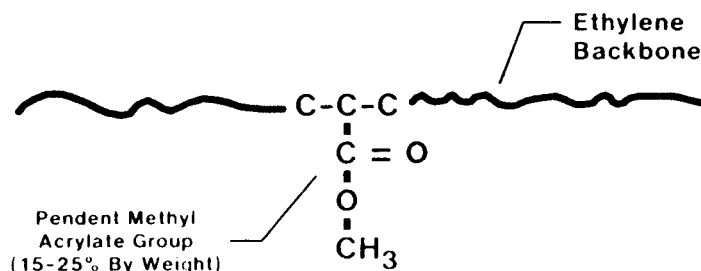


FIGURE 6

ETHYLENE METHYL ACRYLATE (E-MA)

Structure:



- Properties:
- Low Modulus
 - High Elongation To Break
 - Compatability With PE And PP
 - Thermally Stable Up To 325°C
- Limitations:
- Branching Comparable To LDPE

FIGURE 7

PP/E-MA/LLDPE COMPOSITE STRUCTURE

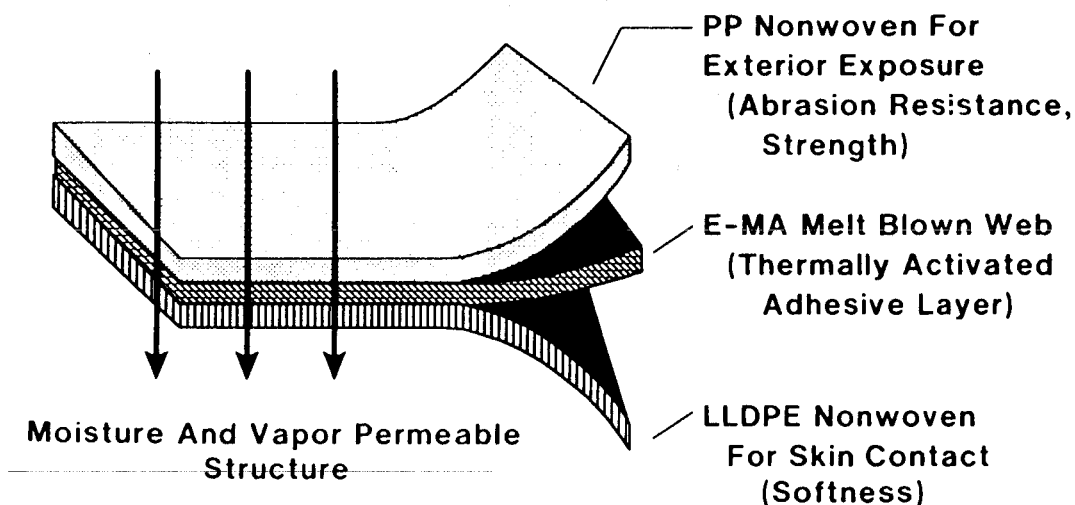


FIGURE 8

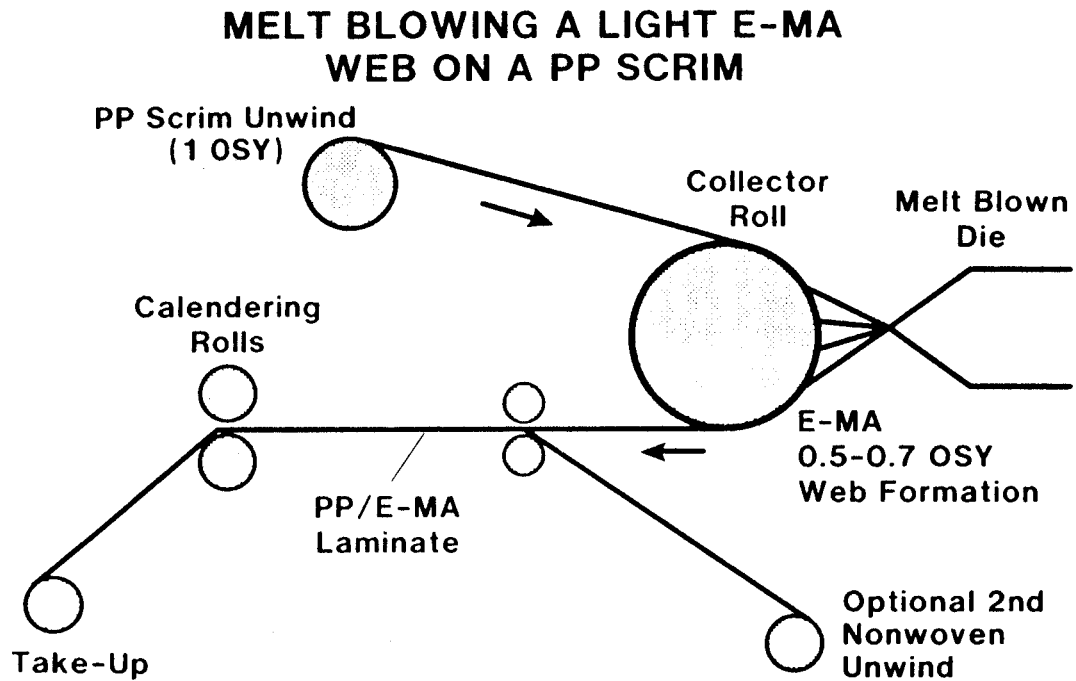
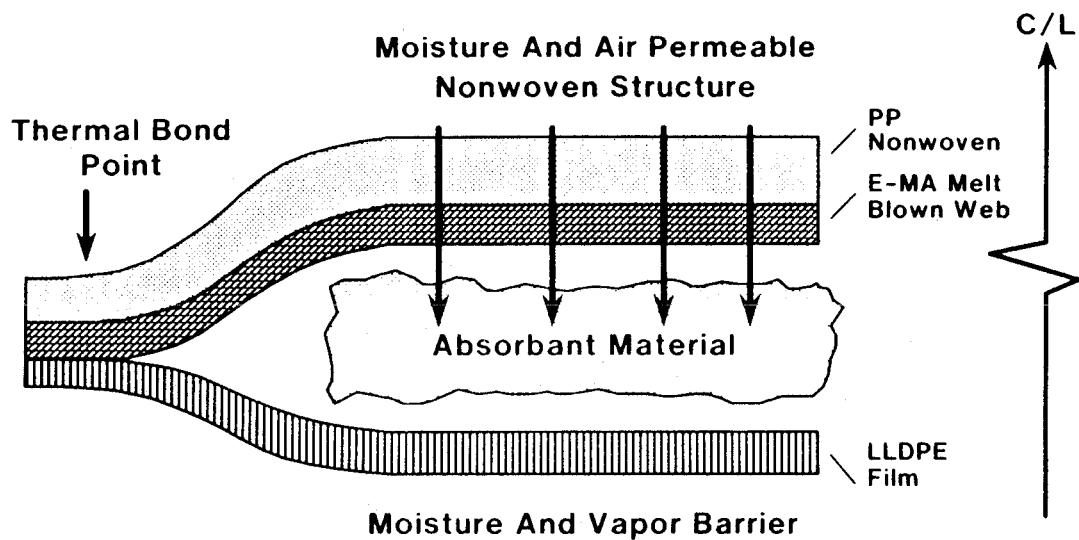


FIGURE 9



INTRODUCTION

Polyethylene has been utilized in fiber form for many years. However, only recently have specific grades of polyethylene been developed for melt spinning into fiber for use in the nonwovens areas. These fiber/fabric forming technologies can be broken down into three basic areas: spunbonded fabric formation, staple fiber manufacture, and melt blown web formation. Each of these processes is extremely complicated, placing widely varying demands on the polymer being processed. In fact, variability between equipment manufacturers poses processing challenges, even though the overall unit operations remain the same. While these three spinning processes differ from one another, they do have a fundamental common denominator - they can each produce fiber by extruding a thermoplastic polymer to a suitable diameter. Historically, polypropylene has been used with great success in these processes. Linear low density polyethylene has now joined the list of options available to nonwovens manufacturers as polymers suited to processing by these demanding techniques.

While these processing technologies may not be familiar, products made from them certainly are. Some of these products include oil sorbent pads, baby diapers, feminine hygiene products, hospital gowns and filter masks. Attributes offered by use of linear low density polyethylene can enhance the performance and value of some of these products, without affecting processability. This paper will briefly discuss key differences offered by use of linear low density polyethylene in the nonwovens area, and offer some insight as to polyethylene-based thermally bonded fabric strengths.

POLYETHYLENE

Most of the nonwovens industry is very familiar with polypropylene and its use in fiber or fabric form. Polyethylene, however, is a newcomer in the fine denier, or small diameter, fiber markets. Since both of these polymers are considered members of the polyolefins family, many people are unaware that polymer structure differences can impart drastically different processability requirements and performance variations. A recurring mer unit of polyethylene is structurally significantly different from a recurring mer unit of polypropylene, as Figure I indicates. As their names identify, polypropylene is manufactured by polymerizing propylene, while polyethylene is made by polymerizing ethylene. Either of these polymers can be copolymerized with other materials to modify or enhance certain property characteristics. For example, the density of polyethylene can be manipulated by the type and amount of comonomer reacted with ethylene to make the polymer. This comonomer, in combination with the manufacturing process, affects the type, frequency, and length of branching occurring in the molecule. This variation results in the different types of polyethylene described in Figure II. High density homopolymer (typically 0.950 gm/cc and higher), low density (0.910-0.925 gm/cc), and linear low density polyethylene (density ranges of low density and medium density polyethylene) are represented here showing the branching differences created by the manufacturing process. Overall, the varying density ranges of polyethylene can be labeled according to Table I. Density can vary for polyethylene from less than 0.910 g/cc for ultra low density polyethylene to 0.960 g/cc for homopolymer.

As mentioned earlier, the comonomer incorporated into the polyethylene backbone can influence end product properties. Two of the more common comonomers used today are butene and octene. Since these two monomers are different in molecular weight, the amount of each utilized to achieve the same copolymer density is different. That is, more branches are necessary for an ethylene-butene copolymer to have the same density as an ethylene-octene copolymer. This is significant since it indicates the efficiency of the branches using octene as comonomer for density control. This particular structural difference, represented in Figure III, can later manifest itself in fiber/fabric form when exposing the polymer to gamma or electron beam irradiation for the sterilization of nonwoven goods. The more tertiary carbon bonds present in the ethylene copolymer, the more susceptible that material is to carbon/carbon bond breakage as a result of chain scission. Since ethylene-octene copolymers have less of these tertiary carbon bonds than say ethylene-hexene or ethylene-butene copolymers at the same density, they are less susceptible to this chain scission phenomenon and more likely to have crosslinking of the molecules dominate. Both chain scission and crosslinking occur in polyethylene during exposure to high energy electron bombardment.¹

Density manipulation, as already mentioned, is a key property handle of polyethylene which differs from traditional polypropylene fiber forming resins. While some propylene copolymers exist (most dependent on economic justification²), few have entered the fine denier fiber forming areas. Density variation, in fact, can modify the softening point of a polymer. Since density is synonymous with crystallinity (the remainder of the polymer essentially amorphous), the lower the density, the lower

the percent crystallinity and hence the lower the softening point.³ For high density polyethylene (homopolymer) the peak melting point is approximately 134°C. For the range of LLDPE fiber forming resins, the peak melting point is about 124°C. Contrasted with polypropylene, which has a typical peak melting point of 175°C, this lower melting point allows processing latitudes previously not available for a cost-effective fiber forming material. For example, staple fiber formed from LLDPE can be useful as a sacrificial binder fiber which melts at a lower temperature than the matrix fibers thus allowing the basic web structure, typically polypropylene or polyester based, to be unaffected, but securely bound by the melted LLDPE fiber. In fact, such staple fiber is being touted as useful in this growing area⁴, in addition to its usefulness as a relatively radiation resistant fiber as previously mentioned.

This lower melting point of fiber or fabric made from polyethylene allows it to be bonded to film as well as to itself for making innovative composite structures. For example, in the diaper market, coverstock (historically polypropylene) is often bonded to the backsheet (usually polyethylene in film form) by using adhesives. By using the same material (LLDPE) to perform both functions, a clean heat seal or thermal bond can integrate the entire structure.

SOFTNESS

In addition to the melting point variation controlled by density, polyethylene (especially linear low density ethylene-octene copolymers) has

unsurpassed softness in fiber form, owing in part to this density manipulation. As Figures IV and V demonstrate, polyethylene has over an order of magnitude difference in stiffness versus polypropylene at the same density (say 0.910 g/cm³)^{5,6}. Product properties can therefore be tailored to meet the needs of the consumer using these density/stiffness relationships. For example, for personal care products softness in the fiber or fabric is desired to avoid skin irritation. For high loft structures, stiffness is beneficial, since stiffer fiber increases stacking height.

THERMAL BONDABILITY

Another key difference between polyethylene-based fabrics and polypropylene-based fabrics is their thermal bonding properties. Polyethylene has a typical heat capacity of 0.9533 cal/gm°C while polypropylene is considerably lower at 0.9203 cal/gm°C. This makes thermal bonding of LLDPE-based materials challenging! Added heat input to overcome this delta may be necessary to enable effective bonding of the fiber.

Two of the fabrication techniques previously mentioned - staple fiber and spunbonded fabric formation - both rely heavily on the fiber-to-fiber bonds which occur when webs are passed between heated calender rolls. These rolls are typically oil heated and compressed so that the web passing between them not only thermally fuses in places, but also mechanically fuses. One of the calender rolls is embossed with

roughly a 16% land area (or embossed pattern area). The other roll is smooth and usually operates a few degrees cooler than the embossed roll to prevent the polymer web from sticking. The speed of these rolls is variable, so that it may be matched with other pieces of equipment to effectively run an entire manufacturing process. In the case of a spunbonded line, the fibers are hopefully continuous! Using staple fiber requires an extra step of carding the fiber into the web, since the fiber is discontinuous, or cut. The web formed from each of these processes is then passed between these rolls for fusion.

As part of a study undertaken at Dow to understand thermal bondability of LLDPE-based fiber compared to polypropylene-based fiber, a test was designed to simulate the carding and bonding steps for staple fiber. The small fabrics formed from this procedure were then cut into one inch by four inch strips, mechanically stretched to break on an Instron, and the results normalized to one ounce/square yard basis weight. The test procedure for this is as follows:

Fibers are collected continuously onto spools using a throughput rate necessary to achieve a desired denier per filament, about 2.5 dpf. After collection of sufficient sample size, the fibers are cut off of the collection spool and cut into 1.5-inch staple fibers. One and one quarter gram samples of these 1.5-inch staple fibers are weighed out and formed into slivers using a RotoRing sample preparation device (manufactured by Spinlab, Inc.). A sliver is an ordered collection of fibers such that the fiber ends are randomized while the fibers themselves are all paralleled. The structure is about four inches wide by about ten inches long after gently opening the sliver tow. This opened

sliver tow is then fed into a Beloit Wheeler bench-top calender bonder for thermal tie down of the filaments where pressure and temperature are adjusted for optimal bonding conditions and fabric strength.

The Beloit Wheeler calender bonder has an embossed area of approximately 20%. It is important to note that fibers used in this particular test are not crimped. Also, the machine direction tensile strengths are the only meaningful test, since the RotoRing sample preparation device only aligns the fibers parallel to one another and not in the cross direction as a typical industrial card would. The one inch by four inch fabric strips are then cut from this bonded web.

By using this test and by examining Dow ethylene-octene type linear low density polyethylenes of various molecular weights (reported as melt index which is measured at 190°C), a trend is observed as illustrated in Figure VI.

For a decrease in melt index or an increase in molecular weight of the LLDPE, the bonded fabric tenacity increases. Note that a typical fine denier producing polypropylene resin used on the same test has a noticeably higher bonded fabric tenacity. This is certainly a significant difference between the two fiber forming polymers and may limit the growth of 100% LLDPE- based fabrics into the nonwovens business. While extrapolation of the data presented in Figure VI may lead to an educated guess of using a LLDPE with a melt index of 4 (gms/10 minutes) to achieve the same bonded fabric tenacity as polypropylene, spinnability of that resin into fiber is still an issue. Drawdown of higher molecular weight polyethylene into fine denier fiber, which can then be

thermally bonded into useful fabrics, remains the biggest challenge facing this new polymer choice for nonwovens manufacturers.

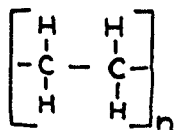
SUMMARY

Polypropylene has enjoyed success and subsequent growth in the nonwovens market areas in recent time. Polyethylene, especially linear low density polyethylene, could be an effective improvement over polypropylene for some of these nonwovens applications, offering such attributes as lower thermal bonding temperatures and very fine hand or softness. While these benefits may be lucrative, some questions and challenges still remain. Probably the biggest of these hurdles is increasing bonded fabric tenacity, without losing processability. While this particular obstacle may seem unattainable, many interesting and unique approaches are still not fully explored. These innovations may indeed lead to a linear low density polyethylene capable of processing into fiber at speeds of 4000 meters per minute and bonded into fabric with tenacities greater than 4000 grams!

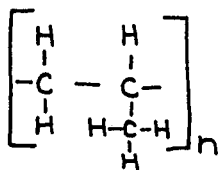
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- 6 Raff, R. A. V., and K. W. Doak, Crystalline Olefin Polymers, p. 709 (Copyright 1965 by John Wiley & Sons, Inc.). Reprinted by permission of John Wiley & Sons, Inc.

FIGURE I



POLYETHYLENE ($T_M \approx 120^\circ\text{C}$)



POLYPROPYLENE ($T_M \approx 175^\circ\text{C}$)

FIGURE II

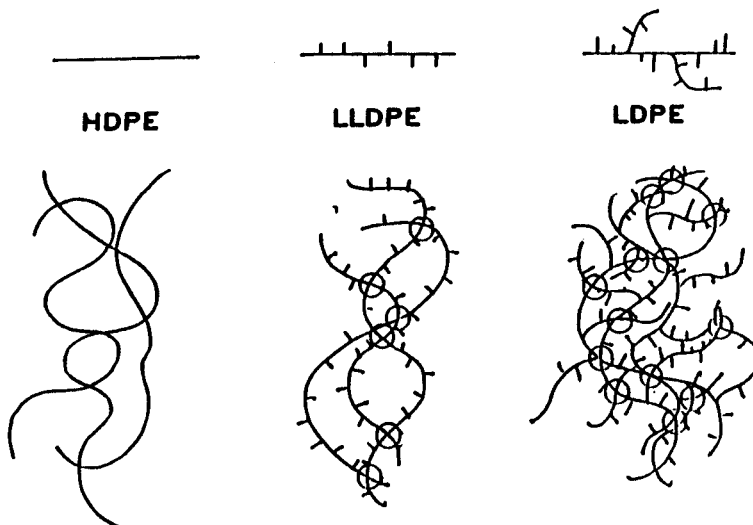
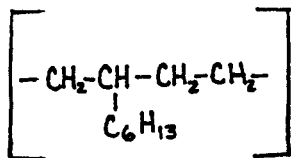


FIGURE III

SIDE BRANCH STRUCTURE OF ETHYLENE-OCTENE COPOLYMER



SIDE BRANCH STRUCTURE OF ETHYLENE-BUTENE COPOLYMER

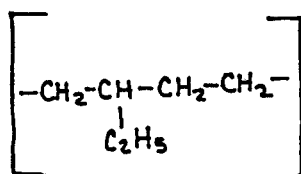
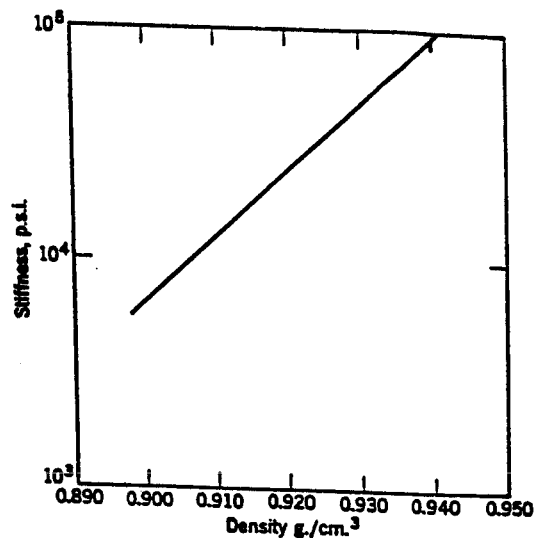
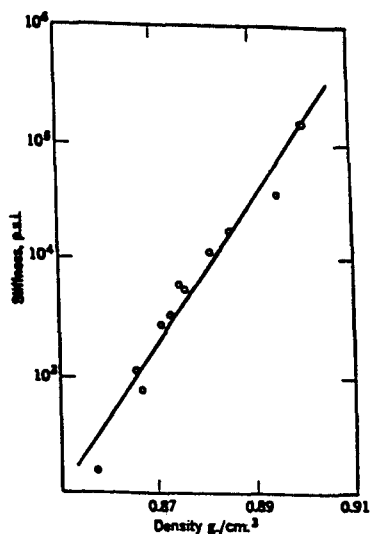


FIGURE IV



POLYETHYLENE STIFFNESS VS. DENSITY
 ("SOURCE: J. AM. CHEM. SOC., 1953, 75, 6129. COPYRIGHT
 1953 AMERICAN CHEMICAL SOCIETY.")

FIGURE V



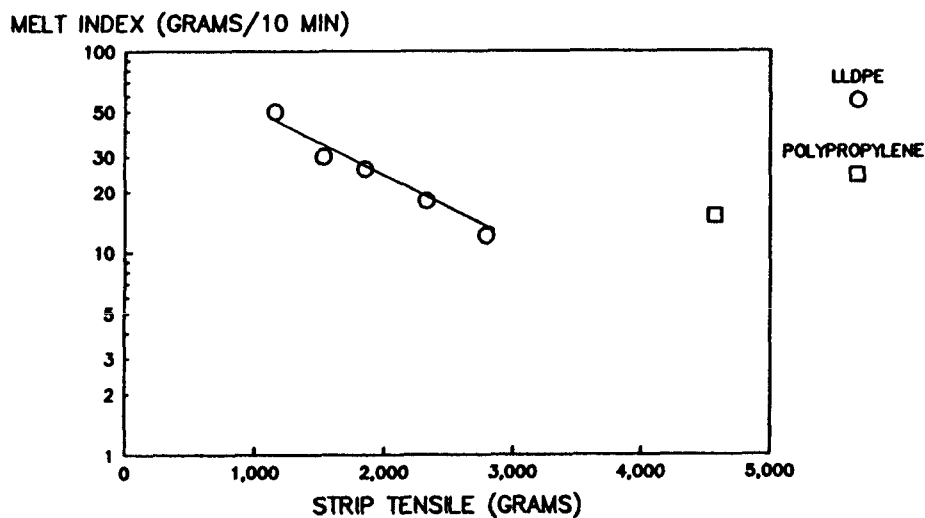
POLYPROPYLENE STIFFNESS VS. DENSITY

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TABLE I

ULTRA LOW DENSITY POLYETHYLENE (<0.910 GRAMS/CUBIC CENTIMETER)
LOW DENSITY POLYETHYLENE ($0.910 - 0.925$ GRAMS/CUBIC CENTIMETER)
MEDIUM DENSITY POLYETHYLENE ($0.926 - 0.940$ GRAMS/CUBIC CENTIMETER)
LINEAR LOW DENSITY POLYETHYLENE (DENSITY RANGE OF LDPE AND MDPE)
HIGH DENSITY POLYETHYLENE (>0.941 GRAMS/CUBIC CENTIMETER)

FIGURE VI
EFFECT OF LLDPE RESIN MELT INDEX
ON FABRIC STRENGTH



FIBER IS 2.5 DPF, 1.5 INCH LENGTH, UNCRIMPED
FABRIC BASIS WEIGHT APPR. 1.0 OZ/SQ. YD
PRESSURE CONSTANT @300 PLI

SPK:5/27/88

FLUID TRANSPORT CHARACTERISTICS OF ABSORBENT STRUCTURES

J. E. Rice

Abstract

The paper develops modified versions of traditional Lucas equations for use in predicting the flow of fluid in fibrous matrices. A fundamental analysis of the forces involved in the flow of fluids in low density absorbent structures coupled with a critique of the simplifying assumptions used with the Lucas equations has led to a mathematical model which better predicts the transport performance of low density structures. The theoretical basis is discussed and actual wicking data are presented as evidence.

I. INTRODUCTION

The theoretical approach to capillary rise in fibrous materials dates back to R. Lucas in the early 1900's [1]. The quantity of material published on the topic since that time is considerable. The frequency at which Lucas is referenced in these works gives testimony to the importance of his derivations. The purpose of this work is to explore a relatively simple modification to the classic Lucas derivations. It is confined to the specific case of absorbent fibrous structures at low network densities. It is intended to provide greater insight into the phenomenon of vertical wicking in these structures.

II. NOTATION

A	cross sectional area
C_f	fiber coarseness, weight per unit length
D	distance between fibers in a matrix
d_f	diameter of a fiber
F	force
g_o	gravitational constant
h	vertical height
h_o	maximum or equilibrium height of capillary rise
h_T	length of a pore
k	constant
L	length of solid-liquid-vapor interface
N	number of pores
P_f	perimeter of a fiber

p	pressure
r	radius of a capillary
r_e	effective radius of an irregular pore
r_h	hydraulic radius
t	time
V_f	fiber volume
V_p	pore volume
W_f	weight of fibers
γ	surface tension of liquid
η	viscosity
Θ	contact angle between liquid and solid at interface
ρ	density of the liquid
ρ_n	density of a fibrous network

III. LUCAS DERIVATION

Lucas observed that the forces acting on a fluid inside a circular tube consist of the surface tension term and a gravitational term. The magnitude of the surface tension term is "determined by the length of the cross section ($2 \cdot r \cdot \pi$) times the surface tension". The force is therefore proportional to the capillary radius r . The direction the force is acting is determined by the angle of contact with the solid such that the magnitude of the vertical component is proportional to the cosine of the angle. The gravitational term is determined by the weight of fluid supported. He went on to note that the maximum height (h_o) occurs when these two forces are exactly equal.

$$1. \quad 2 \cdot r \cdot \pi \cdot \gamma \cdot \cos \Theta = \pi \cdot r^2 \cdot \rho \cdot g_c \cdot h_o$$

$$2. \quad h_o = \frac{2 \cdot \gamma \cdot \cos \Theta}{r \cdot \rho \cdot g_c}$$

γ = surface tension of the fluid
 Θ = contact angle
 r = radius of the tube
 ρ = density of the fluid
 g_c = gravitational constant

The net force on the fluid at height h is given by the following:

$$F = 2 \cdot r \cdot \pi \cdot \gamma \cdot \cos \Theta - \pi \cdot r^2 \cdot \rho \cdot g_c \cdot h$$

Substituting from 1:

$$F = \pi \cdot r^2 \cdot \rho \cdot g_c \cdot h_o - \pi \cdot r^2 \cdot \rho \cdot g_c \cdot h$$

$$F = \pi \cdot r^2 \cdot \rho \cdot g_c \cdot (h_o - h)$$

Since pressure is force per unit area we can divide the above equation by the cross sectional area of the capillary to obtain the net pressure drop at height h .

$$p = \rho \cdot g_c \cdot (h_o - h)$$

Lucas substituted this expression in the Poiseuille formula for volumetric flow in circular tubes to derive the following equation for the rate of capillary induced flow:

$$3. \quad \frac{dh}{dt} = \frac{r^2 \cdot \rho \cdot g_c}{8 \cdot \eta} \cdot \frac{h_o - h}{h}$$

η = viscosity of the liquid

Integration resulted in:

$$4. \quad \ln \left[\frac{h_o - h}{h_o} \right] + \frac{h}{h_o} = - \frac{r^2 \cdot \rho \cdot g_c}{8 \cdot \eta \cdot h_o} \cdot t$$

Lucas went on to show that when $h_o \gg h$ (equivalent to dropping the gravity term) the integration yields:

$$5. \quad h \approx \sqrt{\frac{r \cdot \gamma \cdot \cos \Theta}{2 \cdot \eta} \cdot t}$$

In the case of large capillary radii h_o is small enough that the approximation given by equation 5 is not valid. In this case we must revert to the exact equation 4.

IV. LOW DENSITY FIBROUS NETWORKS

Wicking in low density fibrous networks primarily occurs in the open areas between the fibers. The wetting force exerted on a fiber partially immersed in a liquid can be expressed by the Wilhelmy equation [2]

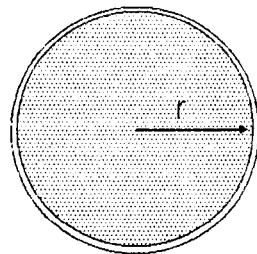
$$F = L \cdot \gamma \cdot \cos \Theta$$

where L is the length of the s-l-v interface. In the case of a fluid wicking inside a circular tube the length of the s-l-v interface is equal to the capillary circumference, $2 \cdot \pi \cdot r$. This leads to the formulas derived by Lucas. In fibrous networks the length of the interface is not equivalent to the pore circumference. For the sake of this derivation, we will assume an ideal network of fibers. All the fibers are of equal diameter d_f , parallel to the direction of flow and equally spaced.

Each fiber will be bounded by four pores, contributing 1/4 of its perimeter to each pore. A pore will be the area bounded by four of these fibers [3]. The length L of the s-l-v interface for each pore will therefore be $4 \times 1/4 P_f$ or

$$L = \pi \cdot d_f$$

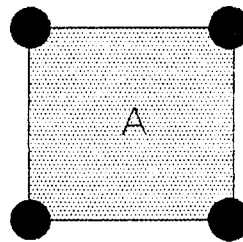
and the area will be expressed in terms of an effective capillary radius r_e (figure 1).



CAPILLARY

$$A = \pi \cdot r^2$$

$$L = 2 \cdot \pi \cdot r$$



NETWORK PORE

$$r_e \equiv (A/\pi)^{0.5}$$

$$L = \pi \cdot d_f$$

Figure 1. Cross sections of a capillary tube and an ideal fiber network pore.

The force balance for capillary rise now takes the form

$$F = \pi \cdot d_f \cdot \gamma \cdot \cos \Theta - \pi \cdot r_e^2 \cdot \rho \cdot g_c \cdot h$$

and the maximum vertical height h_o is

$$2i. \quad h_o = \frac{d_f \cdot \gamma \cdot \cos \Theta}{r_e^2 \cdot \rho \cdot g_c}$$

This treatment recognizes that wicking is a surface dependent phenomenon. It originates from the attraction forces at the molecular level between the liquid and the solid surface it is contacting. In the case of a true capillary, the length of the interface increases as the capillary radius increases. In low density fibrous networks this is not so if the pore size is increased by lowering the network density. The pores are larger but each is still bounded by four fibers for a total interface length of P_f .

V. EVIDENCE OF WILHELMY FORCES

In a fibrous network the density of the structure ρ_n is

$$\rho_n = \frac{W_f}{V_f + V_p}$$

where W_f is the weight of fibers and V_f and V_p are the volumes occupied by the fibers and the pores respectively. For low density structures where the pore volume is much greater than the solid volume

$$V_p + V_f \approx V_p$$

and

$$\rho_n \approx \frac{W_f}{N \cdot \pi \cdot r_e^2 \cdot h_T}$$

N is the number of pores
 h_T is the pore length

In our ideal structure the number and length of pores is constant. We can therefore combine W_f , N , π and h_T into one constant k where

$$k = \frac{N \cdot \pi \cdot h_T}{W_f}$$

Rearranging:

$$\frac{1}{r_e^2} \approx k \cdot \rho_n$$

We can substitute this equation in both 2 and 2i, combine constants and rearrange to obtain the predicted dependency of equilibrium height on network density. From the Lucas equation for h_o , 2, we obtain

$$6. \quad h_o \approx k_1 \cdot \sqrt{\rho_n}$$

and from the modified equation

$$7. \quad h_o \approx k_2 \cdot \rho_n$$

Equilibrium heights were determined experimentally at several different network densities to see which relationship, 6 or 7, actually applies. As can be seen in figure 2 the relationship between network density and equilibrium height fits the linear form predicted by the modified equation, not the exponential form derived from the Lucas equation. This supports the existence of a Wilhelmy force effect in these matrices, since that substitution is the only difference between equations 2 and 2i.

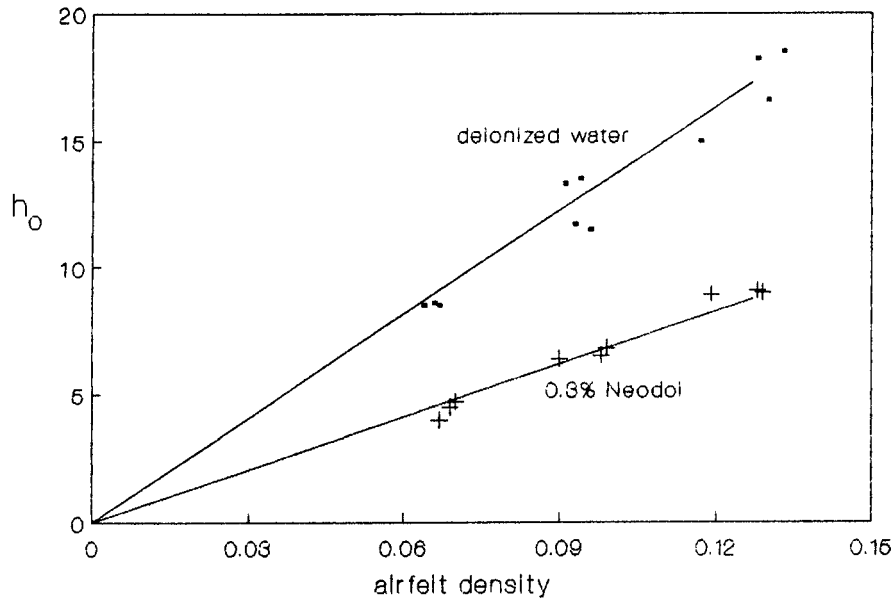


Figure 2. Equilibrium height vs. network density

VI. PORE SIZE

To test the reasonableness of equations 2 and 2i, pore sizes were determined by each and compared with values calculated from system geometry. To determine pore size from equation 2 or 2i one simply measures the equilibrium height for a surfactant solution (contact angle ≈ 0) and solves for r_e . To calculate the pore size from system geometry requires knowing the fiber coarseness, fiber diameter and network density. Boyd [3] derived the following formula relating the fiber diameter and distance D between fibers to the fiber coarseness C_f and structure density.

$$(D + d_f)^2 = \frac{C_f}{\rho_n}$$

Since $(D + d_f)^2$ is the average cross sectional area taken up by the pore and the fiber segments associated with it, the cross sectional area of the pore alone is simply this quantity less the cross sectional area of one fiber

$$8. \quad A = \frac{C_f}{\rho_n} - \frac{\pi \cdot d_f^2}{4}$$

and

$$r_e = (A/\pi)^{0.5}$$

For a southern slash pine kraft pulp typical values for the fiber properties of interest are:

$$\begin{aligned} d_f &\approx 30 \text{ to } 40 \text{ microns, use } 35 \\ C_f &\approx 28 \text{ to } 35 \text{ mg/100M, use } 32 \end{aligned}$$

Equilibrium height values were determined experimentally on airfelt samples of this fiber at different densities. The surfactant solution used was 0.3% Neodol in water with a surface tension of 28.1 dynes/cm. The density and viscosity are the same as water at that concentration. Table 1 compares the pore radii determined by both methods at different airfelt densities.

Table 1. Comparison of pore radii determined from equation 2i with reasonable values based on physical properties of the fibers and airfelt density.

airfelt density g/cc	h_o , cms.	pore size, r_o (microns)		
		Lucas(2)	Rice(2i)	Boyd(8)
0.050	5.4	106	43	42
0.056	5.7	101	42	39
0.059	5.9	97	41	38
0.074	8.1	71	35	33
0.075	8.0	72	35	32
0.084	8.2	70	35	30

From the results in table 1 we can see that the modified equation predicts reasonable values for pore size while the Lucas equation overestimates pore size.

VII. RATE OF CAPILLARY RISE

The relationship between capillary height, equilibrium height and time derived by Lucas (equation 4) is unchanged. What has changed is the estimate of the equilibrium height. It is not clear that Poiseuille's equation is valid for a fibrous network structure since it is based on a very specific geometry. Certainly the estimation of frictional head loss should be highly dependent on the geometry of the pores. How well Poiseuille's equation predicts capillary rise rates can be determined experimentally by plotting actual height and time data against those predicted by equation 4. Figures 3 and 4 show results for a southern pine kraft fiber at two different airfelt densities. Capillary radii were predicted from the physical properties of the fibers taking into account differences between summerwood and springwood fibers. Actual equilibrium height data were used to generate the theoretical curves.

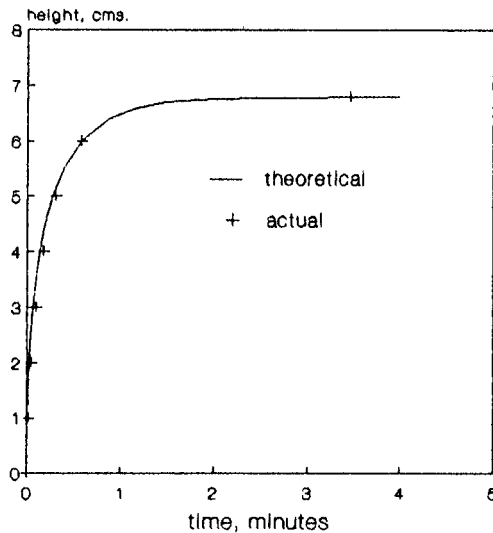


Figure 3. Capillary rise. Airfelt density = 0.05 g/cc.

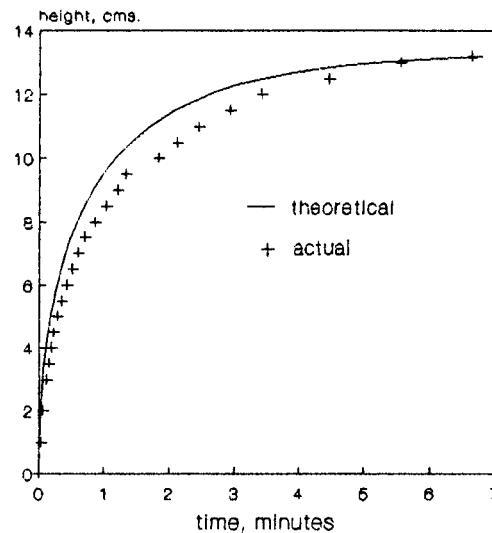


Figure 4. Capillary rise. Airfelt density = 0.092 g/cc

At low density the actual data agrees quite well with values predicted by Poiseuille's equation (figure 3). At higher densities though we begin to see some deviation from theory (figure 4). This deviation appears to worsen as pore size is reduced through densification or substitution of finer fibers. It is therefore prudent to restrict the use of equation 4 to low density structures with relatively large capillary radii.

VIII. DETERMINING APPARENT CONTACT ANGLES

It should be possible to use equation 2i to determine contact angles experimentally if we know the values of the other terms. The surface tension and density of the fluid are easily obtained. The pore radius and fiber diameter can be much more difficult to determine, particularly when working with natural fibers such as cellulose woodpulp fibers. Fortunately, only the ratio of the pore area to the fiber circumference is needed.

$$\frac{r_o^2}{d_f} = \frac{\pi \cdot r_o^2}{\pi \cdot d_f}$$

This ratio can be determined experimentally from equilibrium height data for a surfactant solution. We assume the contact angle for the surfactant solution is near zero so $\cos \Theta \approx 1$. Equation 2i reduces to:

$$h_o = \frac{d_f \cdot \gamma}{r_o^2 \cdot \rho \cdot g_c}$$

rearranging:

$$9. \quad \frac{r_e^2}{d_f} = \frac{\gamma}{\rho \cdot g_e \cdot h_e}$$

Once this ratio is known the apparent contact angle for a fluid of interest can be determined by measuring its equilibrium height at the same structure density and solving equation 2i for Θ .

$$10. \quad \Theta = \cos^{-1} \left[\frac{r_e^2}{d_f} \cdot \frac{\rho \cdot g_e \cdot h_e}{\gamma} \right]$$

For simplicity, equations 9 and 10 can be combined.

$$\Theta = \cos^{-1} \left[\frac{\gamma_s}{\gamma_l} \cdot \frac{h_{e,l}}{h_{e,s}} \right]$$

The subscripts *s* and *l* have been added to distinguish surfactant solution values from those for the other liquid.

Table 2 contains experimental values for the contact angle of deionized water with a fully bleached southern pine kraft fluff pulp. As expected, the values obtained are independent of the airfelt density.

Table 2. Experimental values for Θ for a southern pine kraft fluff pulp.

airfelt density g/cc	$\frac{\gamma_s}{\gamma_l}$	$\frac{h_{e,l}}{h_{e,s}}$	contact angle, degrees
0.035	0.40	1.72	47
0.056		1.37	57
0.060		1.53	52
0.072		1.48	54
0.076		1.51	53
0.084		1.56	51
average:		1.53	52

The greater variability in results at very low density is probably due to height measurement errors. At those densities the liquid front is more difficult to determine. Errors in measurement coupled with lower absolute values result in greater variability in the equilibrium height ratios.

Aberson [4] has reported contact angles of about 60 degrees for kraft pulps using wicking rate measurements. Hodgson and Berg [5] obtained values of about 23 degrees for similar fibers using advancing and receding force measurements on individual fibers. The

disagreement with the results of Hodgson and Berg is puzzling since the Wilhelmy wetting force is the basis for their measurements as well. It may be that fiber orientation is affecting the equilibrium height of the structure. The Wilhelmy force is describing force in the direction of the solid surface. The wicking height is dependent on the vertical component of that force. In the case of an ideal structure where all the fibers are oriented vertically the Wilhelmy force is the vertical force. If the fiber surface is inclined α degrees from vertical the vertical component of the Wilhelmy force will be the cosine of the difference of the angles (α may be positive or negative):

$$\Theta_o = \Theta - \alpha \quad [6]$$

Θ_o is the observed contact angle (eq. 2i)
 α is the angle between vertical and the fiber surface at the s-l-v interface.

In the structure tested the fibers were randomly oriented such that most of the fibers had some orientation other than vertical. If the contact angle between the fiber and water is 23 degrees then the maximum enhancement of the vertical force is the difference between 23 and 0 degrees while the maximum detriment is the difference between 23 and 90 degrees. The effects of inclination may not cancel each other out.

IX. PREDICTING CAPILLARY RISE

It should now be possible to predict the influence of certain variables on the rate and ultimate height of capillary rise using equations 2i, 3 and 4. Two variables we can influence in the structure are pore size and fiber diameter. Figures 5 and 6 show the predicted influence of each on capillary rise.

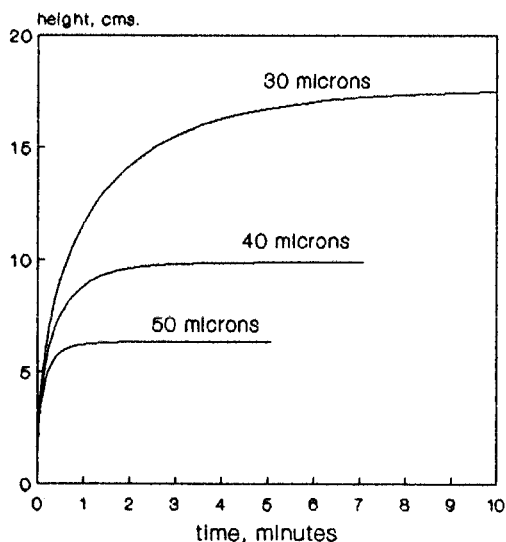


Figure 5. Effect of pore radius on capillary rise. $d_f = 35$ microns

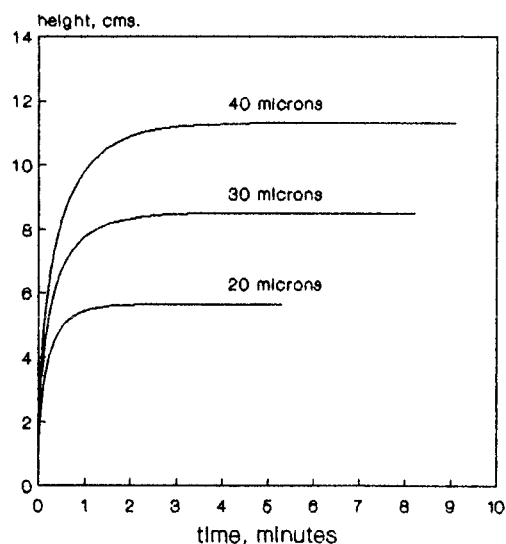


Figure 6. Effect of fiber diameter on capillary rise. $r_p = 40$ microns

The fiber diameter is controlled by the selection of the fibers. The pore size can be controlled in two ways. The density of the structure can be altered or fibers of greater or less coarseness can be selected. (A coarser fiber has fewer fibers for the same weight resulting in fewer but larger capillaries for a given airfelt density.) Figures 7 and 8 show the predicted effects of airfelt density and fiber coarseness on capillary rise. The results in figure 8 are based on the assumption that the fiber diameter is constant. This can occur when differences exist in cell wall thickness such as exist in summerwood and springwood fibers.

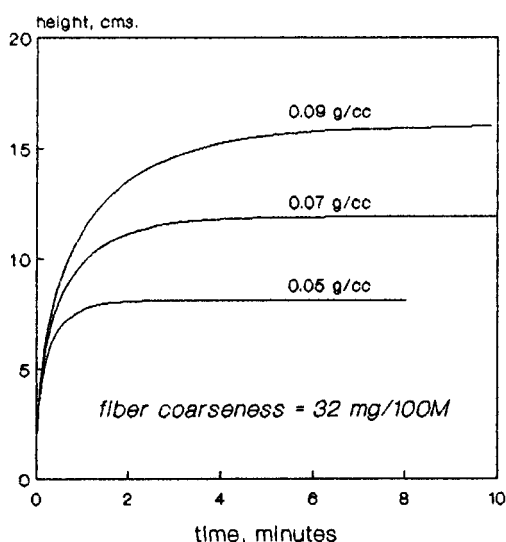


Figure 7. Effect of airfelt density on capillary rise.

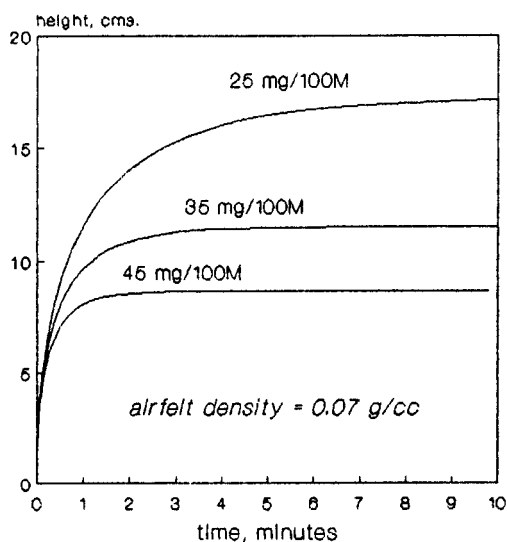


Figure 8. Effect of fiber coarseness on capillary rise.

If we fractionate a southern pine kraft pulp into summerwood rich and springwood rich fractions the resulting two samples would have very different coarseness values but somewhat similar diameters. This model predicts that at equal airfelt density the springwood fraction should wick fluid higher than the unfractionated fiber and the summerwood fiber should not wick as high as either. Furthermore the rates of capillary rise should all be roughly equal in the early stages with the summerwood fraction falling off first. This is in contradiction to the Lucas equations which predict the larger capillary radii wicking faster initially. Figure 9 shows actual height and time measurements on a fractionated sample.

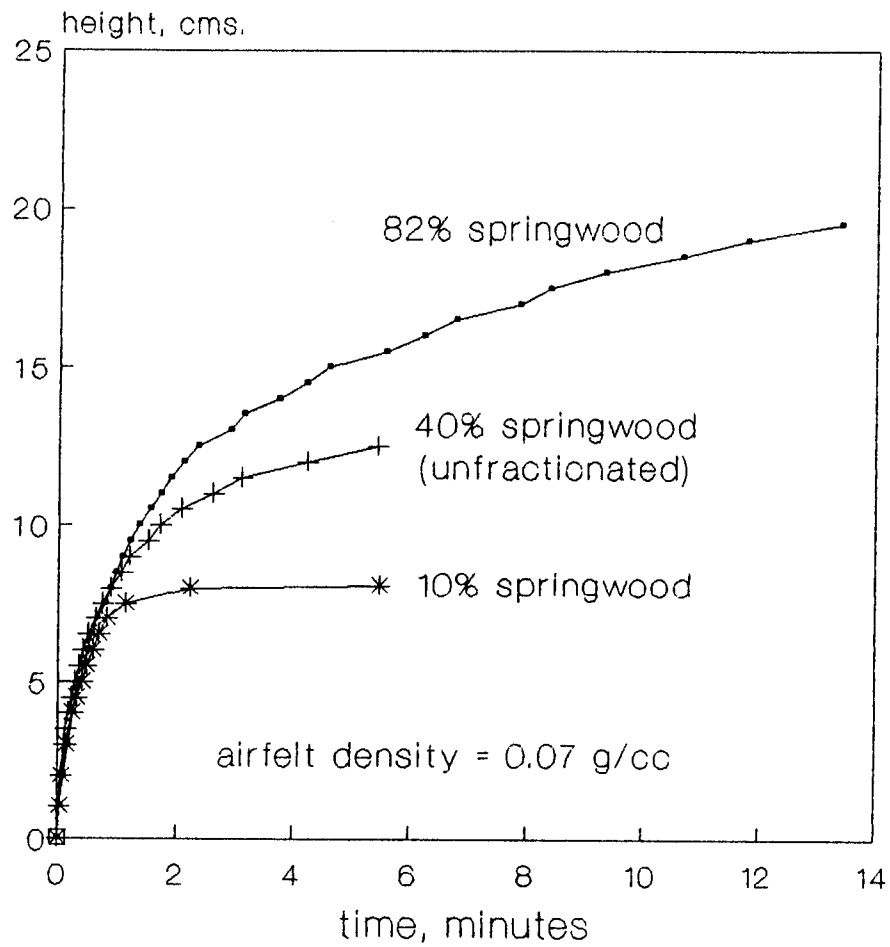


Figure 9. Effect of fractionation on capillary rise.

X. CONCLUSIONS

Capillary rise in low density fibrous structures is attributable to Wilhelmy forces and approximated by Lucas' equation for rate in terms of equilibrium height. The equations for equilibrium height (h_0), velocity at height h (dh/dt) and time to reach height h are:

$$2i. \quad h_0 = \frac{d_f \cdot \gamma \cdot \cos \theta_0}{r_e^2 \cdot \rho \cdot g_c}$$

$$3. \quad \frac{dh}{dt} = \frac{r^2 \cdot \rho \cdot g_c}{8 \cdot \eta} \cdot \frac{h_0 - h}{h}$$

$$4. \quad \ln \left[\frac{h_0 - h}{h_0} \right] + \frac{h}{h_0} = - \frac{r^2 \cdot \rho \cdot g_c}{8 \cdot \eta \cdot h_0} \cdot t$$

The equilibrium height may depend on the fiber orientation as well as the contact angle between the fiber surface and the advancing liquid. Simply stated, the height is governed by the quantity of liquid each fiber is required to support and the vertical component of the Wilhelmy wetting force. The validity of the equation should be independent of pore size.

The rate equations are based on very specific geometry. They assume round straight continuous walled capillaries. They seem to be predictive of fibrous networks with large pores only. They overestimate the rate of capillary rise in nonideal networks of small capillary radii. Deviation from theory is significant at densities above 0.09 g/cc.

XI. LITERATURE CITED

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APPENDIX

Experimental Procedures

Pulp samples were mechanically defibrated and airlaid into 14"x14" pads at a basis weight of approximately 360 g/M². Test strips were then cut from the pad and pressed with a laboratory press to densities slightly below the desired values. They were then trimmed to final dimensions of 2.5 centimeters wide by 30 centimeters long and weighed prior to testing. Observations were made with the sample held in a holder such that the sample width and thickness were held constant. The sample holder consisted of two clear plexiglass plates 5 centimeters wide by 30 centimeters long. Shives 5 centimeters wide by 27 centimeters long were placed between the plates at each side to control the distance between the plates. This left an open area between the plates 2.5 centimeters wide by 30 centimeters long with the thickness set by the shives. The sample was placed in this area and the holder held together with clamps. A centimeter scale was attached to the front plate to measure heights during capillary rise. To initiate capillary rise the lower end of the sample and holder were lowered into a liquid reservoir to a depth of 2.5 centimeters. The holder was held in position with a buret clamp mounted to a stand and a florescent light was positioned behind the holder to aid viewing of the advancing liquid front. A timer was started when the end of the sample was placed in the liquid and times were recorded at either .5 or 1.0 centimeter increments until wicking stopped. The final height was also recorded. The densities of the samples were determined from the air dry weight of the samples and the height (30 cms.), width (2.5 cms.) and thickness.

Samples with a high degree of vertical fiber orientation were prepared on a dynamic handsheet former and tested in a similar manner. The densities of the sheets were about 0.16 g/cc which required longer sample strips. The same approach was taken to testing these with the sample holder scaled up in length.

THE EFFECT OF FLUID CHARACTERISTICS ON ABSORBENCY IN NONWOVENS

by

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ABSTRACT

In this study, the role the fluids play in absorbency was investigated. Fluids chosen varied broadly in their physical and chemical properties. They were water, salines, ethanol, formamide, hexadecane and dimethyl phthalate. Two synthetic body fluids, namely, urine and menstrual fluid, were also constituted and studied. Each of these fluids was characterized as to pH, density, viscosity, surface tension and contact angle.

Airlaid, needle punched fabrics composed of rayon and polypropylene fibers were used as the substrates for absorbency. The variables which characterized fabric structure were the constituent fiber (rayon, polypropylene or the blend thereof), fiber denier, needling depth and the environmental pressure. Two parameters were assessed, namely the absorbent capacity and the rate of absorbency.

The results obtained were compared with predictions from models based on classical theories. Results clearly indicated that properties of both the fabric and the fluid governed the absorbent behavior. While the capacity depended mostly on the factors in the fabric structure, the rate was greatly affected by both the properties of the fluid and the structure of the fabric.

INTRODUCTION

Absorbency is the result of absorbate-absorbent interaction. To optimize the performance of an absorbent product, this interaction must be optimized. A review of the literature, however, indicates that much of the research in this area has dealt with the composition and the structure of the product, and relatively little attention has been paid to the fluids which must be absorbed into them. In most product developments, the fluids generally used have been water or dilute saline solutions. These do not represent body-like fluids in either the chemical constitution or the physical characteristics. Also, the medical literature shows that the composition of the body fluid is not constant but varies from person to person, and with the dietary habits and the age of individuals [1,2]. A detailed study is thus needed in which the role the fluids actually play in absorbency is investigated.

The fluids chosen in this research varied in properties over a broad range so that a systematic study could be developed. Surface tension, contact angle, pH and viscosity were the properties considered. These were measured at room

temperature (25°C) as well as the body temperature (37°C) to enable us to assess the changes in properties with temperature. In addition to a number of standard fluids, two fluids with body-like characteristics were also developed. These were the synthetic menstrual fluid and urine. The fabrics used in the study were composed of blends of rayon and polypropylene, the latter of three different deniers. These were needle punched to different depths and tested for absorbency with the above fluids under different environmental pressures.

EXPERIMENTAL

Fluids

The fluids chosen for this investigation included three different sets. The first set contained commercially available compounds of good purity (89.9–99.9%). These included dimethyl phthalate, ethanol, formamide, hexadecane, commercial saline and water. They are shown, ordered by ascending surface tension values, in Table I. These values came from the chemical literature [3,4,5].

In order to further vary the properties and observe their effects, two chemicals were chosen for mixing with water in a second set of fluids. This series was formed by diluting formamide and ethanol with water in volume ratios of 75/25, 50/50 and 25/75. Formamide and ethanol were chosen due to their solubility in water and the observable difference in their surface tensions from that of water. This was not the case with hexadecane and dimethyl phthalate which were immiscible with water, and the solvents in which they could be mixed had nearly the same values of surface tension as did the chemicals themselves. Included in this set were also salines which have generally been used as fluids in invitro studies. Sodium chloride solutions of 1% to 5% concentration were formulated. The commercial 0.7% saline solution included in the first set and listed in Table I was different in that it was buffered and contained preservatives.

Two synthetic body fluids, urine and menstrual, were also formulated. The recipes for these were selected after a review of the patent literature [6,7,8,9] and discussions with researchers in the industry. The details of these fluids are given in Table II. The artificial urine was simply agitated after the addition of the various salts. The synthetic menstrual fluid was heated slightly and continuously stirred in order to dissolve the carboxymethyl cellulose. Both samples were filtered using an aspirator to remove any undissolved particles.

Surface Tension Measurements

Surface tension of fluids was measured on the Fisher Scientific Model 215 Autotensiomat® Surface Tension Analyzer using a Du Nouy Ring which traversed at a rate of 0.2" per minute. The apparent value so obtained was converted into absolute value using standard procedures.

Contact Angle Measurements

Contact angle in this investigation was measured using a Ramé-Hart contact angle goniometer telescope. A 2-3 μ l droplet of fluid was released onto a flat substrate by means of a micro-syringe. Four model substrates, Teflon®, acetate, Mylar® and a glass, were used in these measurements. This allowed the study of the wettability properties of various fluids on several different substrates. Contact angle values after an initial period of spreading are reported. All measurements were carried out at room temperature.

pH Measurements

pH measurements were made using the Fisher Scientific Occumet pH Meter Model #630.

Density Measurements

The density of the fluid mixtures was measured by carefully weighing a given volume of each sample. The density value of pure compounds was obtained from the chemical literature.

Viscosity Measurements

Measurements of viscosity were made using a Cannon-Fenske Opaque (Reverse Flow) Capillary Viscometer and a constant temperature bath.

Tests were made on the three formamide/water mixtures, the three ethanol/water mixtures, the six sodium chloride solutions and on the two synthetic body fluids. The viscosities of the undiluted samples were taken from the literature [10,11] and are shown in Table I. These measurements were made at a temperature of 25°C.

Nonwoven Fabrics

Airlaid nonwoven fabrics, composed of 100% rayon, 100% polypropylene and 50/50 blend of polypropylene and rayon, were made using a Rando-Feeder Rando-Webber system. The rayon fibers used were 3 denier with a 2 inch staple length. The polypropylene fibers were 2 denier, 3 denier and 4 denier, with staple lengths of 1.5", 2.0", and 3.25", respectively. The polypropylene fibers had a small amount of hydrophilic finish (0.43-0.75%).

Webs produced by the Rando-Webber system had a weight of about 4 oz/yd². Three such webs were superimposed and cut into strips. Each strip was needle punched on a laboratory model James Hunter needling machine. A total needling density of 244 needles/in² was achieved after two passes through the machine. The depth of needle penetration was varied over two levels. In the lightly needled webs, one set of barbs of the needle penetrated through the thickness of the fabric, while in the deeply needled webs, all three sets of barbs penetrated through the fabric.

Absorbency Measurements

Test samples were die cut from each fabric using a circular die 1.25" in diameter. Each sample was individually weighed. Absorbency was measured on the Gravimetric Absorbency Testing System (GATS) [12,13] manufactured by M/K Systems (Figure 1). This instrument gives a digital readout of the weight (in grams) of fluid absorbed into the sample. In order to operate the GATS, the filling reservoir is filled with approximately 600 milliliters of test fluid. This reservoir fills the fluid test chamber by means of a plastic tubing which is opened or closed by a valve. An electronic balance controls the amount of fluid passed into the fluid test chamber. Testing begins when the digital readout measures a loss of fluid from the test chamber and testing ends when the readout reaches a peak steady value. The height of the sample holder is adjusted with respect to the level of the fluid in the chamber in order to achieve the desired hydrostatic head of pressure. In the present work, a zero hydrostatic head was used.

Calculations of absorbent capacity (C , grams fluid/grams dry fabric) were made by dividing the mass of fluid absorbed by the mass of the dry sample. Calculations of the rate of absorbency (Q , grams/gram-second^{1/2}) were made by dividing the value of C so obtained by the square root of the absorbency time.

Tests were made using two different environmental pressures, namely 9 and 22 gf/cm², obtained by placing fixed weights on the die cut samples. Sample thickness was measured during absorbency tests with the assistance of a cathetometer. The values obtained must, however, be considered approximate since the resolution of the scale on the telescope vernier was low for the thickness of the fabric specimen.

RESULTS AND DISCUSSION

Surface Tension Results

Surface tension is an important property which plays a significant role in the absorbency of fluids. In addition, the wettability behavior of the liquids also affects their absorbency behavior. High surface tension in the liquid is a favorable condition for capillary rise. Low surface tension in the liquid is favorable for spreading on a given plane surface. Evaluation of the surface tension and spreading characteristics of a given liquid can help in devising systems with optimum absorbency characteristics. Thus the surface tension and spreadability of several model systems were measured in this study. The results are described in the following paragraphs.

The values of surface tension are given in Table III. A close agreement is seen between the experimentally determined values and those reported in literature (Table I) except in a few cases. The experimentally determined value of water is somewhat lower than that given in the literature. This may be due to the presence of impurities in the fluid. The effect on surface tension of mixing water with ethanol and with formamide in various volumetric ratios are shown in Figures 2 and 3.

The surface tension of a given liquid was lower at 37°C than at 25°C, as was expected. However, the change in the surface tension with mixing ratio was not linear. Water/ethanol mixtures show a particularly non-linear behavior. For example, the 50/50 mixture has a value which is close to that of the 100% ethanol. This is due to the ethanol concentrating preferentially at the surface of the sample solution which could be expected due to ethanol's lower surface tension and free energy.

Surface tension of saline solution (Table III) gradually increased as the sodium chloride concentration increased, a result which has also been seen in the chemical literature. The commercial saline showed a markedly lower surface tension value than did the other salines. This may be due to the buffers added to the former to make it more acceptable to the human body, which may be surface active.

Surface tension values for synthetic urine and synthetic menstrual fluid are also included in Table III. Synthetic menstrual fluid shows a higher surface tension value than does its main component, blood (55.5-61.2 mN/m). This is in line with the value of the natural menstrual fluid, which is also believed to be higher than that of blood.

Contact Angle Results

The contact angle values of various liquids on glass, Teflon®, cellulose acetate and polyester Mylar® are given in Table IV. For a given substrate, the value of θ varies from fluid to fluid, with urine and menstrual fluids having the highest values. For any given liquid, the value of θ differs from substrate to substrate with glass having a minimum and Teflon® a maximum value. This shows that the surface behavior of the material plays an important role and if it was well characterized and its interaction with fluids understood then its properties could perhaps be modified to achieve optimum results with a given fluid.

It is noted, by comparison of results in Tables III and IV, that for a given substrate, the higher the surface tension of a liquid, the higher the contact angle and, thus, the lower the spreadability. A plot of the cosine of the contact angle (θ) against surface tension is shown in Figure 4 for glass. This plot clearly demonstrates a direct relationship between these two properties. It is noted that the overall plot is not linear. However, a straight line could be approximately drawn through points belonging to a specific homologous series, such as salines, ethanol/water mixtures or formamide/water mixtures. The scatter of data in this plot can arise from the presence of (1) impurities in standard liquids and (2) additives in the synthetic fluids.

pH Results

Recent work by a number of authors has established the importance of acid-base interaction between a fluid and a substrate in determining the absorption phenomenon in polymers. For example, a polymer containing a carboxylic acid group would favorably interact with a liquid containing a basic group such as an amine, or vice versa.

Examination of the values of pH in Table V shows that these fluids cover a range from 4.25 to 9.95. However, most fall below 7.0 emphasizing acidity. The pH of mixtures of ethanol and water fell between those of undiluted ethanol and water. Interestingly, this was not found to be the case for mixtures of formamide and water. Since formamide acts as an acid in the presence of water, pH values for these mixtures were lower than those for either of the pure compounds.

The pH of saline solution decreased with increase in sodium chloride concentration. The commercial saline showed a lower pH than did the 1% solution. This difference was most probably due to the preservatives added to the former. The pH of synthetic urine was 6.49 which fell in the range 4.18-7.8 given in the medical literature for natural urine [2]. This wide range for the latter was largely due to the influence of a person's diet on this property. The pH of the synthetic menstrual fluid was 9.95 which is significantly higher than that reported in the medical literature for the whole blood of adults (7.31-7.40) [2].

It would be of great interest to know the role pH of a fluid plays in determining absorbent characteristics of a material. This effect, however, could only be properly evaluated if the polymers used in the absorbency experiments also possessed a wide range of acid-base characteristics.

Density Results

The results of the density measurements can be seen in Table VI. The density of a fluid coupled with the volume of the interstitial space in a fabric act together to determine the absorbent capacity of the structure in the units of mass.

Dimethyl phthalate showed the highest density of the fluids tested followed by formamide and synthetic menstrual fluid. Ethanol showed the lowest value. Among the laboratory saline solutions, density increased slightly as the amount of sodium chloride increased. As the proportion of formamide increased in its mixtures, so did the density. The same was not true for the ethanol mixtures which were basically unchanged as the volume of ethanol increased.

Density values for human whole blood, 1.055 g/ml, compared favorably with that of the synthetic menstrual fluid [2]. The value of synthetic urine also fell in the range generally found to hold for human urine (1.00 to 1.10 g/ml). This wide range is again attributed to the variation in the dietary habits of the individuals.

Viscosity Results

One of the most important of the fluid properties which greatly affects absorbency is the viscosity. Among all characteristics, this parameter often proves to have the dominant effect. According to Washburn's equation, the rate of fluid uptake is inversely proportional to the square root of the viscosity. Measurements of this property along with those of others, discussed earlier, could facilitate the prediction of the absorbency rate.

The values of viscosity are given in Table VII. A twenty-fold range is seen among the values, with dimethyl phthalate having the highest and water the lowest values. The synthetic menstrual fluid gave the second highest value. An interesting result is seen when ethanol and water are mixed together, i.e. the viscosity of the mixture is higher than that of the pure fluids. This is the effect of a bonding reaction between the two liquids. Mixing of formamide with water did not produce this effect, the value of the mixture lay between those of the compounds.

One major effect of the addition of salt to water is seen to be an increase in the viscosity, an addition of 1% salt giving a 15% increase. Viscosity continued to increase further but only slightly with increase in salt concentration. The viscosity of the commercial saline was much higher than those of the other salines.

The synthetic urine gave a viscosity in the range of those of the salines while the artificial menstrual fluid gave the much higher value of 8.55 cp. Medical literature shows the viscosity of blood to be about 1.6 cp [2]. Viscosity is seen to be highly temperature sensitive. Values measured at body temperature (37°C) are significantly lower (~ 30%) than those measured at room temperature. This result should be taken into consideration when attempting to model the absorbent characteristics of personal hygiene products which involve fluids at near body temperature.

Absorbency Results

The results of absorbency tests on nonwoven fabrics are given in Tables VIII to XI. Considering first the absorbent capacity, it is noted from Table VIII, which gives results for one needling depth and environmental pressure, that the denier of the polypropylene fiber did not produce a noticeable effect on the value of capacity. Polypropylene fiber, however, consistently gave a higher capacity than rayon. This shows that the webs made from the former maintained a higher interstitial volume than those containing the latter under all conditions of the test. Although mechanical tests on fibers were not conducted in this investigation, this result should come from rayon either having a lower modulus than polypropylene, or, as is highly likely, losing a fraction of its modulus when coming in contact with various fluids, or both. The initial modulus of rayon is known to undergo a substantial decrease when it is brought in contact with aqueous fluids, the category in which the majority of the test fluids fell. Blends gave values which lay between those of the single fiber webs; however, no difference is noted between blends containing different deniers of polypropylene. In Table IX, the results of capacity are presented for different fabric structures but for polypropylene fiber of one size, namely 4 denier.

The results show that higher needling depth gave appreciably higher capacity values. This is an interesting result and the reasons should lie in the fact that the higher needling depth was expected to lead to web structure containing channels which were more rigid and which resisted collapse under pressure to a greater degree. A lower environmental pressure gave substantially higher value of capacity, obviously due to lower compression of fabric and thus higher residual volume fraction of interstitial space for holding a fluid. Some differences in absorbent capacity are noted among

different fluids. Generally, dimethyl phthalate and formamide gave the highest values while synthetic menstrual fluid gave the lowest value. The addition of salt to water resulted in a decrease in the value; the greater the salt concentration, the lower the capacity.

As a general hypothesis, it should be clear that absorbent capacity as defined in this work is a function of the interstitial space between the fibers and the density of the fluid. Ignoring the amount of fluid that may be absorbed within the structure of the fiber, one could estimate absorbent capacity using the following relation [15]:

$$C \text{ (theoretical)} = V_s \cdot \rho_\ell = \left(\frac{AL - V_F}{M} \right) \rho_\ell$$

where V_s = the total pore volume

ρ_ℓ = fluid density
 A = sample area
 L = sample thickness
 V_F = total fiber volume
 M = fabric mass

The following equation can be used for obtaining the value of the fiber volume, V_F :

$$V_F = M \left(\frac{m_1}{\rho_1} + \frac{m_2}{\rho_2} \right)$$

where m_1 = mass fraction of fiber 1 in the blend
 m_2 = mass fraction of fiber 2 in the blend
 ρ_1 = density of fiber 1
 ρ_2 = density of fiber 2

As mentioned earlier, a set-up was used which allowed the measurement of the value of the fabric thickness during absorbency tests. The value of fabric thickness along with the values of the other parameters was substituted in the equation of C , the capacity, to estimate the latter for various fabric structures and fluids. The predicted and the measured capacities generally fell quite close to each other. An example of this comparison is given in Figures 5-7 in which the results for water as the fluid are presented. In spite of the scatter in the results, it is seen that there is a good agreement between the two. The scatter in the values can result from (1) a variability in the structure of the fabric which could lead to a variability in the degree of saturation of the fabric, and (2) lack of precision in the measurement of fabric thickness.

Tables X and XI give the results of absorbency rate. From Table X, which gives results for one needling depth and environmental pressure, but for different fibers, fiber deniers and blends, it is clearly seen that for any given fluid the rate of absorbency is not affected by the fiber type, fiber denier or the blending of fibers of two different types. Although in the

literature, hydrophilic fibers have generally been shown to give a higher rate of absorbency than hydrophobic fibers [16], the presence of hydrophilic finish on the latter could be expected to minimize this difference. The data presented in Table XI illustrates the effect of the other factors, namely the needling depth and the environmental pressure - the factors which govern the structure of the fabric during absorption - and the fluid type. Increasing the needling depth gave a consistent increase in the rate obviously due to more rigid and better defined flow channels existing in the more deeply needled fabrics. A decrease in environmental pressure gave a substantial increase in the rate. This should be clearly due to the less distorted and more oriented channels expected in the direction of flow with lower pressure. For any given fluid, an enhancement in the rate of 50% is seen with change in the fabric structure for most fluids. In some cases, such as the dimethyl phthalate and the menstrual fluid, the improvement is even bigger. However, comparatively the greatest effect on the rate of absorbency is produced by the differences in the properties of the fluids. Irrespective of the fabric structure, generally ethanol gave the highest value, followed by formamide, water, hexadecane, synthetic urine, salines, synthetic menstrual fluid, and dimethyl phthalate, in that order.

This last fluid reacted poorly to testing in the GATS apparatus. It frequently clogged in the fluid valve which caused difficulties in resetting and restarting of the apparatus. This behavior was most certainly due to the viscosity of this fluid which was the highest of all fluids.

The rate of absorbency showed a dependency on the amount of salt present. As the concentration of sodium chloride increased, the rate decreased. This trend is well supported by the results in the literature. When compared with water, the rate of absorbency of salines was much lower. This was also true for the commercial saline solution.

Synthetic urine gave a slightly lower rate of absorbency than did water which could also be due to the presence of salt in the former. The synthetic menstrual fluid gave a low value, the second lowest to that of the dimethyl phthalate. An expected reason for this also is its viscosity which was the second highest to that of dimethyl phthalate.

In absorbency tests, the rates are usually recorded as fluid uptake divided by the square root of time. This practice has been based on the work of Washburn [17]. The integrated version of his equation predicts the following dependency:

$$S^2 = \frac{r T_{lv} \cos \theta}{2\eta} t$$

In this, S is the distance to which the penetration extends in time t, r is the effective radius of the capillaries and η is the coefficient of viscosity. The rate, thus, is given by

$$\frac{S}{\sqrt{t}} = \left(\frac{r T_{lv} \cos \theta}{2\eta} \right)^{1/2}$$

Assuming fabrics to be made up of capillaries, one could calculate the hypothetical rates for the present samples if the values of r , the capillary radius, were available. Estimations of pore radius were made using a model due to Gupta [15]. This model estimates the average area bounded by three fibers, oriented parallel or randomly, in a fabric. The capillary radius is then computed by transforming the area into that of a circle. The equation for the capillary radius obtained is as follows:

$$r = \left[\left(\frac{\sqrt{3}}{4} Y^2 - S \right) / \pi \right]^{1/2}$$

where

$$Y = \left[\frac{4}{\sqrt{3}} \frac{AL \cdot S}{V_F} \right]^{1/2}$$

$$S = \frac{1}{6K} \left[\frac{n_1 d_1}{\rho_1} + n_1 = 3 - n_2 \right]$$

$$n_1 = 3 - n_2$$

$$n_2 = 3 m_2 d_1 / (m_1 d_2 + m_2 d_1)$$

$$d_1 = \text{denier of fiber 1}$$

$$d_2 = \text{denier of fiber 2}$$

$$K = 9 \times 10^5$$

(The definitions of A , L , V_F , ρ_1 , ρ_2 , m_1 and m_2 have been given earlier.)

The results of the theoretical calculations are compared with those obtained from experimental measurements in Table XII for two sets of fabrics, namely blend of rayon with 4 denier polypropylene needled to depths of 1 and 3 barbs and tested under a pressure of 22 gf/cm². The units of the theoretical values being cm/sec^{1/2}, those of the measured were converted into volumetric rates, i.e., cc/g-sec^{1/2}, by dividing the values of Table XI by the densities of the fluids involved. The results show a qualitative agreement between the measured and the predicted rates. The most interesting observation is that while the non-aqueous fluids, with one exception, gave numbers which were higher than the predicted, the aqueous fluids (water, salines, synthetic urine and synthetic menstrual fluid) gave numbers which were lower than the predicted. The exception noted is the high viscosity fluid dimethyl phthalate, the results of which could not be considered highly reliable. It is suspected that a reason for the different trend obtained with the aqueous fluids is the chemical interaction between the latter and the fiber material which is not accounted for in the Washburn's equation. The latter assumes ideal capillaries with little chemical interaction between the fluid and the capillary material.

SUMMARY

Many studies are available in the literature on the absorbent characteristics of nonwovens which used salines and water as the absorbates. In this

investigation, the behavior of several model fluids including two synthetic body fluids were examined in addition to saline solutions and water.

Use of model fluids provided an insight into the mechanisms governing the fiber-fluid interaction and the capillary absorption into nonwovens.

The results showed that both the properties of the fluid and the structure of the web played a role in governing absorbency. While the capacity was mostly governed by the fabric structure, the rate was greatly affected by both the fabric structure and the fluid characteristics. Given the fluid properties and the parameters of the fabric structure, fabric absorbent capacity and the rate could be estimated, although qualitatively at this stage, by the classical equations governing capillary absorption. Departures are noted in systems in which absorbate/absorbent interaction is particularly strong. A modification of classical equations accounting for such interaction would close an important gap in the absorbency literature.

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TABLE I. FLUIDS FOR THE STUDY AND THEIR PROPERTIES

<u>Fluid</u>	<u>Surface Tension mN/m at 20°C</u>	<u>Viscosity cp (at °C)</u>
ethanol	22.3	1.20 (20)
hexadecane	27.8	3.59 (22)
dimethyl phthalate	41.8	17.00 (20)
commercial saline, 0.7%	47.3*	4.03 (25)
formamide	58.3	3.30 (25)
water	72.8	0.89 (25)

*This value was measured in the present work and reported at 25°C

Table II. CONSTITUTION OF THE SYNTHETIC BODY FLUIDS

<u>Synthetic Urine</u>	<u>grams</u>
urea	19.4
sodium chloride	8.0
calcium chloride	0.6
magnesium sulfate	1.0
water	971.0
<u>Synthetic Menstrual Fluid</u>	<u>grams</u>
sodium chloride	10.0
sodium carbonate	4.0
glycerol	100.0
carboxymethyl cellulose	4.0
water	880.0
coloring	trace

TABLE III. RESULTS OF SURFACE TENSION MEASUREMENTS

Fluid	Vol. Ratio	Surface Tension Values (mN/m)	
		At 25°C	At 37°C
ethanol		22.9	20.8
ethanol/water	25/75	44.6	42.8
ethanol/water	50/50	30.6	28.6
ethanol/water	75/25	28.7	26.5
formamide		58.5	56.8
formamide/water	25/75	67.9	66.8
formamide/water	50/50	64.1	63.0
formamide/water	75/25	61.1	60.3
water		71.9	70.4
dimethyl phthalate		42.3	41.9
hexadecane		26.7	25.7
commercial saline		47.3	46.5
saline 1%		72.2	71.0
2%		72.9	71.3
3%		73.6	72.7
4%		74.5	73.8
5%		75.3	74.7
synthetic urine		72.5	71.3
synthetic menstrual fluid		70.2	69.7

The above values are averages of three measurements.

TABLE IV. RESULTS OF CONTACT ANGLE MEASUREMENTS

Fluid	Vol. Ratio	Contact Angle (°) on Substrate			
		Glass	Teflon®	Acetate	Mylar®
ethanol		4	36	6	8
ethanol/water	25/75	16	72	39	40
ethanol/water	50/50	10	69	12	27
ethanol/water	75/25	9	54	5	7
formamide		19	92	29	25
formamide/water	25/75	34	105	59	68
formamide/water	50/50	25	101	54	64
formamide/water	75/25	21	82	35	59
water		51	112	61	72
dimethyl phthalate		12	69	14	18
hexadecane		6	56	6	8
commercial saline		16	80	24	47
saline 1%		42	108	58	67
2%		44	110	58	68
3%		46	110	59	68
4%		50	111	60	69
5%		62	112	61	69
synthetic urine		66	113	65	74
synthetic menstrual fluid		71	115	78	87

The above values are averages of three readings.

TABLE V. RESULTS OF pH MEASUREMENTS

<u>Fluid</u>	<u>Vol. Ratio</u>	<u>pH</u>
ethanol		6.01
ethanol/water	25/75	7.08
ethanol/water	50/50	7.02
ethanol/water	75/25	6.47
formamide		7.20
formamide/water	25/75	5.52
formamide/water	50/50	5.98
formamide/water	75/25	6.45
water		7.09
dimethyl phthalate		4.25
hexadecane		4.96
commercial saline		6.79
saline 1%		6.90
2%		6.69
3%		6.32
4%		6.29
5%		6.09
synthetic urine		6.49
synthetic menstrual fluid		9.95

The above values are averages of five readings.

TABLE VI. RESULTS OF DENSITY MEASUREMENTS

<u>Fluid</u>	<u>vol. ratio</u>	<u>density (g/ml)</u>
ethanol		0.79
ethanol/water	25/75	0.86
ethanol/water	50/50	0.85
ethanol/water	75/25	0.85
formamide		1.13
formamide/water	25/75	1.02
formamide/water	50/50	1.06
formamide/water	75/25	1.08
water		1.00
dimethyl phthalate		1.19
hexadecane		0.77
commercial saline		1.02
saline 1%		1.01
2%		1.01
3%		1.02
4%		1.02
5%		1.03
synthetic urine		1.01
synthetic menstrual fluid		1.06

The above are averages of three measurements.

TABLE VII. VISCOSITY RESULTS

Fluid	Vol. Ratio	Viscosity Values (cp)	
		At 25°C	At 37°C
ethanol		1.20 (20°C)	0.83 (40°C)
ethanol/water	25/75	2.31	1.55
ethanol/water	50/50	2.56	2.16
ethanol/water	75/25	2.11	1.69
formamide		3.30	
formamide/water	25/75	1.62	1.48
formamide/water	50/50	2.56	1.62
formamide/water	75/25	3.01	2.22
water		0.89	0.70
hexadecane		3.59 (22.2°C)	
dimethyl phthalate		17.00 (20°C)	
commercial saline		4.03	3.55
saline 1%		1.02	0.86
2%		1.03	0.87
3%		1.04	0.89
4%		1.04	0.90
5%		1.05	0.90
synthetic urine		1.42	1.03
synthetic menstrual fluid		8.55	8.24

The above are averages of four readings. The values of the pure compounds were taken from the literature (14).

TABLE VIII. ABSORBENT CAPACITY (g fluid/g fabric)
(Needling Depth 3, EP 22 gf/cm²)

	<u>R</u>	<u>PP2D</u>	<u>PP3D</u>	<u>PP4D</u>	<u>R/PP2D</u>	<u>R/PP3D</u>	<u>R/PP4D</u>
E	8.8	12.9	13.4	13.3	11.3	11.4	11.3
E/W 1:3	8.4	12.3	12.3	12.3	10.9	11.0	11.0
E/W 2:2	8.5	12.4	12.5	12.4	11.1	11.2	11.1
E/W 3:1	8.7	12.7	13.0	12.9	11.2	11.3	11.3
F	10.5	14.5	14.8	14.7	12.5	13.2	13.1
F/W 1:3	9.6	13.2	13.6	13.5	11.9	12.1	12.0
F/W 2:2	10.1	13.3	13.9	13.9	12.2	12.4	12.4
F/W 3:1	10.3	14.0	14.2	14.2	12.4	12.7	12.5
W	8.9	13.4	13.7	13.6	11.7	11.7	11.5
DMPT	11.3	15.1	15.5	15.2	13.9	14.0	13.9
H	8.5	12.7	13.1	12.9	11.2	12.0	11.6
CS	8.3	12.6	13.0	12.9	11.2	11.2	11.2
S 1%	8.6	13.4	13.3	13.5	11.4	11.9	11.4
S 2%	8.5	13.2	13.2	13.2	11.3	11.9	11.3
S 3%	8.3	12.9	13.0	13.0	11.2	11.4	11.2
S 4%	8.1	12.8	12.9	12.6	11.0	11.1	11.2
S 5%	-	12.7	12.8	-	10.8	11.0	-
SU	8.1	11.9	12.7	12.0	11.1	11.2	11.1
SMF	8.0	10.2	10.8	9.6	9.8	10.4	8.9

R-Rayon; PP-Polypropylene; D-Denier; E-ethanol; W-Water; F-Formamide;
DMPT-Dimethyl phthalate; H-Hexadecane; CS-Commerical saline; S-Saline;
SU-Synthetic urine; SMF-Synthetic menstrual fluid.

TABLE IX. ABSORBENT CAPACITY (g fluid/g fabric)

	<u>ND1, EP22</u>			<u>ND3, EP22</u>			<u>ND3, EP09</u>		
	R	PP4D	R/PP	R	PP4D	R/PP	R	PP4D	R/P
E	8.2	13.1	10.4	8.8	13.3	11.3	12.3	14.3	15.9
E/W 1:3	7.2	12.1	9.8	8.4	12.3	11.0	10.7	12.1	12.1
E/W 2:2	7.3	12.6	10.1	8.5	12.4	11.1	10.9	12.4	13.5
E/W 3:1	7.6	12.9	10.3	8.7	12.9	11.3	11.9	13.1	13.8
F	9.1	14.6	12.2	10.5	14.7	13.1	13.2	16.6	16.3
F/W 1:3	8.2	13.2	10.4	9.6	13.5	12.0	11.6	16.1	15.8
F/W 2:2	8.4	13.8	10.7	10.1	13.9	12.4	12.2	16.1	15.9
F/W 3:1	8.7	14.1	10.9	10.3	14.2	12.5	12.8	16.2	16.3
W	8.3	13.1	10.6	8.9	13.6	11.5	12.4	15.3	16.3
DMPT	9.6	14.8	12.7	11.3	15.2	13.9	13.3	15.0	16.3
H	8.1	12.9	9.9	8.5	12.9	11.6	12.2	13.9	14.5
CS	7.7	12.3	9.6	8.3	12.9	11.2	11.0	15.9	16.3
S 1%	8.0	12.7	10.9	8.6	13.5	11.4	11.2	15.3	16.2
S 2%	7.8	12.5	10.3	8.5	13.2	11.3	11.1	15.3	15.8
S 3%	7.6	12.3	9.9	8.3	13.0	11.2	11.0	15.2	15.3
S 4%	7.5	12.1	9.9	8.1	12.6	11.2	10.9	14.1	14.2
S 5%	7.4	12.1	9.8	-	-	-	10.5	13.7	13.8
SU	8.0	10.6	9.9	8.1	12.0	11.1	12.0	15.2	15.2
SMF	7.9	9.2	8.0	8.0	9.6	8.9	11.3	12.2	12.5

ND-Needling depth; EP-Environmental pressure (gf/cm²)
For the rest of the legend, see footnote under Table VIII.

TABLE X. RATE OF ABSORBENCY (g/g-sec^{1/2})
(Needling Depth 3, EP 22 gf/cm²)

	<u>R</u>	<u>PP2D</u>	<u>PP3D</u>	<u>PP4D</u>	<u>R/PP2D</u>	<u>R/PP3D</u>	<u>R/PP4D</u>
E	2.08	2.09	2.09	2.07	2.03	2.07	2.06
E/W 1:3	1.72	1.73	1.73	1.75	1.77	1.76	1.73
E/W 2:2	1.86	1.85	1.87	1.90	1.91	1.85	1.87
E/W 3:1	1.99	1.97	1.96	1.95	2.00	1.97	1.95
F	1.83	1.80	1.78	1.76	1.82	1.81	1.79
F/W 1:3	1.29	1.26	1.26	1.26	1.30	1.28	1.24
F/W 2:2	1.45	1.45	1.39	1.40	1.48	1.44	1.43
F/W 3:1	1.71	1.70	1.68	1.65	1.73	1.70	1.66
W	1.64	1.66	1.64	1.59	1.68	1.66	1.59
DMPT	0.30	0.25	0.20	0.33	0.29	0.46	0.32
H	1.25	1.26	1.25	1.19	1.26	1.29	1.20
CS	1.16	1.12	1.16	1.09	1.18	1.18	1.11
S 1%	1.08	1.10	1.04	1.02	1.09	1.03	0.99
S 2%	1.09	0.94	0.93	1.08	0.96	0.93	0.93
S 3%	0.95	0.89	0.90	0.95	0.90	0.90	0.88
S 4%	0.92	0.87	0.82	0.85	0.86	0.83	0.83
S 5%	0.90	0.84	0.81	0.85	0.85	0.82	0.80
SU	1.04	1.10	1.05	1.00	1.09	1.03	1.01
SMF	0.58	0.38	0.49	0.51	0.62	0.49	0.53

For legend, see footnote under Table VIII.

TABLE XI. RATE OF ABSORBENCY (g/g-sec^{1/2})

	<u>ND1, EP22</u>			<u>ND3, EP22</u>			<u>ND3, EP09</u>		
	<u>R</u>	<u>PP4D</u>	<u>R/P</u>	<u>R</u>	<u>PP4D</u>	<u>R/P</u>	<u>R</u>	<u>PP4D</u>	<u>R/P</u>
E	1.94	1.95	1.93	2.08	2.07	2.06	3.48	3.54	3.62
E/W 1:3	1.61	1.56	1.59	1.72	1.75	1.73	2.56	2.59	2.61
E/W 2:2	1.73	1.67	1.67	1.86	1.90	1.87	2.71	2.70	3.02
E/W 3:1	1.89	1.85	1.87	1.99	1.95	1.95	3.02	3.06	3.26
F	1.72	1.59	1.68	1.83	1.76	1.79	2.49	2.58	2.62
F/W 1:3	1.22	1.20	1.23	1.29	1.26	1.24	2.17	2.22	2.22
F/W 2:2	1.36	1.36	1.34	1.45	1.40	1.43	2.32	2.34	2.37
F/W 3:1	1.53	1.48	1.48	1.71	1.65	1.66	2.37	2.37	2.39
W	1.54	1.53	1.51	1.64	1.59	1.59	2.31	2.33	2.36
DMPT	0.27	0.26	0.19	0.30	0.33	0.32	1.22	1.29	1.32
H	1.25	1.19	1.24	1.25	1.19	1.20	2.35	2.37	2.49
CS	0.96	0.96	0.97	1.16	1.09	1.11	1.76	1.79	1.79
S 1%	0.97	0.90	0.91	1.08	1.02	0.99	1.61	1.67	1.69
S 2%	0.95	0.88	0.91	1.09	1.08	0.93	1.53	1.57	1.59
S 3%	0.90	0.87	0.88	0.95	0.95	0.88	1.44	1.45	1.48
S 4%	0.88	0.85	0.80	0.92	0.85	0.83	1.32	1.35	1.35
S 5%	0.84	0.78	0.80	0.90	0.85	0.80	1.24	1.26	1.27
SU	1.01	0.96	0.99	1.04	1.00	1.01	1.96	2.18	2.20
SMF	0.36	0.32	0.43	0.58	0.51	0.53	1.32	1.16	1.17

For legend, see footnote under Tables VIII and IX.

TABLE XII. COMPARISON OF THE MEASURED RATES WITH THE PREDICTED RATES
BY THE WASHBURN EQUATION

Fluid	R/PP4d; EP 22gf/cm ² ; ND 1		R/PP4d; EP 22gf/cm ² ; ND 3	
	Theoretical cm/sec ^{1/2}	Measured cc/g-sec ^{1/2}	Theoretical cm/sec ^{1/2}	Measured cc/g-sec ^{1/2}
Ethanol	1.51	2.44	1.61	2.61
E/W 50/50	1.19	1.96	1.26	2.20
Formamide	1.42	1.49	1.50	1.58
F/W 50/50	1.65	1.26	1.75	1.35
Water	2.89	1.51	3.06	1.59
Dimethyl phthalate	0.55	0.16	0.58	0.27
Hexadecane	0.94	1.61	1.00	1.56
C. Saline	1.16	0.95	1.23	1.09
Saline 1%	2.51	0.90	2.66	0.98
Saline 3%	2.45	0.86	2.59	0.86
S. Urine	1.59	0.98	1.69	1.00
S. Menstrual Fluid	0.55	0.41	0.59	0.50

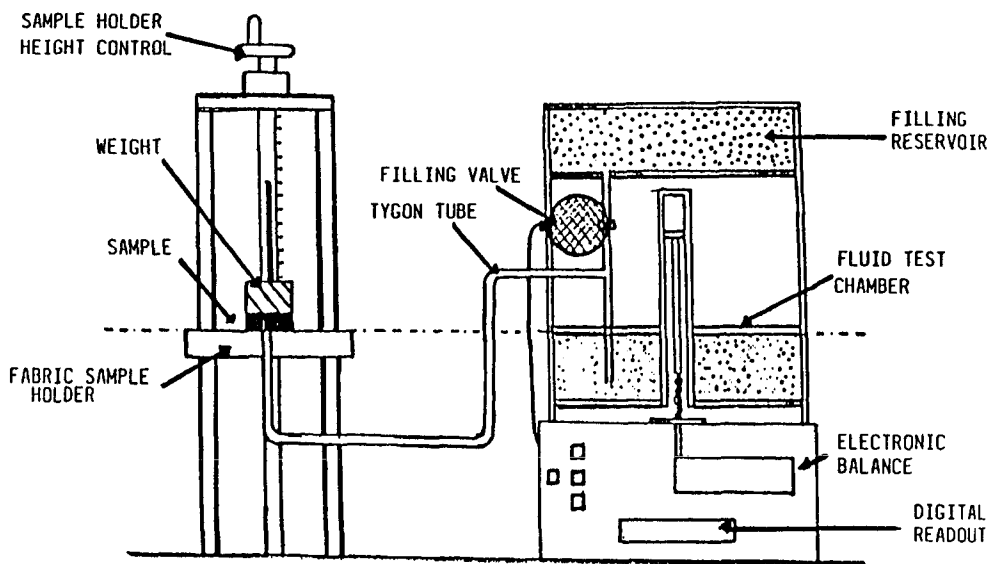


Figure 1. The Gravimetric Absorbency Testing System

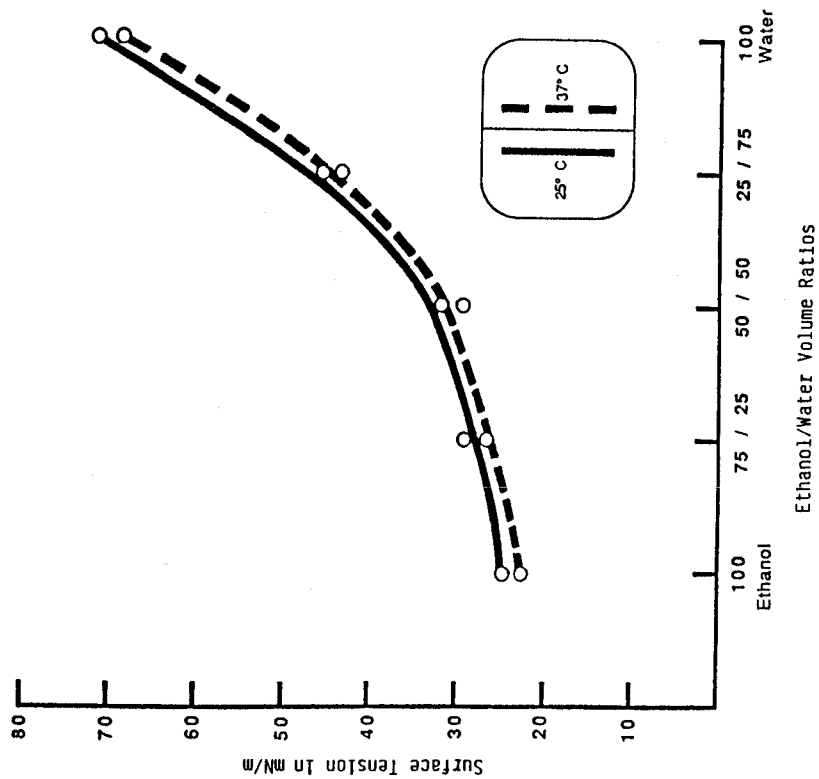


Figure 2. The effect of mixing ethanol with water on surface tension

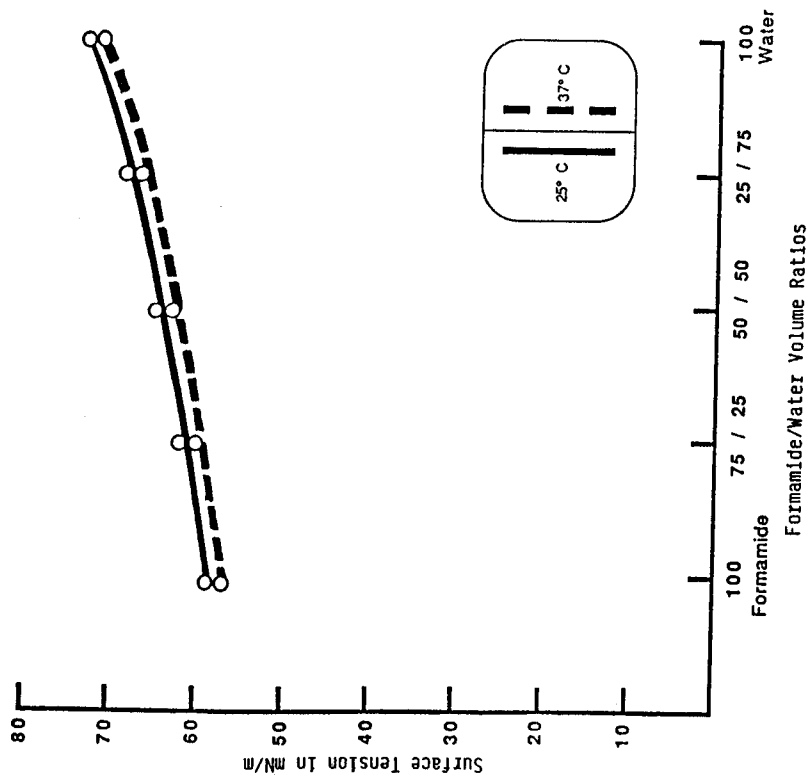


Figure 3. The effect of mixing formamide with water on surface tension

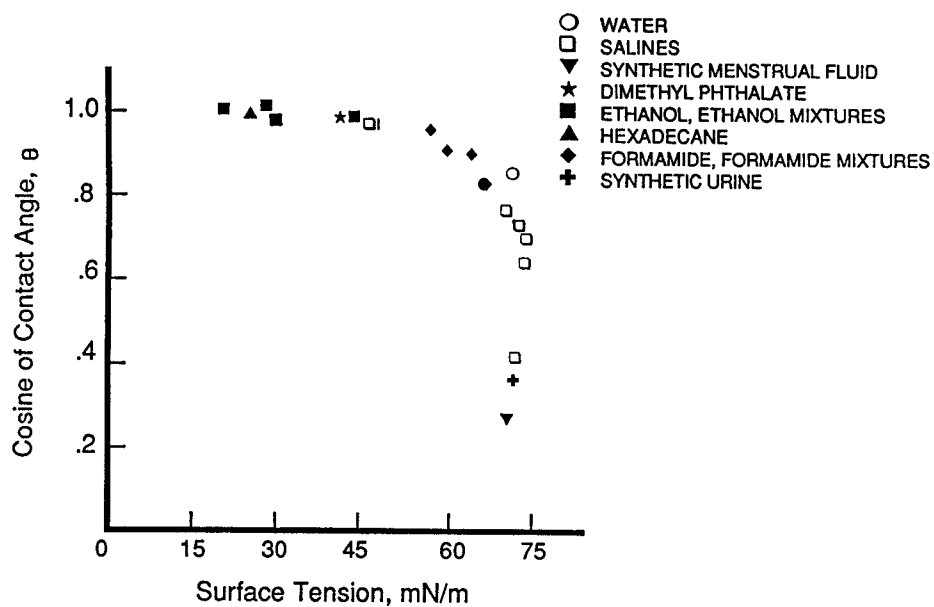


Figure 4. Relationship between the cosine of contact angle and the surface tension for various liquids on glass

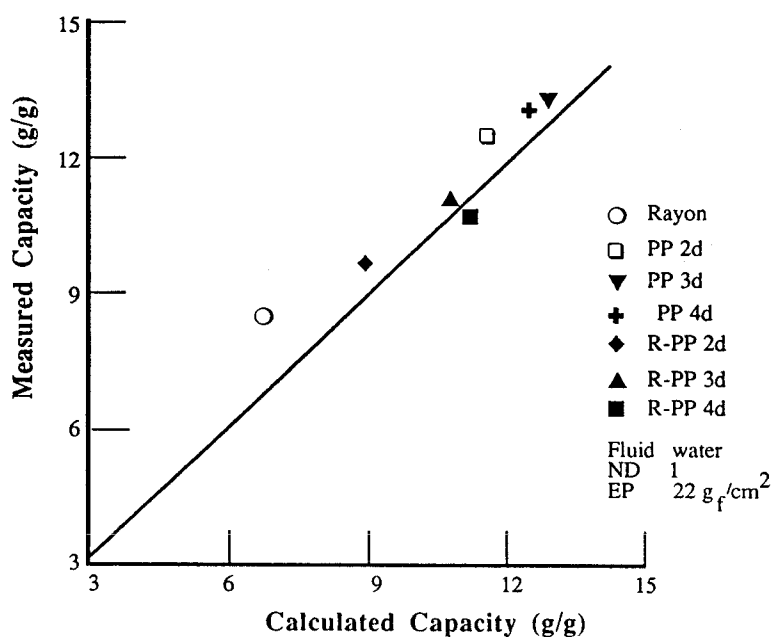


Figure 5. Comparison of predicted and measured capacities for lightly needled fabrics under an environmental pressure of 22 gf/cm²

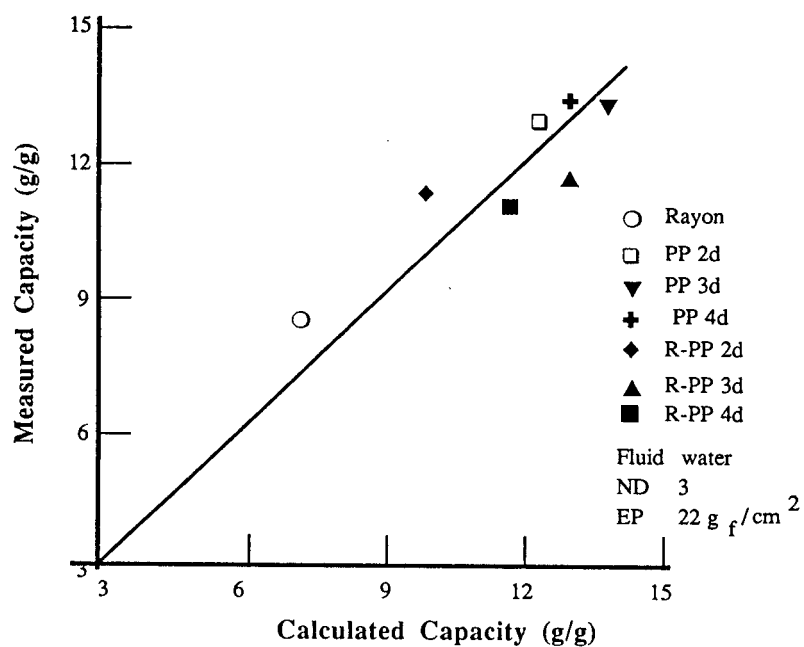


Figure 6. Comparison of predicted and measured capacities for deeply needled fabrics under an environmental pressure of 22 gf/cm²

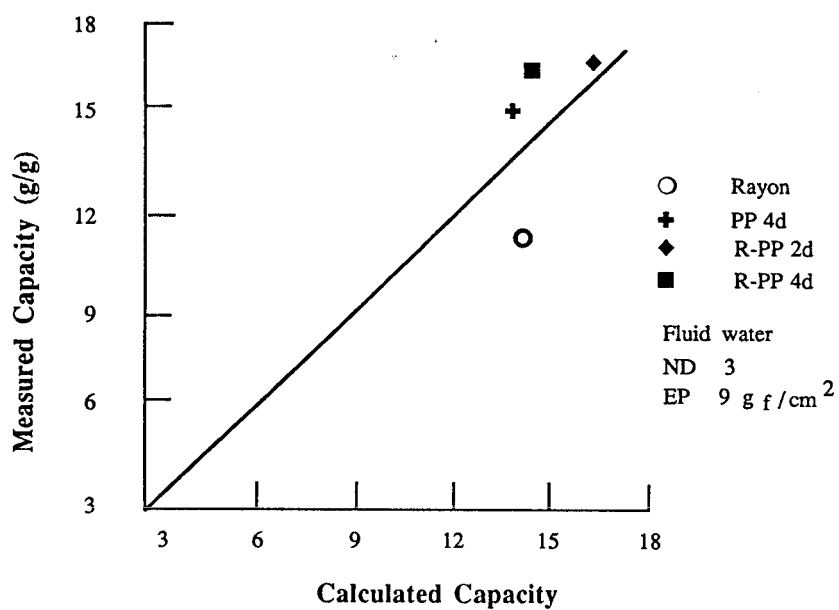


Figure 7. Comparison of predicted and measured capacities for deeply needled fabrics under an environmental pressure of 9 gf/cm²

THE USE OF SINGLE-FIBER WETTING MEASUREMENTS IN THE ASSESSMENT OF ABSORBENCY

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ABSTRACT

The technique of single-fiber microtensiometry yields values for advancing and receding contact angles of liquids against fibers as well as measures of the magnitude and axial frequency of fiber surface heterogeneities and fiber swelling. Such data, obtained as a function of interline velocity and fiber-liquid contact time provide useful insight into the wicking performance of fiber networks and permit a separation of structural factors from surface chemical factors in controlling absorbency. Data are presented for wicking into fluff pads and paper strips by pure liquids and surfactant solutions. In all cases, good correlation is found between wicking performance and the data obtained for the single fibers abstracted from the fiber networks.

Introduction

The absorbency of a porous material refers to its ability to imbibe a liquid, and high absorbency means that large amounts of liquid are imbibed in a short period of time. Products designed for this purpose have, of course, other attributes which are important in their application, such as ability to *retain* absorbed liquids under various conditions and to maintain desirable size, shape and strength properties upon liquid imbibition. Such properties, however, will not be considered part of the definition of absorbency in the present context.

While the process of liquid imbibition into a fibrous medium is highly complex, the starting point for its description is the simple theory of Lucas¹ and Washburn,² dealing with the rate at which liquid is drawn into a single, round tube by capillary action, viz.

$$\frac{dh}{dt} = \frac{r \sigma \cos \theta}{4\mu h} + \frac{r^2 \rho g \cos \beta}{8\mu}, \quad (1)$$

where h is the distance travelled by the liquid from the reservoir, r is the tube radius, θ is the contact angle of the liquid against the tube wall, and μ , σ and ρ are the viscosity, surface tension and density of the liquid, respectively. The parameter β is the angle between the direction of flow and the gravitational acceleration g . The equation computes the rate of quasi-steady laminar (Hagen-Poiseuille) flow resulting from the imposition of the Young-Laplace pressure jump across the curved advancing meniscus, $\Delta p = 2\sigma \cos \theta / r$, over the length h of the liquid in the tube. In writing Eq. 1 it is assumed that the tube is sufficiently small ($r \leq 0.1 \sqrt{\sigma / \rho g}$) that the advancing meniscus shape is a spherical segment suffering no distortion due to gravity and that the time required for the initial formation of the meniscus and its acceleration to a quasi-steady state is negligible. For liquids with properties similar to water, the former requirement is satisfied when the tube radius is less than approximately 0.3 mm, and full numerical analysis of the unsteady-state problem³ has shown that quasi-steady flow is established within a fraction of a second. Furthermore, when liquid penetration is horizontal ($\beta = 90^\circ$) or r is very small ($\leq 10 \mu\text{m}$) and $h \ll h_{\text{eq}}$ (equilibrium rise height), the second term in Eq. 1 may be neglected, leaving

$$\frac{dh}{dt} = \frac{r \sigma \cos \theta}{4\mu h}, \quad (2)$$

or in integral form, with $h = 0$ at $t = 0$:

$$h = \left[\frac{r \sigma \cos \theta}{2\mu} \right]^{1/2} t^{1/2} \equiv K t^{1/2}. \quad (3)$$

Equation 3 is used to describe the linear penetration of a liquid into a porous medium by modelling the medium as a bundle of cylindrical capillaries in parallel. Recognizing that some swelling of the fibers by the liquid will reduce the effective pore radius, Chatterjee⁴ suggested that r in Eq. 3 be replaced by r_w^2/r , where r_w is the reduced radius of the pore behind the advancing meniscus. The dimensions of the meniscus itself, however, and hence the capillary suction, would be determined by the dimensions of the dry capillary, r . The nonlinearity of the actual flow path of the liquid through a fibrous medium can be described using a tortuosity factor, τ , defined as the ratio of the actual flow path length to the nominal straight distance travelled in the direction of the general flow. With the above two changes, the constant in Eq. 2 becomes

$$K = \left[\frac{r_w^2 \sigma \cos \theta}{2r \tau^2 \mu} \right]^{1/2} = \left[\frac{r_e \sigma \cos \theta}{2\mu} \right]^{1/2}. \quad (4)$$

where r_e is the "effective pore radius."

Despite the complicated, non-circular, non-uniform, non-parallel structure of the pore spaces in most actual fiber networks, the functional form of Eq. 3 has been shown to hold in a variety of fibrous media, including paper and textile materials⁴⁻⁷ and three-dimensional pads.⁸⁻⁹ The predicted variation of K with $\sqrt{\sigma/\mu}$ has been verified with pure liquids, with the exception of water, in strips of cellulose¹⁰ and glass-fiber filter paper.⁶ It was found, however, that not even the *form* of Eq. 3 was applicable for sheets containing highly swellable fibers, such as those formed from pulps having very high amounts (> 15%) of hemicellulose.⁴

In view of Eqs. 3 and 4, there are three types of factors which govern the rate of liquid penetration into a fibrous medium: 1) the fluid properties, σ and μ , of the penetrating liquid, 2) structural factors, as represented schematically by r_e , and 3) the effective physical interaction between the fibrous solid and the liquid, as represented by the contact angle θ . In the design and development of absorbent products, one must generally deal with imbibing liquids of fixed properties so that most efforts to achieve improvements focus on either the geometrical structure of the material or upon wettability of the individual fibers of which it is composed. To understand the differences in absorbency between different fiber furnishes and in designing strategies for

enhancing absorbency, it is important to be able to separate these two factors. For example, if the fibers are treated with a rewetting agent, it would be useful to know if any observed enhancement were attributable to improved wetting of the individual fibers or to a debonding of the network (permitting more rapid swelling). It would also be useful to know the extent of individual fiber swelling upon contact with the imbibing liquid. Cellulose fibers are known to be swollen by water and a variety of other liquids, while synthetic superabsorbent fibers or granules may swell to such a large extent that the open pore spaces in the structure become effectively blocked, so that subsequent liquid transport must occur by the much slower process of diffusion. The latter situation is referred to as "gel blocking."

Significant effort in recent times has gone into designing composite absorbent structures consisting of varying amounts of wood pulp fibers and superabsorbent material,¹¹⁻¹³ either as synthetic fibers, granules or powders, as components in alloy fibers derived from cellulose, or in other geometric forms.¹⁴ It would be especially useful in optimizing these designs to have quantitative information on the properties of the individual components of the composite structures, such as the wettability and swellability of the different fibers or granules, etc. When such data are available, they may be compared to bulk absorbency data for the macroscopic networks (i.e., paper strips, fluff pads, etc.) consisting of these components.

The objective of the present work is to examine the technique of single-fiber microtensiometry for measuring the wettability and swellability of individual fibers and to illustrate its use with several examples.

The contact angle of liquids against fibers

The quantitative measure of the wettability of a fiber by a liquid is generally taken as the contact angle. Some comments must be made concerning this property. The contact angle of a liquid against a solid was first related to the energies of the three interfaces meeting at the common interline, as shown in Fig. 1, by Young¹⁵ over 180 years ago, viz.

$$\cos\theta = \frac{\sigma_{SG} - \sigma_{SL}}{\sigma}, \quad (5)$$

where σ_{SG} is the free energy of the solid-gas interface, and σ_{SL} is the free energy of the solid-liquid interface. Equation 5, known as Young's equation, gives the contact angle θ the status of a thermodynamic property, but it has been only relatively recently that this status has been fully accepted. As is well known, the observed contact angle appears to depend on many non-thermodynamic factors, most notably on whether the liquid is advancing or receding over the solid

surface. The generally observed difference between the advancing contact angle, θ_A , and the receding angle, θ_R , i.e., contact angle hysteresis, is now well understood in terms of the roughness and chemical heterogeneity of real solid surfaces.¹⁶ An intrinsic contact angle exists, but when the liquid advances over a rough surface, its interline tends to position itself on the downstream faces of the asperities, setting up a maximum resistance to the imposed motion, and exhibiting a larger nominal contact angle. In the receding mode, just the opposite occurs. In chemically heterogeneous surfaces, there exist patches with different surface energies producing different intrinsic contact angles. An advancing interline positions itself facing the high- θ patches, while a receding interline is positioned facing the low- θ patches. Roughness and chemical heterogeneity explain why the motion of a liquid interline over a solid surface occurs in a sequence of small jumps, i.e., in "stick-slip" motion. The spontaneous penetration of a liquid into a porous medium depends on the *advancing* contact angle. Such penetration may be halted, however, even if the *average* θ_A favors wetting, if the extent of local irregularity is great enough, as suggested in Fig. 2a. Also, if one is concerned with the removal of a trapped air bubble in a pore, the pressure driving force Δp required depends on the contact angle hysteresis in accord with

$$\Delta p = \frac{2\sigma}{r} (\cos\theta_R - \cos\theta_A), \quad (6)$$

for a cylindrical pore, as pictured in Fig. 2b.

As the wetting rate (or dewetting rate) increases, the apparent contact angle shows a dependence on interline velocity as viscous effects begin to distort the meniscus shape. Hydrodynamic studies¹⁷ suggest that such distortions begin when the Capillary Number, $Ca = \mu V/\sigma$, exceeds approximately 10^{-6} , where V is the interline velocity. Theoretical analysis¹⁸ indicates that the critical Capillary Number depends to some extent on the geometry of the wetting system, and experimental evidence¹⁹ suggests that natural wicking rates may frequently be sufficiently great to produce viscous meniscus distortion.

Another factor contributing to apparent contact angle irreproducibility is that it is generally the chemistry of only the outermost monolayer of the solid which is important in determining the wetting characteristics. Thus differences in traces of contamination on two nominally identical solid specimens may lead to very different contact angles. One must also anticipate that nominally cellulosic wood pulp fibers may exhibit widely different wettability characteristics depending on species, pulping process, bleaching sequence and the details of the wet end chemistry.

The above considerations have all conspired to give the contact angle, as a thermodynamic property, a bad name. Nonetheless, the *appropriate* contact angle plays a key role in absorbency.

What may not be recognized is that we often need more than just some single contact angle value to make an assessment of the wetting characteristics that are important in absorbency. One might formulate a "wetting profile," which would include: 1) the average advancing and receding contact angles, 2) the amplitude of the *variations* in both θ_A and θ_R as the interline moves over the surface (a measure of the extent of surface heterogeneity), 3) the stick-slip frequency (a measure of the size scale of the heterogeneities), 4) the dependence of the average (advancing or receding) contact angle on interline velocity, and 5) the time dependence of the contact angle due to adsorption of components from the bulk liquid or other factors.

Finally, it must be noted that if one is to make use of wettability information in making decisions about materials for absorbent structures, one must measure these properties for the exact systems in question. The needed data are not to be found in handbooks.

The most direct means of measuring the contact angle is by examining the profile of a drop formed against a flat solid surface (as suggested by Fig. 1) using a telescope or microscope equipped with a goniometer eyepiece. It is clear that this technique cannot be conveniently used to obtain the information listed in the "wettability profile" listed above. Any optical method is awkward for determining contact angles of liquids against fibers since the profile view of the meniscus is a consequence of two opposing curvatures comparable in magnitude. Such methods are totally unsuitable if the fibers are not perfect right cylinders, and thus cannot be applied to wood pulp fibers.

Single-fiber microtensiometry

The method which is successful in determining the wetting properties of fibers is an adaptation of the Wilhelmy technique devised originally for measuring surface tension. It is pictured schematically in Fig. 3. The fiber is dipped vertically into the liquid while the downward force, $F_{\downarrow}$, relative to the weight of the fiber in air, is measured using a sensitive balance. It is related to the contact angle by

$$F_{\downarrow} = p \sigma \cos\theta + \Delta m g - \rho g \Delta v, \quad (7)$$

where p is the wetted perimeter at the axial position of the fiber-liquid interline and θ is the circumferentially averaged contact angle at the same location; Δm is the mass of any liquid imbibed into the fiber above the surface and Δv is the volume of the liquid displaced by the fiber. The first term on the right represents the surface tension force pulling down on the fiber. The second and third terms represent the weight of the imbibed liquid and the buoyancy force, resp., both of which are generally negligibly small for ordinary wood pulp fibers. (For larger superabsorbent fibers,

the imbibed mass may be large enough to measure with acceptable precision.) For cases in which the fiber is fully wet out by the liquid ($\theta = 0^\circ$), the measured force may be used to obtain the perimeter of the fiber as a function of axial position, the surface tension having been measured independently in the same apparatus using a calibrated platinum rod. A non-swelling, fully wetting reference liquid (such as *n*-heptane, for cellulose fibers) may be used to obtain the dry perimeter. The latter may be used to determine the contact angle profile made by *non*-fully-wetting liquids. The advancing contact angle profile is determined as the liquid is made to rise, while the receding angle is measured as the liquid level is lowered.

A schematic diagram of the apparatus used in the present study is shown in Fig. 4, and is described in more detail elsewhere.²⁰ The wetting liquid is contained in a small Teflon cup placed inside a Ramé-Hart environmental chamber. The fiber is mounted in a small fold of aluminum foil and suspended from a hangdown wire affixed to the arm of a Cahn Model RG 2000 electrobalance (sensitivity $\pm 0.2 \mu\text{g}$ force). The environmental chamber serves as a screen against air currents and provides for temperature and humidity control. It contains opposed windows which permit observation of fiber verticality and centering. The environmental chamber (hence the liquid in the cup) is raised and lowered by means of a Burleigh Instruments electronic "Inchworm Translator." The fiber is held in null position. Interline velocity may be carefully controlled at values between $0.60 \mu\text{m}/\text{min}$ and $6.0 \text{ cm}/\text{min}$. The force signal from the electrobalance is fed to a Hewlett-Packard 87 microcomputer through an HP 59313 A/D converter and to a recording potentiometer to provide a visual force trace. The apparatus is similar to those constructed by Miller²¹ and Okagawa and Mason.²² Force traces of the type shown in Fig. 5 are obtained as the liquid is advanced and receded over the fiber surface. The initial jump in downward force occurs when the liquid first contacts the end of the fiber. As it rises, the advancing force is measured, and the jagged line reveals the stick-slip nature of the interline movement. After a given axial excursion, the Inchworm travel is reversed, and the receding force trace is revealed. Fig. 5 shows the trace for a relatively large, uniform-perimeter Nylon 6 fiber in an aqueous surfactant solution. The slopes of the curves reflect the buoyancy force. It is clear that the receding force is larger (hence the contact angle is smaller) than the advancing force. We have prepared computer software which computes from the measured force traces (sampling five points/sec) the average advancing and receding contact angles (having computed and subtracted out the buoyancy correction), the stick-slip amplitude and the stick-slip frequency (measuring the number of times, in a fixed interval, that the force trace crosses the average force line). A given fiber-liquid system can be cycled repeatedly, allowing one to follow any time changes in wettability occurring, and of course, the rate of interline travel can be varied to investigate hydrodynamic effects.

Wood pulp fibers are considerably more irregular than the Nylon fiber shown in Fig. 5. Figure 6 shows one example, a Douglas-fir fiber in water. Other types of fibers reveal distinctive

fingerprints, and it is clear how all the information named in the fiber wettability profile can be obtained.

In what follows, we present four specific examples of fiber properties determined using the microtensiometer technique and their relationship to wicking and absorbency.

Example 1: The quantitative validation of the θ -dependence of the the Lucas-Washburn slope for wicking into a fibrous medium.

The Lucas-Washburn theory indicates that the slope of the wicking distance vs time^{1/2} should vary as $(\cos\theta_A)^{1/2}$. The fiber balance provides a means for checking this. Wicking measurements were made with paper strips cut from handsheets made from α -cellulose from Whatman #40 filter paper and from a thermomechanical pulp (TMP) of hemlock furnished by the Weyerhaeuser Company, Tacoma, WA. Several different pure liquids were used to produce a range of wetting behavior, and Lucas-Washburn plots were prepared for each case using the technique of the Klemm test, as shown in Fig. 7. The position of the advancing liquid front was followed visually as a function of time. Advancing contact angles were then measured for individual fibers abstracted from these strips. The fibers produced irregular wetting profiles of the type of Fig. 6, but the average advancing angle was computed for each case. Results are shown in Table 1.²³ At least ten fibers were examined for each strip, and the average computed. The variation amongst individual fibers was as great as ± 10 to 20% in terms of $\cos\theta$. The comparison with wicking data can be made in various ways. Toluene was chosen as a reference liquid in the wicking measurements because it completely wets out both types of fibers ($\cos\theta = 1$). Fiber balance measurements can verify this fact, as shown below. A reference liquid is needed to eliminate the unknown effective pore dimensions in the paper strips. One comparison was then made by defining a "wicking equivalent surface tension," σ^* , for each liquid as

$$\sigma^* = \sigma_{\text{ref}} \left(\frac{\cos\theta_{\text{ref}}}{\cos\theta} \right) \left(\frac{\mu}{\mu_{\text{ref}}} \right) \left(\frac{K}{K_{\text{ref}}} \right)^2, \quad (8)$$

where $\cos\theta_{\text{ref}} = 1$, and the other reference properties refer to toluene. The wicking-equivalent surface tension is then compared with the actual liquid surface tension (measured independently), as shown in Table 1. The close agreement between σ^* and σ validates the expected dependence of the Lucas-Washburn slope on the contact angle. This test required that unambiguous identification of the condition in which the contact angle is 0° be made and that the test liquids do not swell the fibers. Both conditions were verified using the fiber balance. Full wet out ($\theta = 0^\circ$) of a fiber is

displayed when the advancing and receding force traces are essentially mirror images of each other, an example of which behavior is shown in Fig. 8.²⁴ Such a situation exists only when $\theta = 0^\circ$ in both the advancing and receding modes, and the force variations can be attributed to variations in the wetted perimeter. The issue of fiber swelling will be considered in a later example.

Example 2: The effect of surfactants on wicking into fiber networks.

One of the simplest ways of altering wetting is through the use of surfactants as wetting or rewetting agents. Also many of the chemicals used in the treatment of papers and textiles, such as dyes, anti-soil additives, etc., are surfactant in nature. Thus it is of interest to inquire how such materials might affect absorbency. A key feature of the imbibition of such solutions is that the surfactant will be *adsorbed* onto the fiber surfaces, i.e., scavanged from the solution as it penetrates the network. It might be speculated that this progressive depletion of surfactant from the advancing meniscus would affect both the surface tension (causing it to increase with extent of penetration) and the contact angle, possibly causing it to increase with penetration. Both effects might lead to failure of Lucas-Washburn kinetics. Again, the fiber balance, in conjunction with wicking tests, has been useful in unravelling the situation. Wicking results, as exemplified in Fig. 9, were found for all cases, i.e., perfect adherence to the Lucas-Washburn format.

The slopes of the curves for the various solutions could then be examined, recognizing that they were constant for each case. Two situations must be distinguished. In the first case, the fibers are completely wet out in the advancing mode, so that $\cos\theta = 1$, and, in view of Eq. 3, one would anticipate the slope to be directly dependent upon the surface tension, σ . In the second case, $\theta > 0^\circ$, so that one may substitute for $\cos \theta$ from Young's equation into the Lucas-Washburn equation, obtaining:

$$h = \left[\frac{r (\sigma_{SG} - \sigma_{SL})}{2\mu} \right]^{1/2} t^{1/2}, \quad (9)$$

which suggests that the wicking slope should be *independent* of the surface tension, except insofar as surface tension varies when σ_{SL} varies. The fiber balance permits determination, without ambiguity, of which situation one has in each case. Consider first the wet-out condition. The measured slope was used in each case to evaluate the wicking-equivalent surface tension, and it was compared in each case to the measured equilibrium surface tension of the solution, as shown in Fig. 10.²³ In each case, the wicking-equivalent tension was significantly higher than the equilibrium tension, until the concentration of surfactant was made quite high. It is clear that

surfactant was being scavenged from the advancing meniscus, but the constancy of σ^* at any given concentration suggests that a dynamic steady state was established almost immediately, and was maintained throughout the penetration process. This is perhaps not surprising since both the diffusion path length and the wicking path length vary as $\sqrt{t}$. The meniscus concentration level, and hence the effective surface tension, corresponds to this dynamic balance. The higher the concentration, the more rapidly is the advancing meniscus replenished with surfactant, until at high enough bulk concentration, the value at the meniscus is effectively the same as in the bulk.

One can also rationalize the difference between the behavior of the three different surfactants shown. The most persistent gap corresponds to the lowest value of the diffusivity, the value for the Triton-X 100 being over an order of magnitude smaller than that for the CTAB or the SDS. The difference between the behavior of SDS and CTAB is largely due to the fact that the extent of adsorption of the CTAB is about twice that of the SDS, their diffusivities and surface activities being approximately the same. Similar results were obtained for wicking by surfactant solutions in partially wet strips of TMP fibers, although what was compared was wicking-equivalent values of $(\sigma_{SG} - \sigma_{SL})$.²³

Example 3: The relationship of pad absorbency to single fiber wettability.

Fiber furnishes used or contemplated for use in the manufacture of absorbent products differ greatly with respect to their morphology and surface chemistry. Thus while pure, clean α -cellulose is fully wet out by water, most actual furnishes are not, and there are wide differences between them. Absorbency into assemblies of these fibers also differs greatly from case to case, and in order to understand these differences and to take advantage of them in designing new products, it is important to distinguish the effects of geometric structure of the network from those of individual fiber-liquid interaction.

We have examined the differences in wettability of a number of different fiber furnishes, as shown in Table 2. Advancing contact angles for three successive cycles are shown, together with the stick-slip amplitudes for the advancing portion of the initial wetting cycle. It is to be noted that there are significant differences in the fiber heterogeneity. The absorbency of air-laid pads of these fibers was studied in a modified fluff absorbent quality tester at the Weyerhaeuser Company, Tacoma, WA, in which a measured weight of fluff was drawn into the tester and put under a constant compressive load. The liquid was raised to the bottom of the pad so that wicking was initiated. The unit permits one to measure the precise position of the pad top with time as it collapses. One can thus obtain Lucas-Washburn plots for imbibition into these pads, as shown in Fig. 11. The total absorption time was only a few seconds, and a small part of that was consumed by the unsteady-state "wetting delay." Subsequently, however, reasonable Lucas-Washburn behavior was achieved.

One may first observe the close correlation between the initial advancing contact angle and total absorption time. Examining the Lucas-Washburn equation for constant absorption distance indicates that absorption time should vary as $1/\cos\theta_A$. This is indeed what was observed, as shown in Fig. 12. The self-sized Douglas fir case is interesting because, although it showed a positive *average* $\cos\theta$, the pad was never wet through. This appears to have been due to the large heterogeneity, as evidenced by the large stick-slip amplitude, leading to a "hung meniscus." One additional point does not fall on the curve (Douglas fir #2), and this may suggest that there were some network structural differences for this case. In general, there appeared to be good correlation between single-fiber wetting characteristics and pad absorbency.

Example 4: Measurement of fiber swelling.

The final example concerns the swelling of fibers by liquids. If the swelling process is sufficiently rapid, it has a significant impact on wicking kinetics. If the network is bonded to the extent that the entire structure cannot expand significantly, fiber swelling will occur at the expense of the open pore spaces in the structure, reducing the effective pore radius. In cases of extreme swelling, as often encountered with superabsorbent materials, the pore spaces become effectively closed, leading to gel blocking. For moderate swelling, one would expect Lucas-Washburn kinetics to continue to hold, but with a reduced rate constant, K . With swelling great enough to cause gel blocking, the Lucas-Washburn model will fail.

The fiber balance provides a convenient method for measuring individual fiber swelling. It can be assumed (although it can always be independently checked) that liquids which swell a given fiber will also wet it out. Since $\cos\theta = 1$, the measured wetting force can be used to determine the wetted fiber perimeter. The change in perimeter upon swelling can be determined by comparing the wetting force trace for the fiber in the swelling liquid against that for the same fiber in a fully wetting, but *non*-swelling, reference liquid. Three liquids which were found to swell the α -cellulose fibers abstracted from Whatman #40 filter paper were water, formamide and ethylene glycol. Interestingly, these liquids did not swell the TMP fibers. A comparison of the perimeter variation of one of the cellulose fibers in water with that in *n*-heptane (the reference liquid) is shown as an example in Fig. 13. The extent of swelling for this fiber, in terms of perimeter increase, was 34.2%. The axial variations in perimeter are seen to be quite large, and swelling shows significant local variation. The excursion distance used, 300 μm , is generally great enough to give a meaningful axial average value. Swelling ratios, in terms of perimeter, are shown for the three liquids mentioned above in Table 3. Each data entry is the average of at least six individual fibers. Wicking experiments with paper strips of the same fibers with the same liquids yielded the results shown in the last column of Table 3 in terms of the wicking-equivalent surface tension. In all cases, Lucas-Washburn kinetics prevailed, but the fiber swelling produced noticeable pore

blockage, i.e., in all cases the wicking-equivalent surface tension was substantially less than the actual surface tension. There is not a one-to-one correspondence between the extent of fiber swelling and the indicated blockage, suggesting the different solvents may have different effects on the network structure.

There was also a marked difference in the fiber swelling *rates*. Water and formamide swelled the fibers almost instantly (within a few seconds) to the final swollen value achieved, whereas the ethylene glycol showed a certain "instantaneous" swelling (shown in Table 3), followed by a continuous increase in swelling to a final value of approximately 25% in 40 minutes.

Finally, Fig. 14 shows the swelling produced by water in a carboxymethylcellulose (CMC) superabsorbent fiber. Here the average perimeter increase is over 100 %. It is interesting to note again that there are evidently strong local variations in the extent of swelling as evidenced by the increased perimeter fluctuations in the swollen fiber.

Conclusions

The technique of single-fiber microtensiometry using the fiber balance shows much promise for the measurement of individual fiber properties important in determining the absorbency of fiber assemblies. It makes possible the accurate determination of wettability and swellability of fibers, which combined with independent wicking measurements, should assist in the design and development of new absorbent products.

Acknowledgement

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Table 1: Results of wicking pure liquids into paper strips.

Cellulose Strips (Whatman #40)				
	Liquid	σ (dyne/cm)	σ^* (dyne/cm)	θ
	Methylethyl Ketone	24.6	27.5	0°
	Isopropyl Alcohol	21.7	27.3	0°
	Hexamethyl Disiloxane	15.9	16.1	0°
TMP Strips				
	Water	72.0	77.6	55.3°
	Formamide	58.2	62.2	21.8°
	Diiodomethane	51.1	45.2	34.8°
	Tetrabromoethane	48.8	48.8	29.9°
	Ethylene glycol	47.4	47.7	21.3°

Table 2: Wetting properties of various wood pulp fibers by water. The three successive advancing contact angles refer to the first, second and third immersions of the fiber.

Fiber	θ_{A1}	θ_{A2}	θ_{A3}	Stick-slip Amplitude (%)
α -Cellulose	14.0°	0°	0°	12.8
Southern pine	23.2°	14.0°	0-10°	11.0
Douglas fir #1	33.0°	20.9°	14.5°	12.7
TMP #1	42.8°	38.2°	36.9°	20.3
TMP #2	51.2°	43.4°	40.9°	20.3
Douglas fir #2	51.3°	36.1°	27.4°	20.3
Self-sized Douglas fir #2	65.4°	60.2°	56.7°	38.5

Table 3: Swelling of individual cellulose fibers (from Whatman #40 filter paper) as measured by percent increase in perimeter and comparison with reduced wicking in paper strips of the same fibers. The latter is noted in terms of the difference between the liquid surface tension and the wicking-equivalent surface tension.

Liquid	Fiber swelling (%)	σ (dynes/cm)	σ^* (dynes/cm)
Water	28	72.0	33.9
Formamide	15	58.2	26.3
Ethylene glycol	6	47.7	24.0

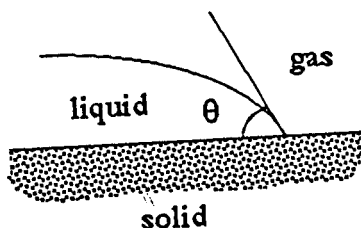


Fig. 1 Contact angle at a liquid-gas-solid interline.

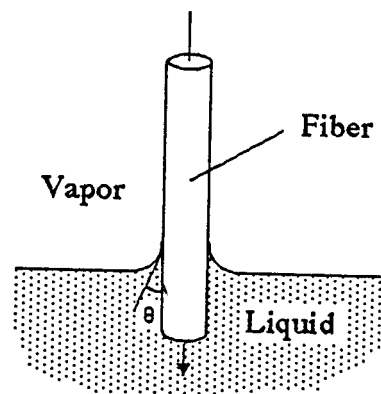
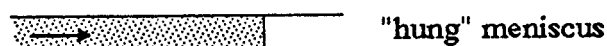


Fig. 2 Schematic representation of the importance of contact angle hysteresis in wicking flow.



(a)



(b)

Fig. 3 Cylindrical fiber partially immersed in a wetting liquid.

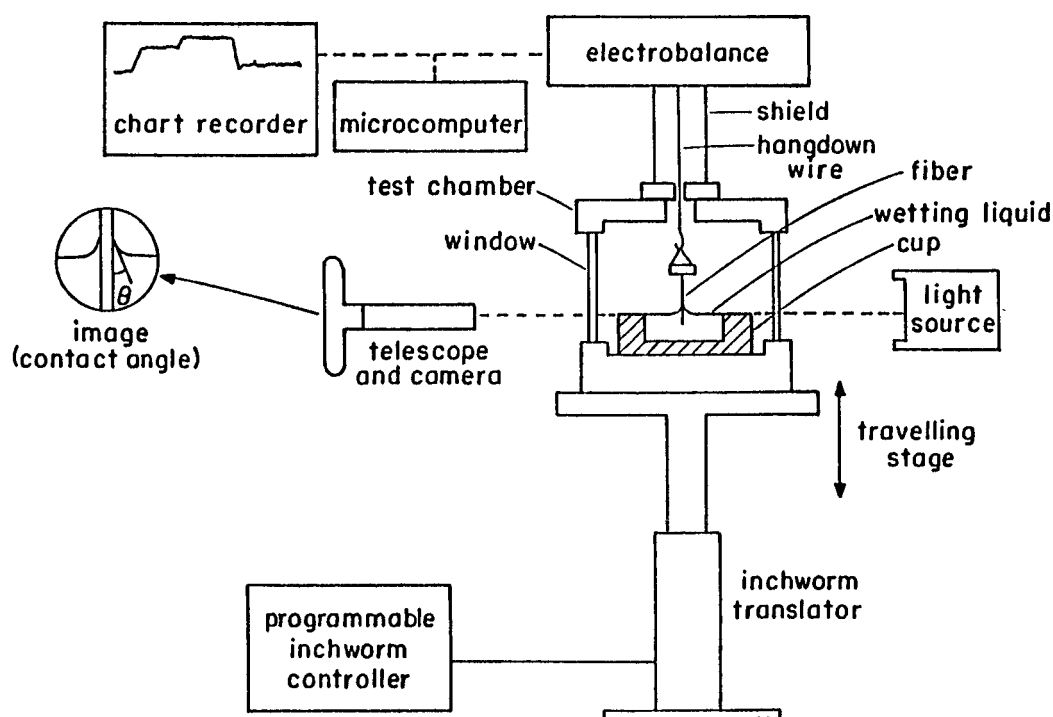


Fig. 4 Schematic diagram of the fiber balance. From Berg (1986), by permission of Elsevier Science Publishers.

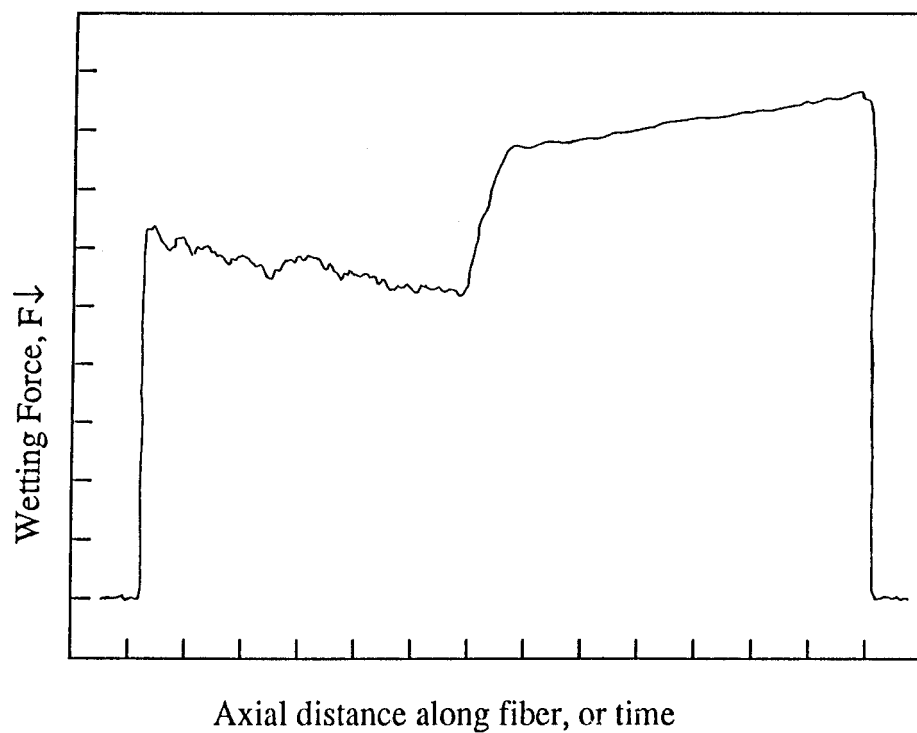


Fig. 5 Force record for the wetting of a fiber of Nylon 6 ($d = 0.71$ mm) by a 5×10^{-4} M aqueous solution of sodium dodecyl sulfate. Excursion distance: 2 mm, interline velocity: $750 \mu\text{m}/\text{min}$.

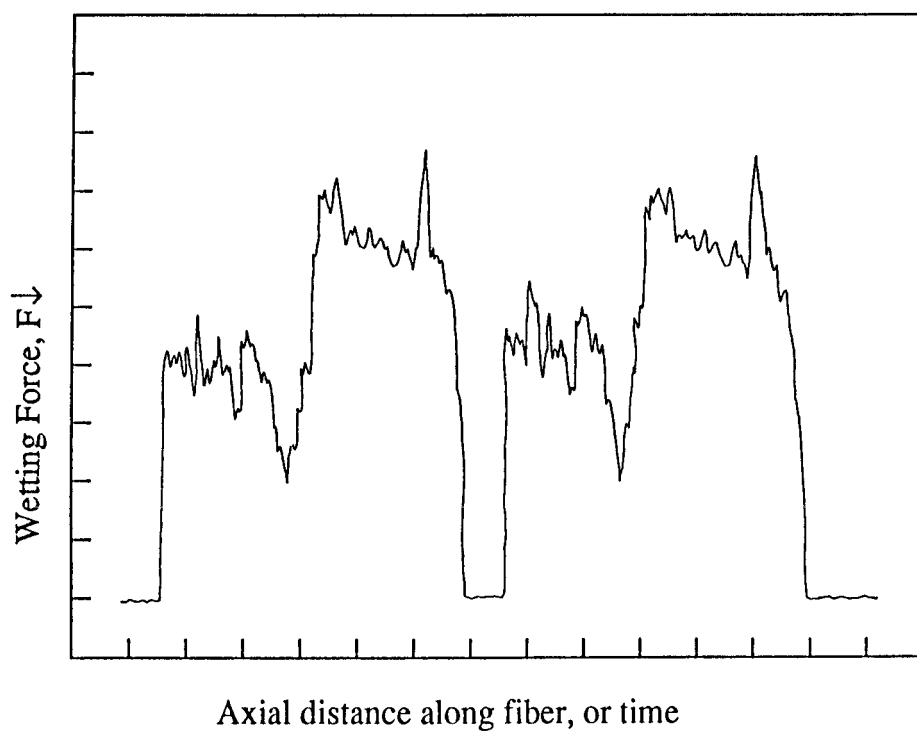


Fig. 6 Force record for wetting of a self-sized Douglas fir fiber with water. Excursion distance: 1.68 mm, interline velocity: $750 \mu\text{m}/\text{min}$.

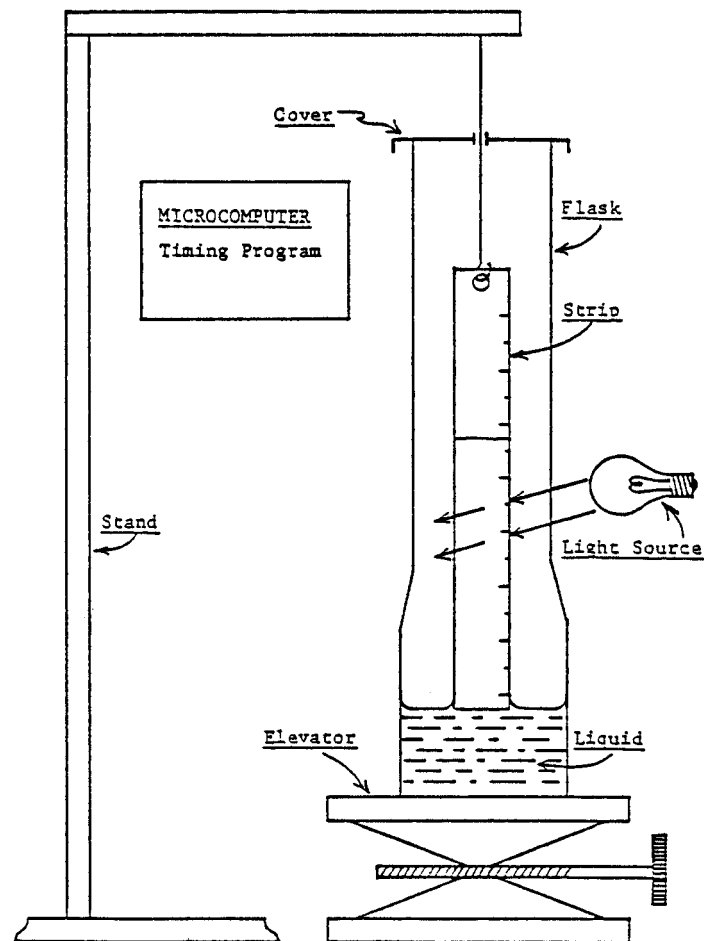


Fig. 7 Schematic diagram of wicking test.

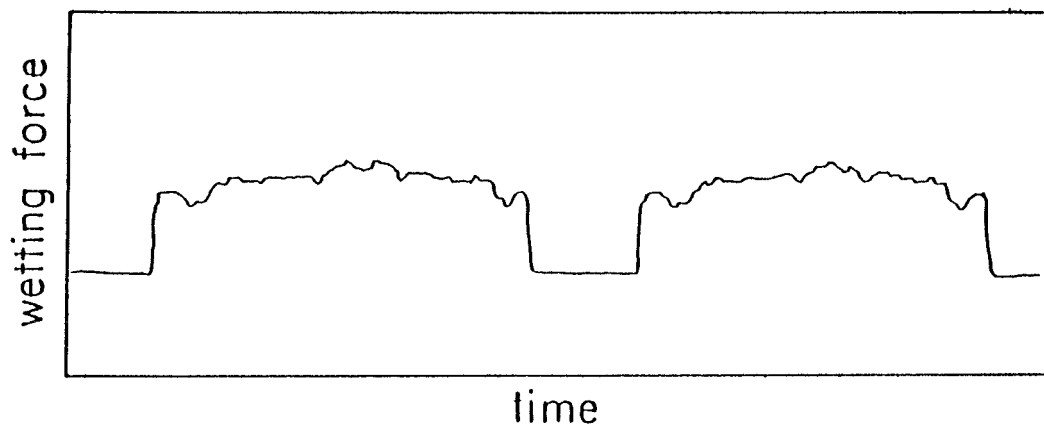


Fig. 8 Force record for wetting of self-sized Douglas fir fiber with methanol, showing absence of hysteresis. Excursion distance: 1.73 mm, interline velocity: 750 $\mu\text{m}/\text{min}$. From Hodgson and Berg (1988), by permission of the Society of Wood Science and Technology.

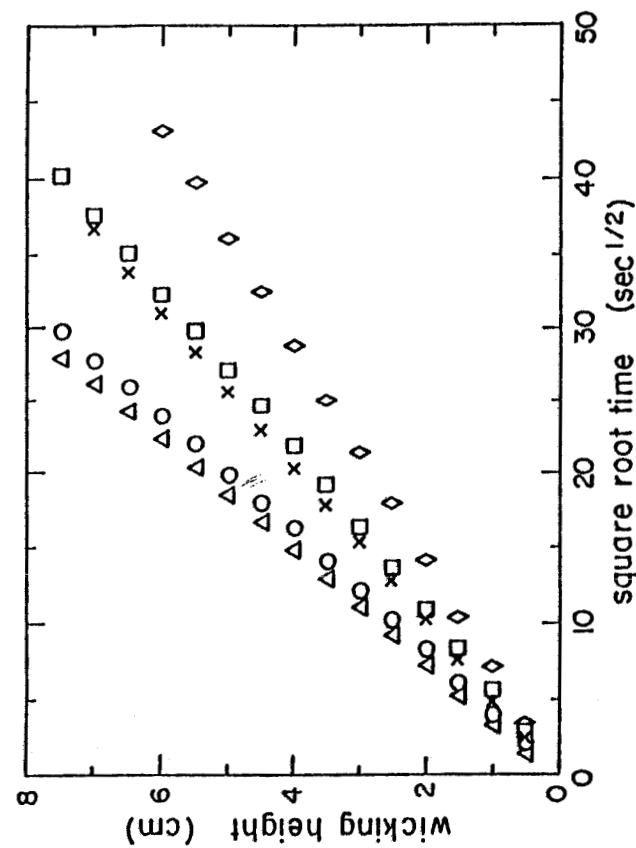


Fig. 9 Lucas-Washburn plots of wicking into strips of Whatman No. 40 filter paper: (Δ) water, ($\times$) 0.01 wt% TX-100, ($\square$) 0.005 M SDS, ($\diamond$) 0.1 M CTAB. From Hodgson and Berg (1988), by permission of Academic Press, Inc.

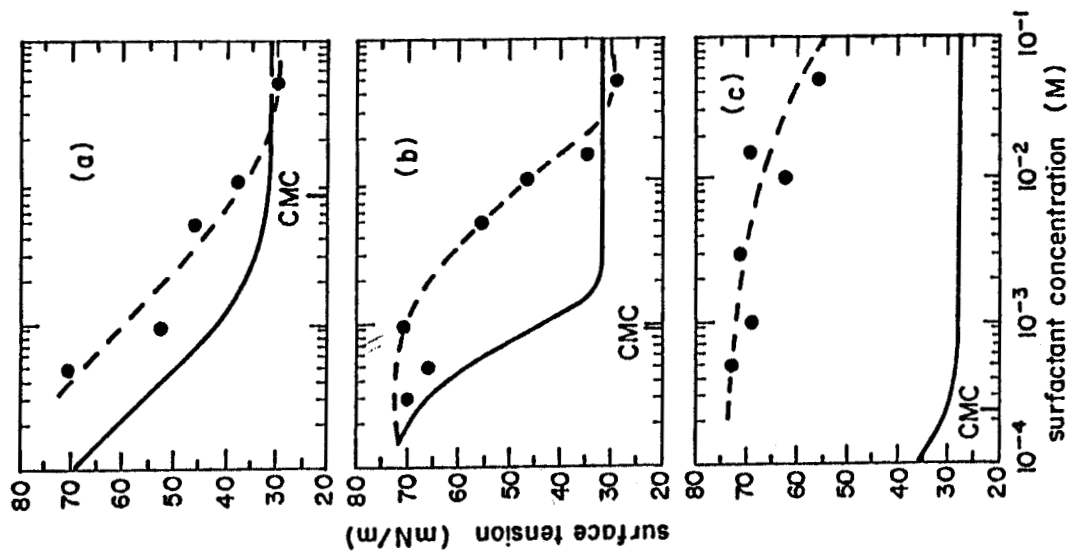


Fig. 10 Comparison of wicking-equivalent surface tension (---) with equilibrium surface tension (—) for aqueous solutions of (a) SDS, (b) CTAB, and (c) TX-100, imbibing into strips of Whatman No. 40 filter paper strips. From Hodgson and Berg (1988), by permission of Academic Press, Inc.

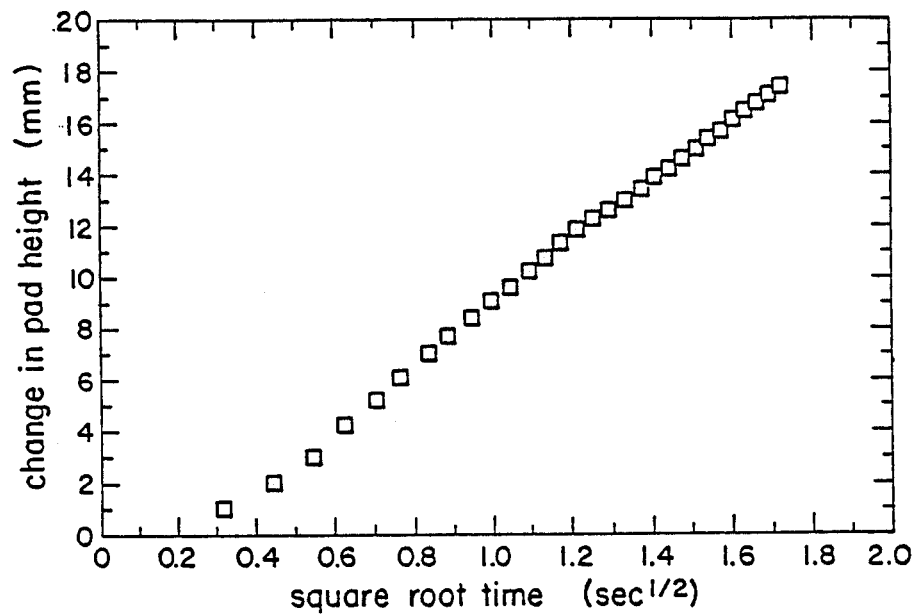


Fig. 11 Lucas-Washburn plot of water wicking into three-dimensional pad of α -cellulose fibers. From Hodgson and Berg (1988), by permission of Academic Press, Inc.

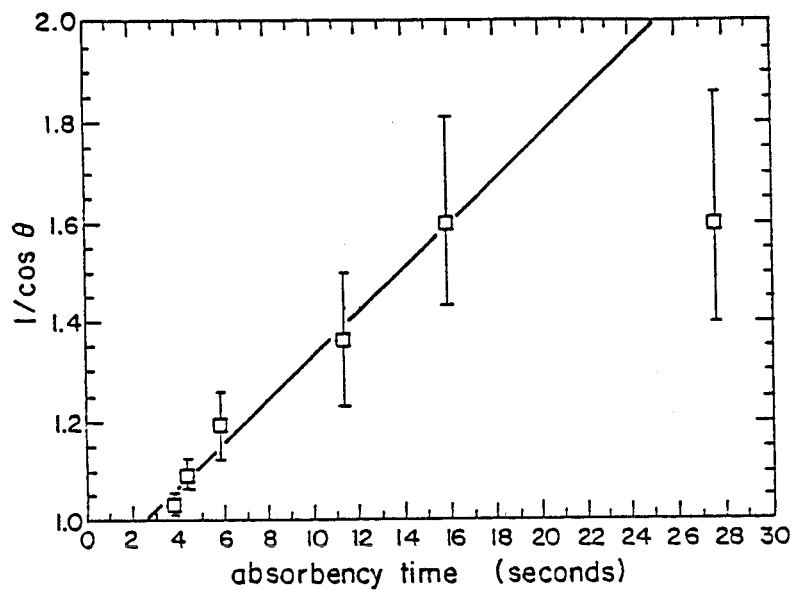


Fig. 12 Relationship between single-fiber advancing contact angle and bulk absorbency time for fluff pads. From Hodgson and Berg (1988), by permission of the Society of Wood Science and Technology.

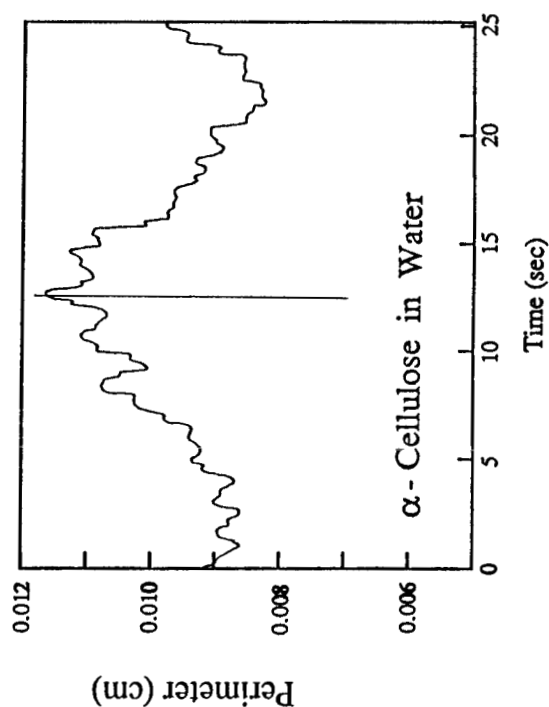
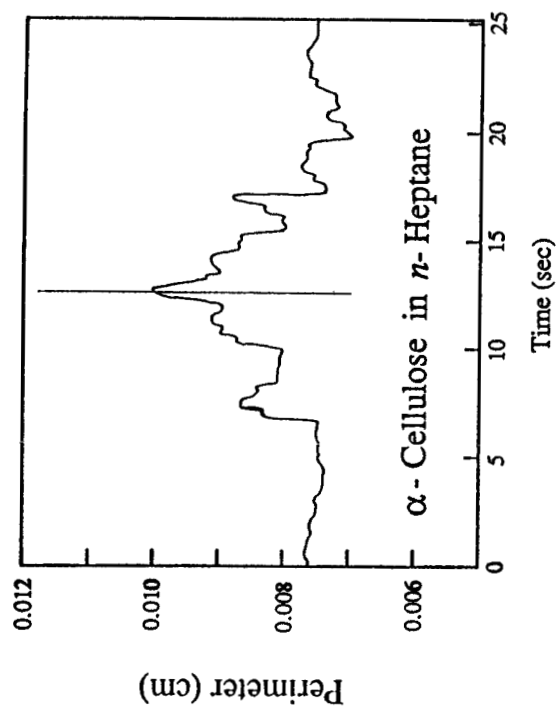


Fig. 13 Comparative wetting force records for a given α -cellulose fiber wetted by a non-swelling reference liquid (*n*-heptane) and by water. Excursion distance: 300 μ m. Centerline shows changeover from advancing to receding mode. The average perimeter of the swollen fiber is 34.2 % higher than that of the unswollen fiber.

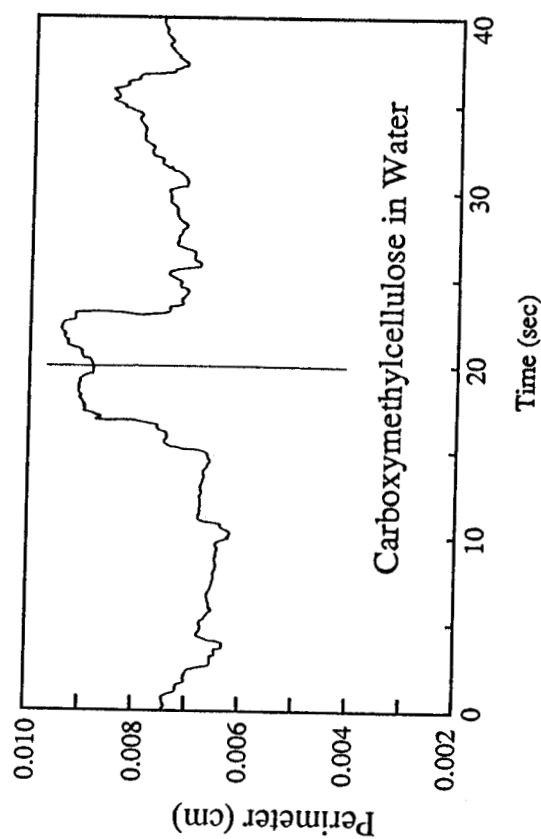
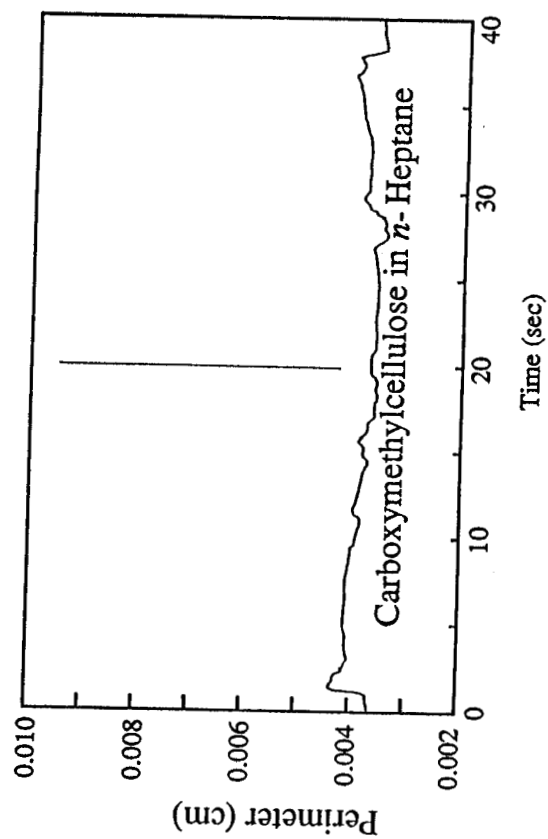


Fig. 14 Comparative wetting force records for a given superabsorbent carboxymethylcellulose fiber wetted by a non-swelling reference liquid (*n*-heptane) and by water. Excursion distance: 300 μ m. Centerline shows changeover from advancing to receding mode. The average perimeter of the swollen fiber is 106 % higher than that of the unswollen fiber.

FLUID TRANSPORT IN THERMALLY BONDED CELLULOSE PADS

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ABSTRACT

Lofty cellulose pads are used as the absorbent cores of many personal hygiene products. The effectiveness of those products depends on their ability to wick fluid and thereby utilize the entire absorbent core. The Lucas-Washburn (L-W) equation, using an average pore radius and an average contact angle, has been used to characterize wicking in porous cellulose structures. These low density cores collapse when absorbing water, making the interpretation of wicking experiments more difficult. By blending cellulose fluff pulp with a fibrillated polyethylene synthetic pulp, absorbent cores may be thermally bonded. Thermal bonding gives the structures greater cohesiveness so that they retain their density while wet, permitting evaluation of a wider range of cellulose wet pore distributions than previously reported.

A series of low density bonded cores was constructed and characterized; wet pore size distributions (PSD) were measured as well as vertical and horizontal wicking.

The wet pore size distributions are fairly narrow and, though skewed, are well represented by normal or log-normal distributions. From an average wet pore size and the L-W eq, an effective matrix contact angle, θ , can be calculated. In these low density absorbent structures, θ changes with the PSD and fluid properties, in disagreement with the L-W eq. Further disagreement is seen in that θ depends on whether the liquid transport is vertical or horizontal.

INTRODUCTION

The absorbent products industry uses numerous special tests to characterize fluid transport in absorbent cores. There are vertical wicking tests, horizontal wicking tests, inclined wicking tests, "u"-shaped wicking tests, sink tests, float tests, forced flow tests, and product performance tests. Some typical product performance tests are the TEFO tests of diapers: capacity, wicking and spreading, leakage, strike through, and rewet. Other product performance tests involve field tests on human subjects. Is there some relationship between one test and another? Can the results of one test be used to predict the performance of another? This paper looks at the relationships among several fluid transport tests for thermally bonded cores and attempts to relate these tests through the pore structure and the Lucas-Washburn (L-W) eq (Washburn, Chatterjee).

P.K. Chatterjee and H.V. Nguyen (1985) discussed the relationship between liquid flow and the structure of porous materials and analyzed the wicking of water in cellulose wood pulp sheet. They found that the wicking was in general agreement with the L-W eq. The L-W eq, derived for a single idealized cylindrical capillary tube, is given by eq 1.

$$\frac{d\ell}{dt} = \frac{\sigma r \cos \Theta}{4\mu \ell} - \frac{r^2 \rho g}{8\mu}$$

(1)

ℓ = distance wicked	ρ = density
t = time	μ = viscosity
g = gravitational acceleration	
σ = surface tension	r = pore radius
Θ = effective matrix contact angle	

The contact angle in eq 1, when applied to porous solids, is the effective matrix contact angle and not the intrinsic contact angle of the solid surface. One expects this effective contact angle to depend on the porous structure and on that intrinsic contact angle(s).

Chatterjee and Nguyen (1985) recognized that in fibrous structures, such as cellulose, these idealized capillaries do not exist, and further, that for cellulose, water would swell the fibers causing dimensions to change. However, even given these restrictions, eq 1 has been validated in a number of cases (Chatterjee, Aberson) and is a useful model for discussing capillary flow in porous media. Most of the analysis of wicking in wood pulp has been for paperboard with a density of 0.2 to 0.7 g/cc. Absorbent cores typically have densities of 0.04 to 0.13 g/cc and lie outside the normal range for which the L-W eq has been verified in porous materials.

Cellulose wood pulp may not be the ideal material for studying capillary flow in these lofty absorbent materials. Painter and Nguyen (1983) described the change in the dimensions of cellulosic structures upon absorbing water. Cellulose absorbent structures denser than 0.2 g/cc will swell and structures loftier than 0.1 will collapse to a neutral or "critical" density of about 0.1. This change in density during wicking complicates the analysis, complicates the comparison of different absorbent cores, and complicates the comparison of test methods. In order to study wicking in lofty porous structures, it is desirable to use a material which maintains its structure during fluid transport processes and yet resembles wood pulp. Thermally-bonded wood-pulp absorbent cores can be used to mimic wood pulp absorbent cores because they have similar fibrous structures and similar wettability characteristics and yet retain the core density better when absorbing water.

According to the L-W eq the only characteristics of the absorbent core which influence spontaneous capillary flow are the pore radius of the capillaries and the contact angle between the fluid and the solid surface. Thus, if one knew the effective pore radius and the effective contact angle of a porous material, and if the L-W eq were a reasonable approximation for capillary flow in that structure, then one should be able to predict the performance of bonded cores in wicking tests. Testing the limits of that hypothesis is one of the goals of this study.

In an earlier report, Wilson (1987) reported wet pore size distributions of thermally bonded cores. In that report, bimodal pore size distributions were observed. Another goal of this work was to confirm, with better techniques, those earlier observations, and to determine what effect, if any, those bimodal structures had on fluid transport.

Miller and Tyomkin (1986) reported an improved technique for characterizing the pore size distribution of lofty porous materials. This report incorporates much of that improved technique in reanalyzing pore size distributions of thermally bonded cores.

Others have tried to infer a contact angle in porous materials. Aberson used wicking to characterize the contact angle of wood pulp sheets; that analysis was based on the short-time solution to the Lucas-Washburn eq. Cassie and Baxter (1944) demonstrated that the apparent contact angle of a porous surface depends on the pore size of the surface as well as its intrinsic contact angle. That analysis may have implications for the apparent contact angle of porous volumes as well as for porous surfaces. Expanding on these approaches, the effect of pore size on the effective contact angle is estimated.

EXPERIMENTAL

The data described in this paper were obtained from the analysis of thermally bonded absorbent cores made from a synthetic pulp and cellulose blendsheet; the lofty cores were fluffed, air-laid, and bonded on laboratory equipment.

Pad construction

Blendsheet is paperboard made by blending a wood pulp slurry and an aqueous slurry of synthetic pulp and then following normal paper manufacturing technique. The blendsheet was made at Western Michigan University Paper Lab and its composition was 75 % Rayonier Rayfloc-J Kraft pulp and 25 % Pulpex® E338 polyethylene synthetic pulp. Pads were constructed by milling the blendsheet on a Fitzmill, and air-laying the resultant fluff on a lab Kroyer air-former. The fluff was air laid to form 30 cm x 30 cm pads which were about 0.035 g/cc density and about 400 gsm (grams per square meter) basis weight.

These lofty air-laid pads were placed on tissue and bonded in a laboratory "thru-air" heater for 15 seconds at 163°C. The air velocity through the pad is about 24 cm/s. A schematic of the "thru-air" bonder is shown in Figure 1. The density and pore size distribution were manipulated by varying the gap between the nip rolls and by changing the delay between the time the bonded pad enters the cooling zone (not shown) and the time the cooling air is turned on. At normal line speed, it takes about 3 seconds for the pad to enter the cooling zone. Without the blower the pad cools to room temperature in a few minutes; with the blower on the pad cools within seconds. The air velocity in the cooling zone is about 24 cm/s.

The experimental conditions for making the pads are listed in Table 1.

Pad Characterization

All pads listed in Table 1 were characterized in terms of density, vertical wick height vs time, and pore size distribution. In addition, many pads were evaluated in terms of the vertical wicking of synthetic menses fluid and the horizontal wicking of saline and menses fluids.

Density was measured using a Testing Machines Inc, TM49/21 foam caliper gauge. The average thickness and the measured basis weight yield a calculated density.

Vertical wicking was characterized by the EDANA method (10.0-72 part C), (equivalent to DIN 53924 or Tappi RC-8 amended). As Hercules applies this method, a 3 cm wide, 20 cm long strip of the absorbent core is suspended by a vertical rod into a 1% saline solution which contains 100 ppm Rhodamine B dye. The vertical rod is inserted through a small hole at either end of the strip as shown in Figure 2. Height of the flow front versus time is recorded. Vertical wicking data and pad densities are listed in Table 1.

Wicking solutions used were either 1% saline in deionized water with a surface tension of 66-72 dyne/cm, or a "synthetic menses" fluid with a surface tension of 50 dyne/cm and a viscosity of 0.06 poise. Vertical wicking with "synthetic menses" is listed in Table 2.

Pore size distributions were measured by the technique of Miller and Tyomkin (1986) of the Textile Research Institute (TRI). This technique is an enhancement of an earlier technique (Miller, 1983) which in turn is an extension of the work of Burgeni and Kapur (1967). A major aspect of this new technique is that a positive gas pressure is applied to the top of the absorbent core, rather than a "negative" pressure by raising the core above the liquid reservoir. This technique is an improvement over the previous technique. The pressurized cell eliminates evaporation which was significant at long times, i.e., small pores. Since no part of the apparatus moves, errors due to flexing of the tubing are eliminated. Since the pressure below the support membrane stays constant, trapped gas

bubbles will not change size as the applied pressure increases. Each of these sources of errors was identified as contributing to previously reported results. The pressure cell was purchased from TRI; the rest of the components were constructed of spare parts.

For convenience, we control the pressure with a gas regulator. A tee with a valve is used to establish a controlled leak, permitting easier pressure control. The pressure is measured with a "u-tube" manometer up to 100 cm of water. The sample rests on a 1.2 micron filter which in turn is supported by a 40 micron sintered metal disk; finer filters permit probing smaller pores but increase the experimentation time.

Data from the pore size distributions are listed in Table 3. Several typical distributions are plotted in Figures 4-6. Figures 4A, 5, and 6, show the data plotted according to a Gaussian distribution while Figure 4B shows the distribution plotted according to the log-normal distribution.

Horizontal wicking was measured using a special reservoir in conjunction with the Gravimetric Absorbency Testing System (GATS). The GATS is a commercial version of E.V. Painter's (1984) electronic demand wettability apparatus. Painter also discussed a horizontal wicking test, similar in concept to this test wherein the weight as well as the position of the wicking front are monitored. Our horizontal wicking cell is illustrated in Figure 7. Horizontal wicking was evaluated with 1% saline and with "synthetic menses"; the data are shown in Figures 19-25.

DISCUSSION OF RESULTS

In this discussion the data are analyzed to evaluate several aspects of fluid transport in low density bonded cores:

- o measurement of PSD's
- o effect of pore radius on contact angle in the L-W eq
- o effect of horizontal versus vertical wicking on the contact angle
- o effect of fluid properties on the contact angle.

The Lucas-Washburn eq, eq 1, which describes fluid movement in a pore, was derived for a single perfect capillary, and for those pores, it successfully accounts for vertical orientation, fluid viscosity, surface tension, and pore radius. The data obtained in this work describe fluid transport in very low density fibrous structures very unlike the single perfect capillaries for which the L-W eq was derived and these cores are of lower density than the materials for which the L-W eq has been verified. The data collected in this work are used to test the L-W eq for these low density pads.

From wicking experiments and the measured pore radius, contact angles can be calculated. One test of the L-W eq is to determine if calculated contact angle is constant, as it should be if the L-W eq applies in this regime. If the the measured contact angle depends on experimental conditions, the L-W eq does not properly account for the factors of pore radius orientation, and fluid properties.

Measurement of Pore Size Distributions

The Lucas-Washburn Equation identifies two characteristics of the solid which determine fluid transport: the pore radius, r , and the contact angle, θ . Measurement of these parameters is the key to understanding and modeling flow in these low density porous media. While techniques are not known for the direct measurement of the effective matrix contact angle of these structures, recently, PSD's were reported along with the observation that the distribution was bimodal. This study repeats and extends those measurements using an improved technique.

None of the twenty-two PSD's measured by the new technique shows the bimodal PSD's reported previously. Figure 8 compares the PSD measured by the previous analysis and PSD on the same construction measured by the improved technique. The differences have been traced to a combination of small experimental artifacts introduced by the previous experimental procedure and eliminated by the new apparatus and procedure: evaporation of water from the sample, incomplete degassing of the fluid, and inconsistent flexing of the tubing between the reservoir and the sample while the sample was being raised.

Examination of the distributions indicate that the data are marginally better fit (less skewed) by a log-normal distribution and that the distributions are fairly narrow.

Figure 9A illustrates the effect of the bonder densification technique on the average pad density. As expected, the smaller the nip clearance, the greater the density. Figure 9B shows that for these pads there is significant "springback" with the final thickness about twice the nip clearance. As shown in Figure 9C, the cooling delay has an insignificant effect on springback.

Pads of the same composition were constructed over a wide range of densities to produce a wide range of effective pore size distributions since theory predicts that the pore radius should be a significant factor in fluid transport. In much of the discussion to follow the measured PSD's will be used to interpret fluid transport results and test the range and validity of eq (1) in lofty thermally-bonded cores.

Effect of Pore Size on Contact Angle

A. Vertical Wicking Height (Steady State)

Eq 1 predicts that the height a fluid will wick in a porous structure is inversely related to the pore radius and that the contact

angle is a function of the solid surface, independent of the pore radius. Figure 10 illustrates that the height wicked is only qualitatively described by eq 1, and Figure 11 shows that the calculated contact angle is not constant but does vary with the pore radius.

Eq (2) is the solution of the time dependent Lucas-Washburn Equation.

$$t = \frac{h_{ss} \ln Z}{B} - \frac{h_{ss}(1-1/Z)}{B} \quad (2)$$

where,

$$B = \frac{r^2 \rho g}{8\mu} \quad (3)$$

and, the steady state height is

$$h_{ss} = \frac{2\sigma \cos \Theta}{r\rho g} \quad (4)$$

and,

$$Z = \frac{h_{ss}}{h_{ss}-h} \quad (5).$$

Figure 10 shows the effect of the pore size distribution (PSD) on the vertical wicking height of a saline solution. The radius corresponding to the peak of the PSD is called the mode, and the mode of the PSD is used often throughout this analysis as the representative pore radius since the distributions are fairly narrow. The reciprocal of the mode is plotted versus the wick height in Figure 10. Also plotted on this figure are the predicted wicking heights calculated from the steady-state solution to the L-W eq (eq 4) for $\Theta = 60$.

The data illustrate trends similar to those predicted by eq 4. However, significant deviations from theory are observed. First, in the region of small pores, where the data seem to lie near a straight line, the line does not extrapolate to the origin. Second, in the large pore regime, the data deviates from the straight line.

One interpretation is that the pore radius affects the apparent contact angle, and the data from Figure 10 were used to estimate the contact angle as a function of the pore radius. The slope of the straight line from the origin to each data point was used with eq 4 to estimate $\cos \Theta$, assuming that r is the PSD mode.

Figure 11 shows the effect of r on the apparent contact angle. For reference, several other results are also plotted in Figure 11. Aberson (9) inferred the contact angle of cellulose in handsheets based on wicking rates with saline versus wicking rates of surfactant solution, assuming an infinitesimal advancing contact angle for the surfactant solution. Typical values obtained by Aberson are indicated in the figure. Cassie and Baxter (10) extended the results of Wenzel (11) to estimate the contact angle of porous surfaces. They obtained eq 6.

$$\cos \theta_D = f_1 \cos \theta_A - f_2 \quad (6)$$

θ_D = apparent advancing contact angle

θ_A = intrinsic contact angle

f_1 = solid/liquid interfacial area fraction

f_2 = liquid/air interfacial area fraction

Assuming that eq 6 holds, assuming that the diameter of a cellulose fiber is about 20 microns, and assuming the intrinsic contact angle of cellulose allows one to calculate the curves labelled "Cassie and Baxter". Each Cassie-Baxter curve was derived for a specific, well-ordered geometry leading to an analytical solution; however, the results are not expected to be very different for less well-ordered arrangement of fibers.

One interpretation of Figures 10 and 11 is that eq 4 is, at best, qualitatively correct for lofty, thermally-bonded pads and as the pore radius gets larger, significant deviations from theory are observed. Alternately the PSD mode is a poor substitute for r in eq 4. A much larger estimate of r than the PSD mode yields a better straight line with an intercept nearer the origin. For reasons to be shown later, this larger estimate is not useful in other wick tests.

Further, it appears that the Cassie-Baxter formulation which was derived for surface wettability, is not applicable in this very different regime of porosity and may not be applicable at all for capillary wicking. Eq 6 predicts that even the most wettable of the 20 micron fibers would be hydrophobic for the pore radius distributions seen in absorbent cores. Thus the curve shown in Figure 11 is an obviously inappropriate extension of eq 6.

While the Lucas-Washburn eq indicates the correct trends for vertical wicking height, it is not correct for these lofty thermally-bonded materials. That is, the effective contact angle appears to be a function of the pore radius. A contact angle estimated from a pad of one density does not permit one to estimate the wicking height for a second pad, of the same chemical composition, but of a different density with a different known PSD. Similar trends can be seen in the vertical wicking rate results.

B. Vertical Wicking Rates (height vs time)

Typical vertical wicking rate data are shown in Figures 12,13,14, and 15 along with the predicted wick results from the solution to the time dependent L-W eq. As shown here, the wicking rate data, for large pores, are not well predicted by the L-W eq. What is observed is that, except for the smallest pores (densest pads), the predicted response is always much faster than the observed response.

When grouped as in eq's 2-5, the parameters of the L-W eq are all available from experiments. The steady state height, h_{ss} , and the pore radius, r , (the only unknown in B) can both be measured independently. As seen, these independent measures of the L-W parameters give a poor fit to the wicking data.

If the parameter B is treated as a free parameter, it can be adjusted to give a better fit, if the estimated pore radius is made much smaller. This implies that the hydraulic resistance is greater than predicted from the eq 2 even when the capillary driving force is correct. Thus a constant contact angle requires an estimate of r larger than the mean, while the correct transient response requires an estimate of r smaller than the mean.

The Lucas-Washburn eq is a two parameter model used to fit the fluid transport data. When the pore radius is measured independently, the L-W eq has a single parameter, Θ , the contact angle. If the L-W eq provides the wrong functional form for the radius, then the calculated contact angle will have to change from experiment to experiment to compensate for the inadequacies of the model. It appears that no single value of the pore radius will adequately represent vertical wicking. In order to represent the steady state height, with a constant contact angle, an estimate of r greater than the mode is required. However, to estimate the rate, an estimate of r smaller than the mode is required.

Horizontal Wicking vs Vertical Wicking.

Horizontal wicking was evaluated in a series of pads and typical data are shown in Figures 16-19. Qualitatively, the wicking front is well modeled by eq 7, which is the trend predicted by the solution of eq 1 for horizontal wicking.

$$l \sim \sqrt{t} \quad (7)$$

After an initial lag, the weight wicked horizontally follows the slope which would be expected if the pad were saturated. That is, it appears as if the first few centimeters behind the flow front are not fully saturated while the remaining volume from the front to the reservoir is saturated. The solution of eq 1 for horizontal wicking is given by eq 8.

$$Q^2 = \frac{r \sigma \cos \Theta}{2\mu} t \quad (8)$$

Using the PSD mode for r , one can estimate $\cos \Theta$. Figure 20 illustrates the effect of the PSD mode on $\cos \Theta$; also, repeated in Figure 20 is the effect of PSD mode on $\cos \Theta$ for vertical wicking. There are two key points to be made from this plot. First, in horizontal wicking, the L-W effective contact angle is a function of the PSD mode and is not a constant. Second, and more critically, the L-W effective contact angle estimated from vertical wicking is a poor predictor of the contact angle in horizontal wicking.

Recall, that eq 8 for horizontal wicking is a different functional form for r than is eq 1 for vertical wicking. If the functional form for r is incorrect, for these low density pads, then one expects $\cos \Theta$, estimated by those equations, to trend with changing pore radius.

Effect of Fluid Properties on Contact Angle

The vertical wicking of the synthetic menses was examined and typical data are shown in Figures 21-23. Eq 2 was used to predict the transient response using the PSD mode as the estimate for r . As was seen for saline, the wicking rate is consistently overestimated. However, if the factor B obtained from a best fit of the saline wicking is used, and corrected for viscosity, then eq 2 predicts accurately the wicking of the viscous fluid.

Eq 2 contains two estimates for r , (hss and B); these two estimates give eq 2 more flexibility than eq 8 for representing wicking data. Since eq 8 has only one occurrence of r , modelling horizontal wicking is a stricter test of the L-W model than vertical wicking.

Figure 24 illustrates typical data for horizontal wicking with "synthetic menses". Eq 8 can be manipulated to yield eq 9 for the ratio of wicking rates for two different fluids.

$$\frac{\text{slope}_1}{\text{slope}_2} = \sqrt{\frac{\cos \Theta_1 \mu_2 \sigma_1}{\cos \Theta_2 \mu_1 \sigma_2}} \sim 2.89 \quad (9)$$

Figure 25 is a test of eq 9. The ratio of the slopes, which should be a function of the fluid parameters only, varies as a function of PSD mode. That is horizontal wicking rates for one fluid are not good predictors of horizontal wicking rates of a second fluid with a different viscosity or surface tension. The errors in the prediction are even greater if the prediction is made from one average pore radius to a different known average pore radius.

CONCLUSIONS

Fluid transport in low density thermally bonded absorbent cores was studied. Experimental measurements of pore size distributions, and of horizontal wicking and vertical wicking with test fluids provided the data needed to evaluate the Lucas-Washburn model for these lofty pads.

The thermally-bonded, lofty absorbent cores were made over a range of densities from 0.04 to 0.18 g/cc, and the fluid transport in these cores is interpreted by means of the measured pore size distributions and the Lucas-Washburn equation (L-W). Pore size distributions were measured using Miller's improved experimental technique and apparatus, and the previously reported bimodal distributions are not observed.

Observed pore size distributions are skewed and fit a log-normal distribution better than a Gaussian. The distributions are narrow enough that the mode should be a good measure of the distribution.

Vertical wicking heights and rates were measured by the EDANA wick test and compared to predictions based on the L-W eq for pure capillary wicking. The analysis, for these lofty, porous structures, indicates that the L-W eq gives correct trends; however, there is no single value of the pore size which adequately fits wicking height and wicking rate data. Using the PSD mode to calculate Θ yields estimates of Θ which are not constant but rather functions of the pore radius.

Horizontal wicking rates were determined for the two test fluids, saline and synthetic menses, and contact angles were estimated based on the Lucas-Washburn eq and the PSD mode. These horizontal wicking contact angles were determined to be a function of the PSD mode and the fluid parameters. The horizontal wicking contact angle is different from the vertical wicking contact angle.

The Lucas-Washburn eq predicts the correct trends for wicking: wick height increases with decreasing pore size; distance wicked horizontally increases as the square root of time; wicking rates are slower with more viscous solutions, etc. However, the equation does not quantitatively predict wicking heights or wicking rates based on independent measurements of pore size or contact angle.

As Chatterjee pointed out earlier (1985), a fundamental flaw of the Lucas-Washburn model is that the capillary driving force and the hydraulic resistance are separate, in fact, yet linked by the model to the pore radius. Thus, one sees that the pore radius which best fits the wicking height data is a poor predictor of the wicking rate data and is a poor predictor of horizontal wicking with other fluids.

Due to our inability to predict fluid transport in one test based on experimental results from a different test, one can expect to continue using a wide variety of special tests in this industry. Those lab tests predict the correct trends in product performance and perhaps rank

alternative cores. Quantitative predictions of product performance in independent tests needs better understanding of the physics of wicking in lofty cores and better models of flow in porous media.

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Table 1 - Experimental Conditions and Primary Data

	nip gap cm	time delay sec's	Density	Vertical Wicking Time vs. cm rise						
				15	30	60	90	120	150	180
1	.368	0	.049	2.8	2.9	3.0	3.1	3.2		
2	.368	8	.043	1.8	1.9	2.0	2.1	2.2		
3	.368	12	.042	1.8	1.9	2.0	2.2	2.3		
4	.262	10	.056	3.7	4.1	4.2	4.4	4.5		
5	.317	4	.047	2.2	2.3	2.4	2.5	2.5		
6	.317	8	.047	2.5	2.7	2.8	2.9	3.0		
7	.317	14	.047	2.4	2.6	2.7	2.8	2.9		
8	.262	1	.054	2.3	2.4	2.5	2.7	2.8		
9	.262	5	.055	2.1	2.3	2.4	2.5	2.6		
10	.262	10	.052	3.2	3.3	3.4	3.5	3.6		
11	.262	14	.054	2.3	2.5	2.6	2.7	2.9		
12	.203	1	.074	3.5	4.0	4.5	4.8	5.0		
13	.203	5	.066	3.6	3.9	4.1	4.3	4.4		
14	.203	14	.073	3.8	4.1	4.4	4.5	4.6		
15	.262	10	.052	2.9	3.2	3.3	3.4	3.5		
16	.168	5	.091	4.3	4.9	5.1	5.6	5.9		
17	.168	9	.089	4.2	4.8	5.2	5.8	6.3		
18	.114	1	.170	4.5	5.6	7.3	8.2	8.8	9.5	9.8
19	.114	3	.178	4.2	5.3	6.8	7.8	8.4	8.9	9.4
20	.114	11	.165	4.3	5.7	7.1	8.1	8.6	9.2	9.5
21	.114	14	.181	4.1	5.4	7.0	8.1	9.0	9.1	10.4
22	.262	10	.056	3.0	3.3	3.4	3.5	3.6		

Table 2 - Vertical Wicking With Synthetic Menses

Pad		Time seconds						
		15	30	60	90	120	150	180
13	vertical rise, cm	2.3	2.5	2.9	3.2	3.5		
16	"	2.1	2.4	3.0	3.4	3.7		
21	"	2.1	2.6	3.4	3.9	4.5	4.8	5.1

8320h/2

Table 3 - Wet Pore Size Data

Pressure cm-H ₂ O	Pad No.	Radius, microns	1	2	3	4	5	6	7	8	9	10	11
			% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol
2.		367.	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
4.		183.	97.30	97.05	96.31	96.95	96.95	96.54	96.58	98.53	98.37	98.48	97.26
6.		122.	94.41	93.65	91.24	93.41	93.30	94.66	94.63	96.43	94.06	93.32	95.57
8.		91.	89.95	87.85	85.97	89.87	88.64	91.00	90.14	93.29	91.29	90.29	89.78
10.		73.	73.11	56.94	75.62	85.15	63.89	67.68	78.04	77.88	69.56	68.14	70.94
12.		61.	46.88	34.03	38.50	53.88	37.52	37.84	39.02	44.54	43.15	43.27	44.94
14.		52.	32.74	25.57	25.97	35.49	27.38	26.87	26.14	28.40	30.81	36.29	30.63
16.		45.	24.09	19.31	20.99	25.17	20.68	21.44	20.48	24.42	22.96	25.58	22.84
18.		40.	19.53	15.63	16.51	19.86	17.24	17.88	17.07	19.70	18.85	21.84	18.94
20.		36.	15.90	13.43	14.12	16.81	13.99	14.82	14.63	15.72	15.78	17.08	16.00
22.		33.	13.86	11.49	12.33	14.45	12.27	12.45	12.48	13.83	13.49	14.96	13.89
24.		30.	12.27	10.48	11.14	12.58	10.95	10.96	11.12	12.26	11.67	13.14	11.78
26.		28.	11.16	9.38	9.85	11.30	9.63	9.88	10.14	10.79	10.43	11.93	10.94
28.		26.	9.86	8.73	9.05	10.32	8.92	8.99	9.26	10.06	9.47	11.02	9.89
30.		24.	9.11	8.09	8.45	9.63	8.21	8.39	8.58	9.32	8.89	10.01	9.05
40.		18.	6.88	6.25	6.36	7.17	5.88	6.22	6.52	6.91	6.60	7.58	6.73
50.		14.	5.76	5.42	5.37	5.99	4.96	5.33	5.56	5.66	5.35	6.37	5.68
60.		12.	4.74	4.59	4.87	5.30	4.05	4.34	4.78	5.13	5.07	5.76	5.05
70.		10.	4.27	4.32	4.27	4.62	3.85	3.95	4.19	4.50	4.21	5.15	4.42
80.		9.	3.81	4.04	3.58	4.22	3.54	3.65	4.00	4.08	3.63	4.95	4.10
90.		8.	3.44	3.77	3.58	3.93	3.24	3.16	3.80	3.98	3.44	4.75	3.89
100.		7.	3.34	3.67	3.48	3.83	3.04	3.06	3.51	3.77	3.34	4.44	3.68

8320h/2

Table 3 -- Wet Pore Size Data -- (Continued)

Pressure cm-H ₂ O	Pad No.	Radius, microns	12	13	14	15	16	17	18	19	20	21	22
			% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol	% Vol
2.		367.	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
4.		183.	99.19	98.44	98.41	97.11	98.49	98.45	99.48	98.69	98.91	99.16	97.82
6.		122.	98.16	94.15	96.15	94.33	96.99	94.65	98.20	96.86	97.82	98.32	92.56
8.		91.	96.33	92.24	93.44	90.75	95.62	94.23	97.17	94.77	96.73	96.92	91.83
10.		73.	90.60	78.75	84.40	75.14	93.84	92.12	95.62	92.68	94.55	96.08	89.83
12.		61.	64.94	53.81	62.03	45.42	74.82	76.23	94.08	91.90	92.91	95.25	78.31
14.		52.	42.03	36.99	38.98	30.41	54.03	52.18	91.77	89.55	90.19	93.29	50.81
16.		45.	31.95	26.37	30.50	23.45	38.98	38.95	89.46	87.72	84.74	90.50	33.57
18.		40.	24.28	20.52	24.18	19.08	30.50	30.37	84.83	85.11	73.56	83.79	25.40
20.		36.	20.16	17.54	20.00	15.60	24.35	26.16	77.89	80.15	62.94	80.44	21.32
22.		33.	17.41	14.91	16.72	13.61	20.93	21.23	62.98	68.40	51.49	69.83	17.42
24.		30.	15.69	13.12	14.68	11.92	18.33	18.56	52.95	57.70	42.23	56.42	14.88
26.		28.	13.97	12.05	13.44	10.63	16.14	16.59	44.21	50.91	38.14	49.32	13.33
28.		26.	12.82	10.97	12.20	9.64	14.63	15.18	40.35	44.12	34.05	39.66	11.79
30.		24.	11.68	10.14	11.07	8.74	13.26	13.36	35.98	38.12	20.98	35.75	9.98
40.		18.	9.04	7.51	8.02	6.66	9.57	10.12	23.13	24.02	16.07	23.46	8.62
50.		14.	7.78	6.32	6.89	5.26	7.52	8.01	17.99	18.79	13.35	18.43	7.53
60.		12.	6.64	5.48	5.53	4.47	6.70	7.17	15.42	15.66	11.98	15.64	6.17
70.		10.	6.07	5.13	5.31	4.07	6.01	6.46	13.11	13.57	10.89	13.68	5.62
80.		9.	5.49	4.77	4.97	3.77	5.33	6.04	12.08	12.53	9.53	13.40	5.17
90.		8.	5.15	4.29	4.63	3.67	5.06	5.48	10.53	11.22	9.26	12.01	4.90
100.		7.	5.04	4.17	4.40	3.47	4.78	5.20	9.51	10.18		11.45	4.44

8320h/3

"Thru-Air" Heater

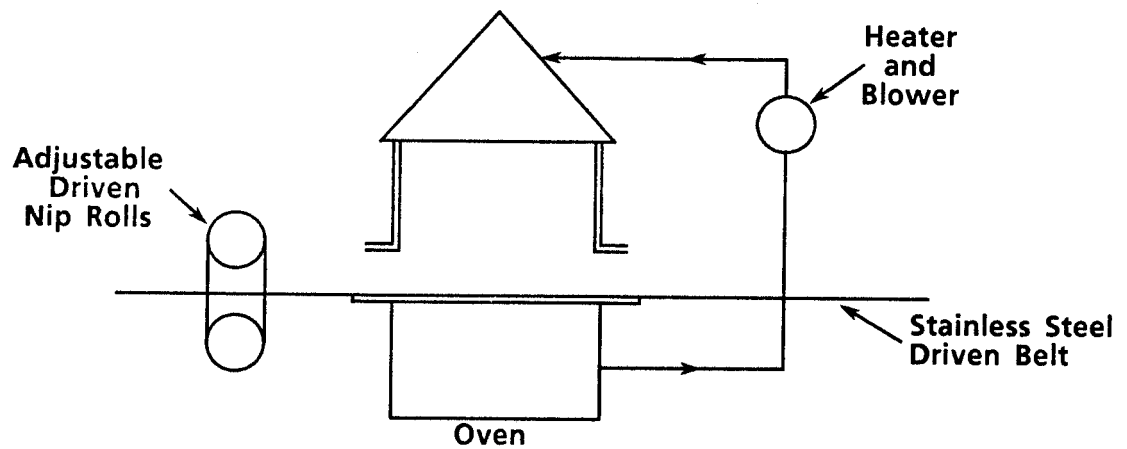


FIGURE 1

Edana Vertical Wicking

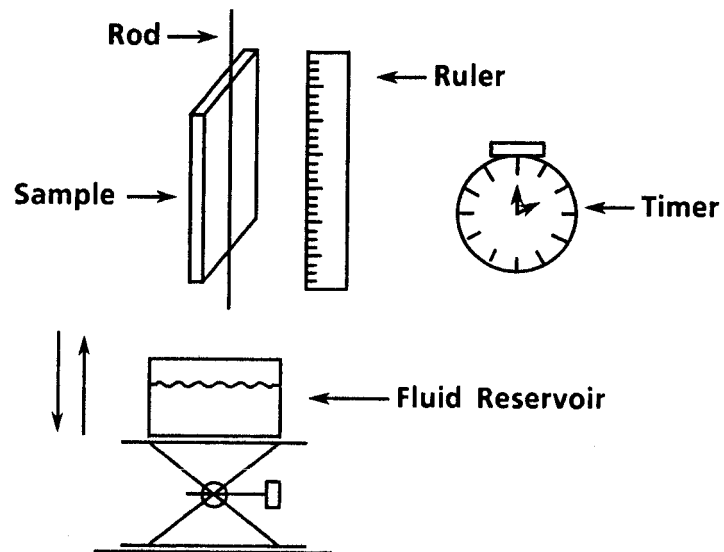
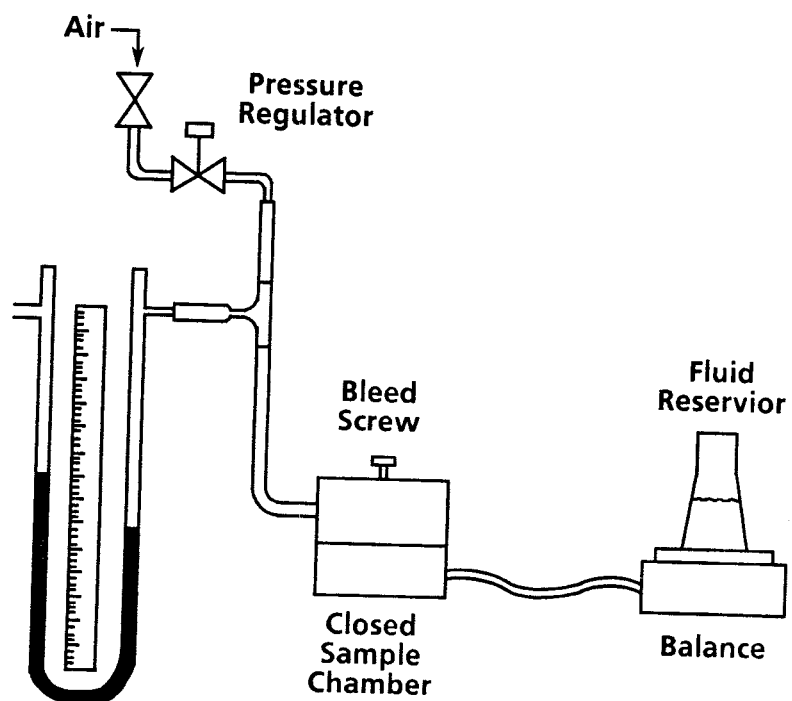


FIGURE 2

Extended Pore Size Apparatus



Schematic of Sample Cell

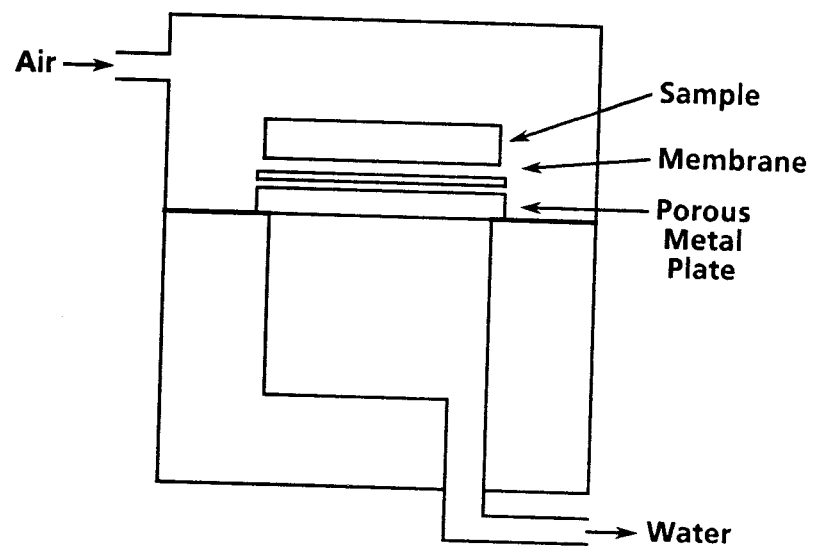


FIGURE 3

FIGURE 4A. WET PORE SIZE DISTRIBUTION
PAD 1

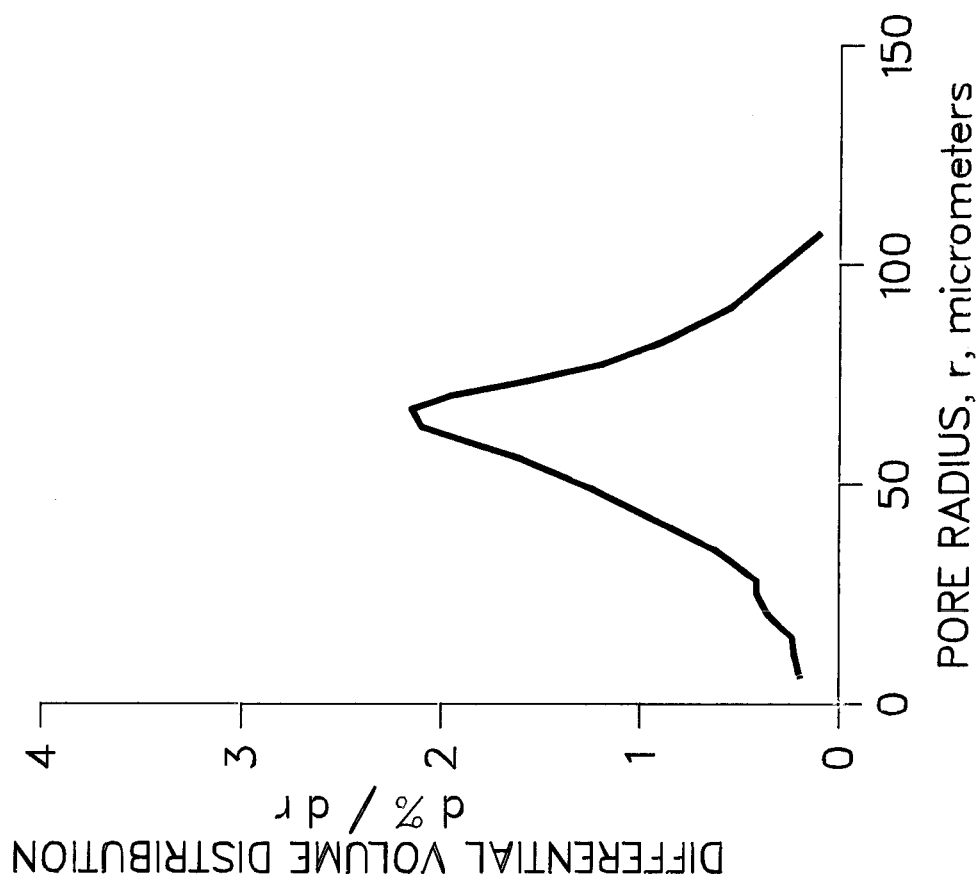


FIGURE 4B. LOG NORMAL DISTRIBUTION
PAD 1

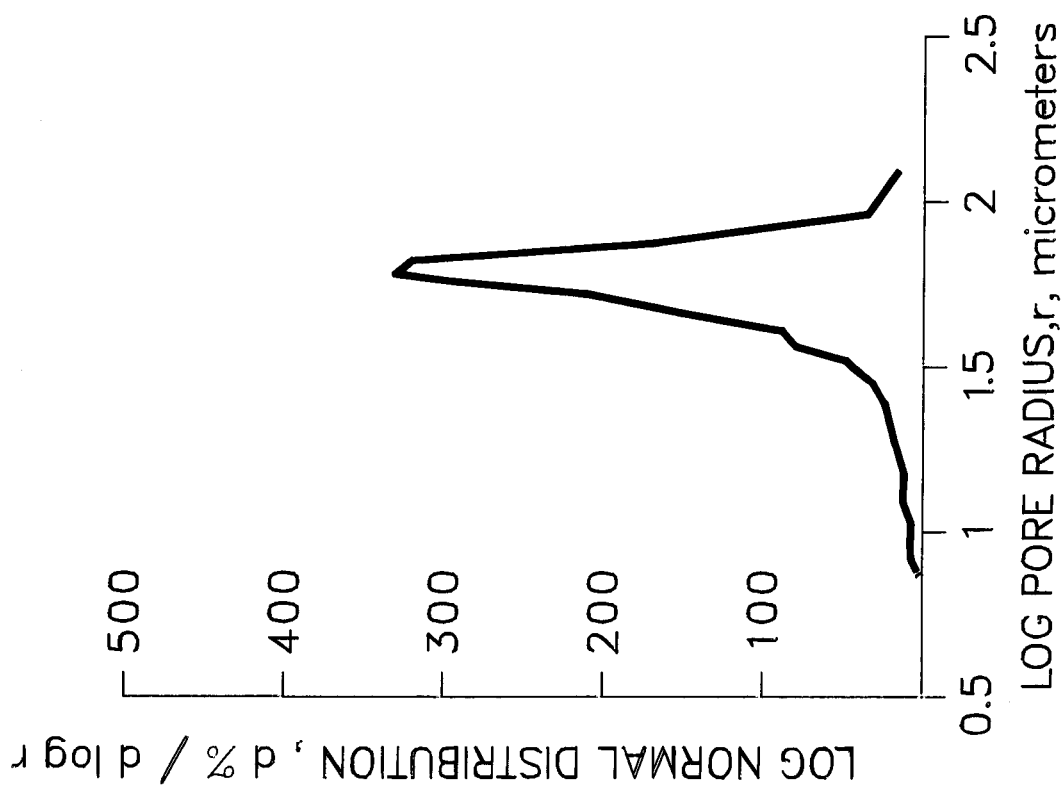


FIGURE 5. WET PORE SIZE DISTRIBUTION
PAD 16

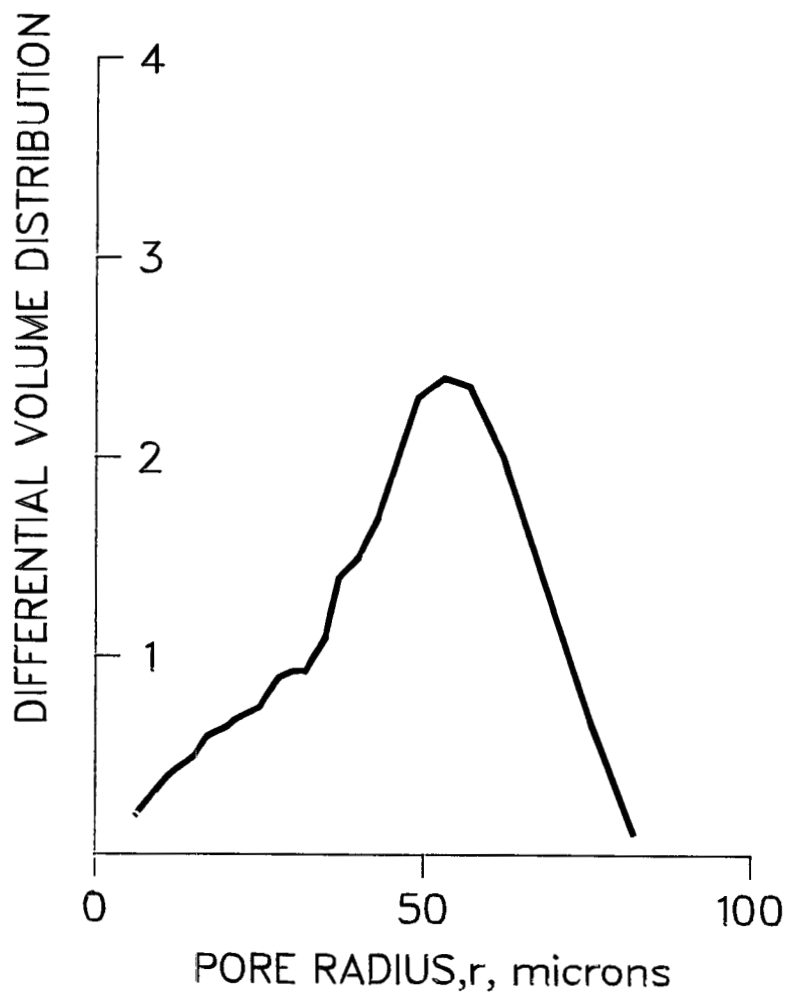
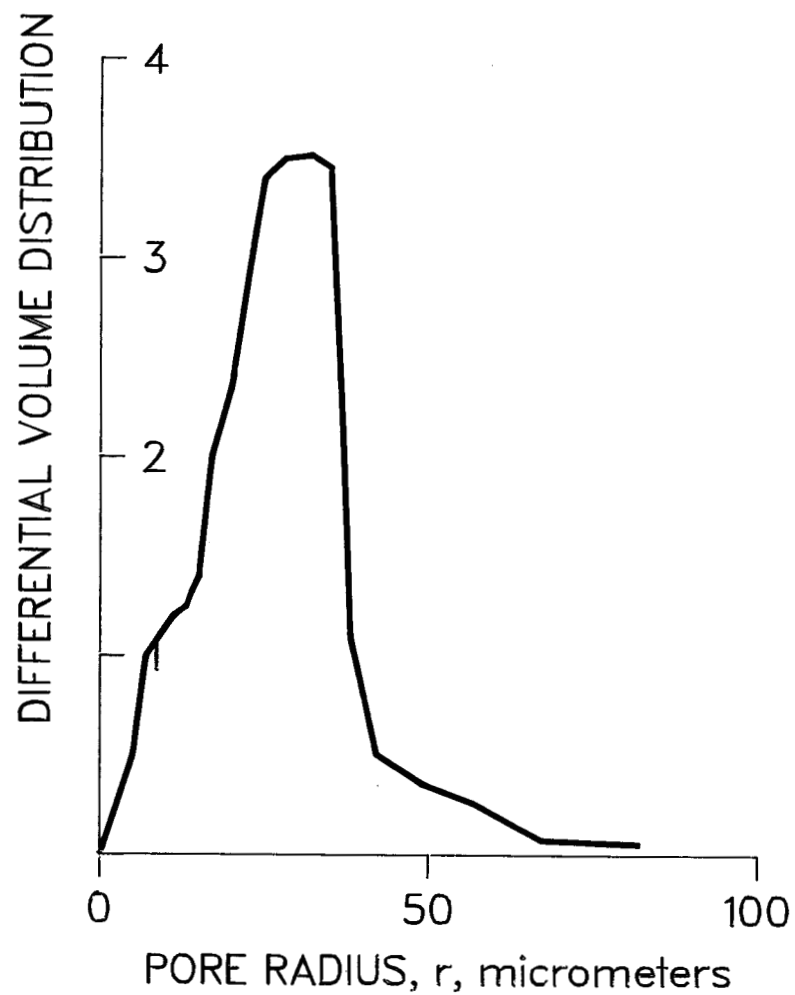


FIGURE 6. WET PORE SIZE DISTRIBUTION
PAD 19



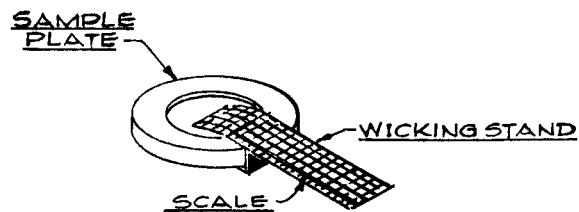


FIGURE 7A

HORIZONTAL WICKING STAND

FIGURE 7B

Schematic Representation of Gravimetric Absorbency Testing System

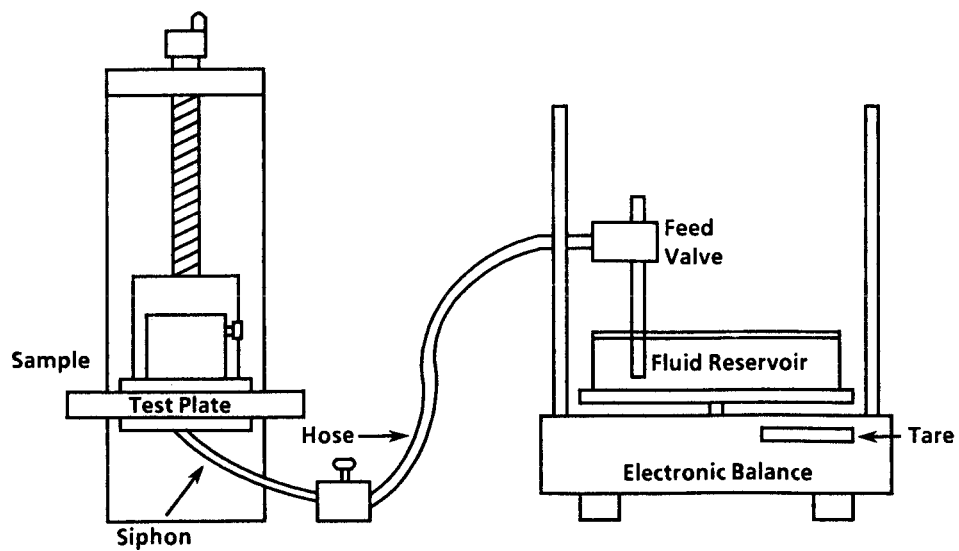


FIGURE 8. WET PORE SIZE DISTRIBUTIONS
PREVIOUS METHOD VS IMPROVED METHOD

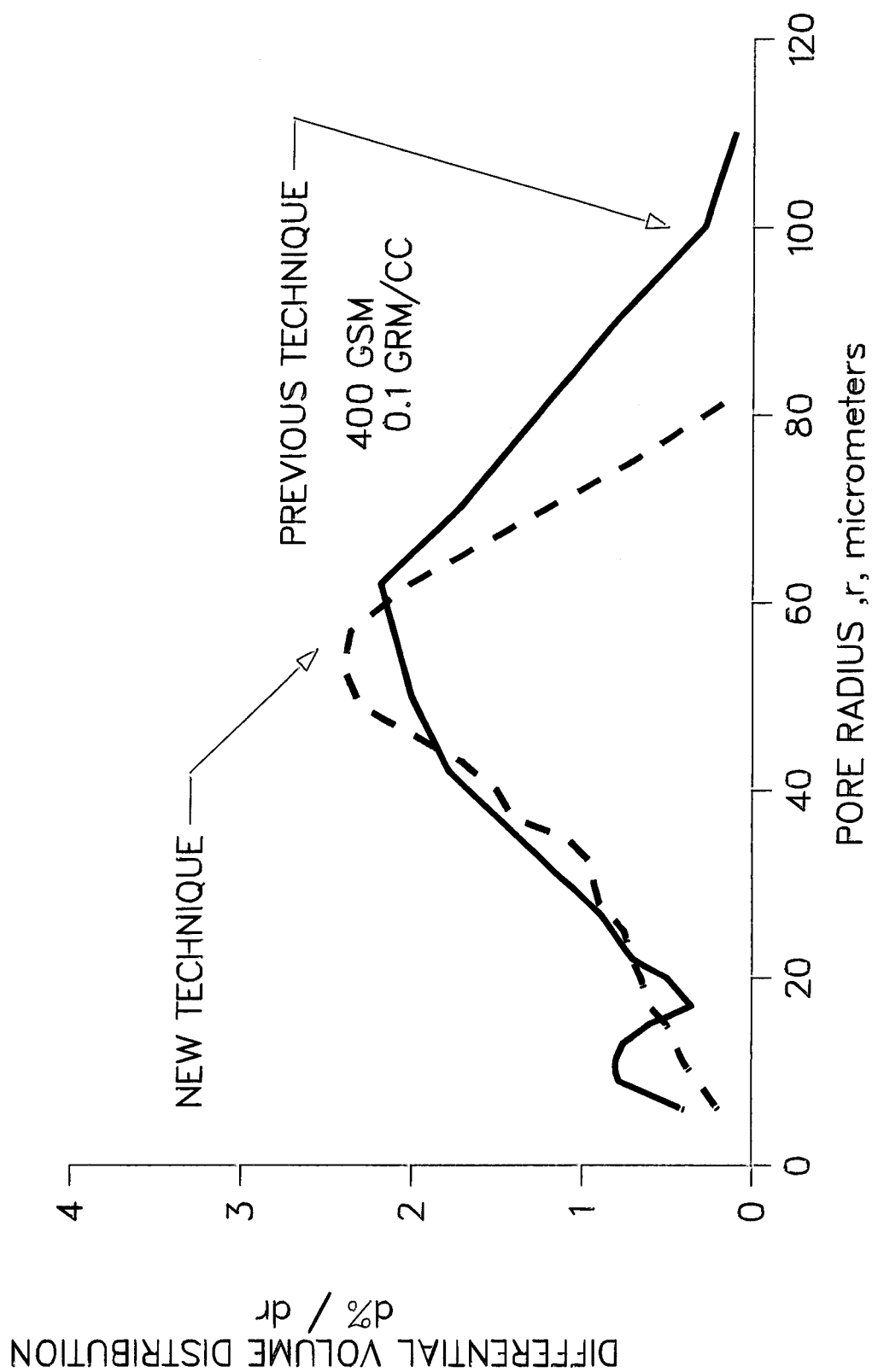


FIGURE 9A

EFFECT OF NIP GAP ON PAD

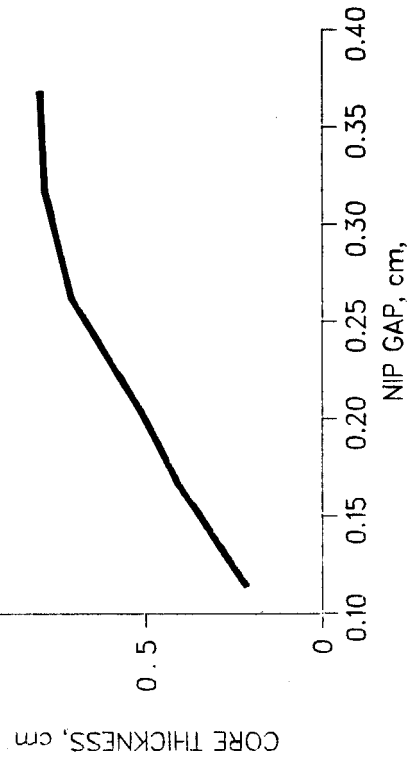
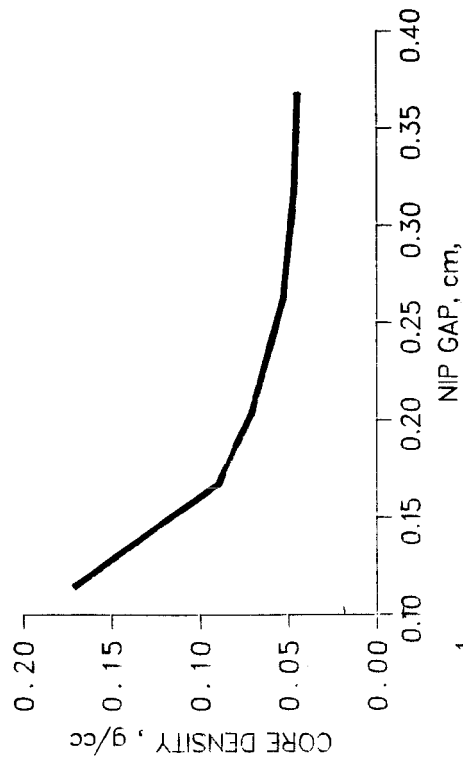


FIGURE 9B

EFFECT COOLING ZONE ON THICKNESS

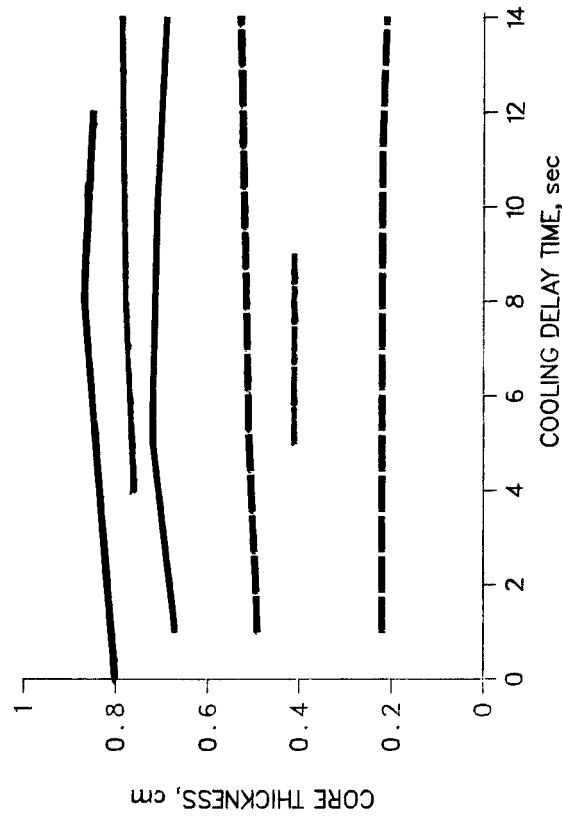


FIGURE 9C

FIGURE 10. HEIGHT WICKED
VS PSD MODE

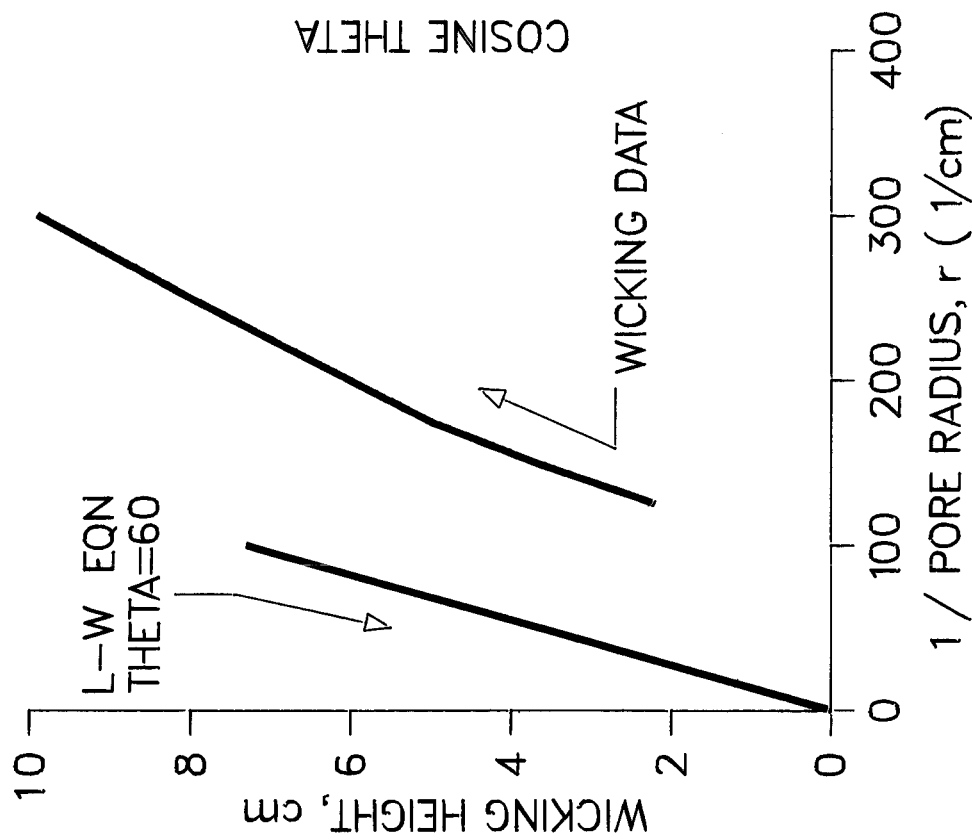


FIGURE 11. EFFECT OF PSD MEAN
ON APPARENT CONTACT ANGLE

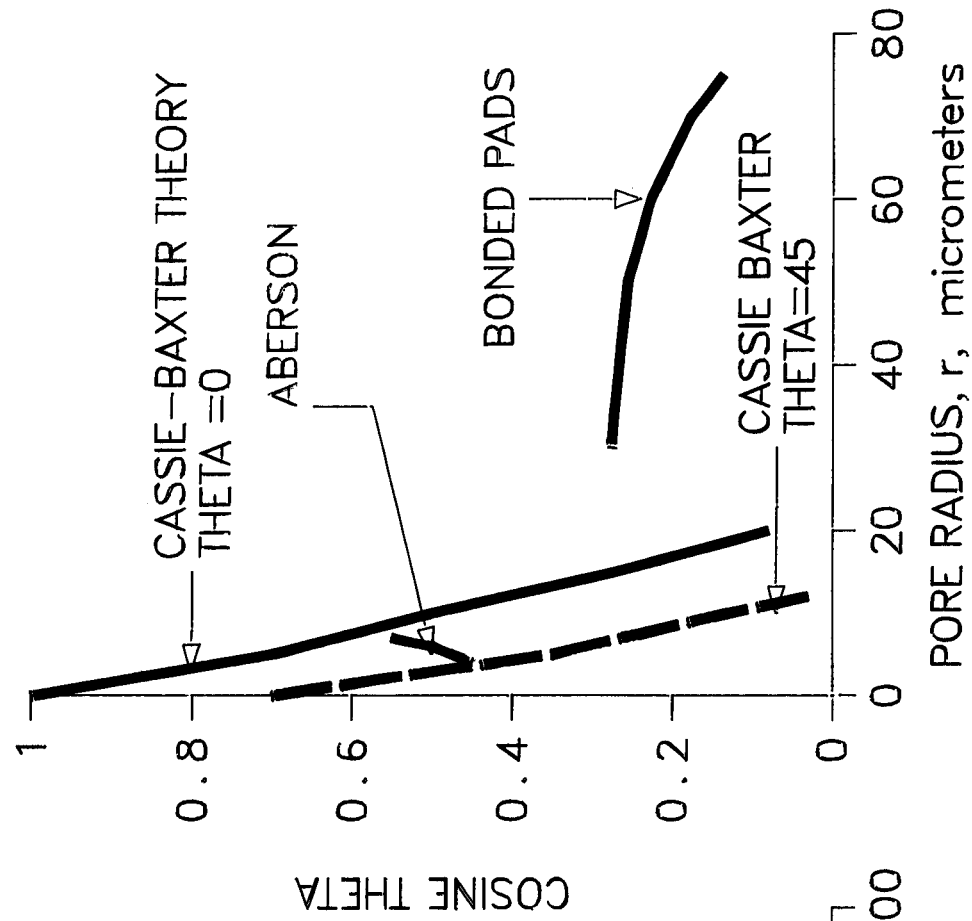


FIGURE 12. VERTICAL WICKING HEIGHT vs TIME
PAD 1

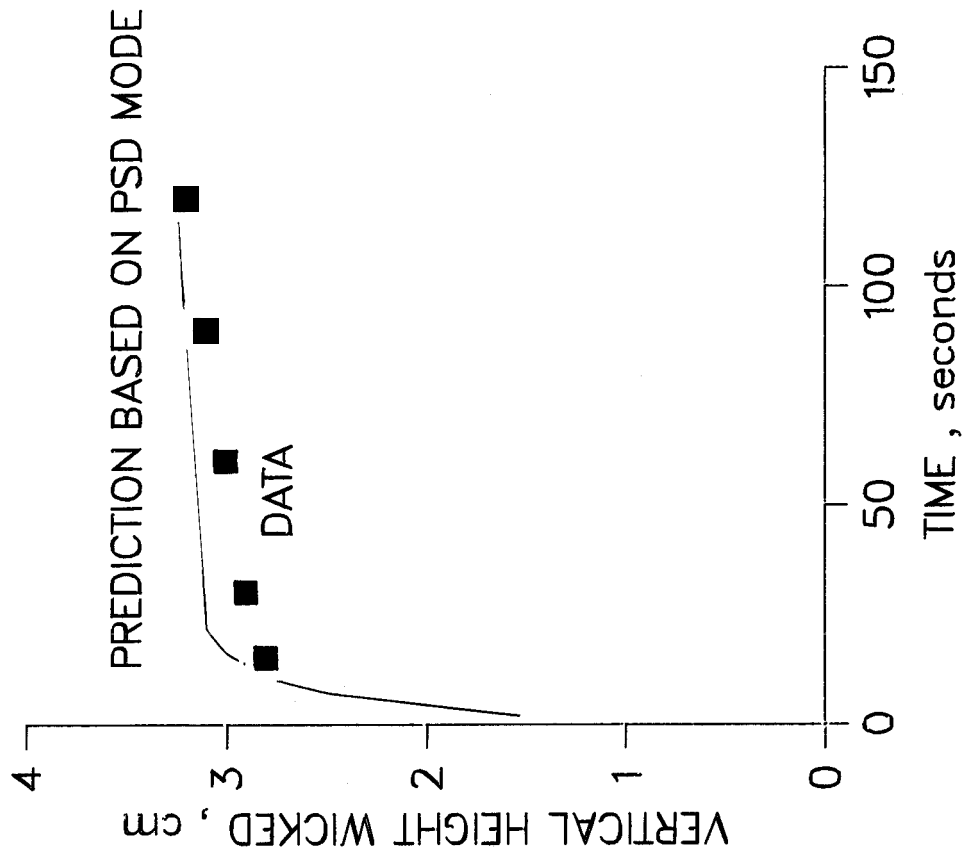


FIGURE 13. VERTICAL WICKING HEIGHT vs TIME
PAD 2

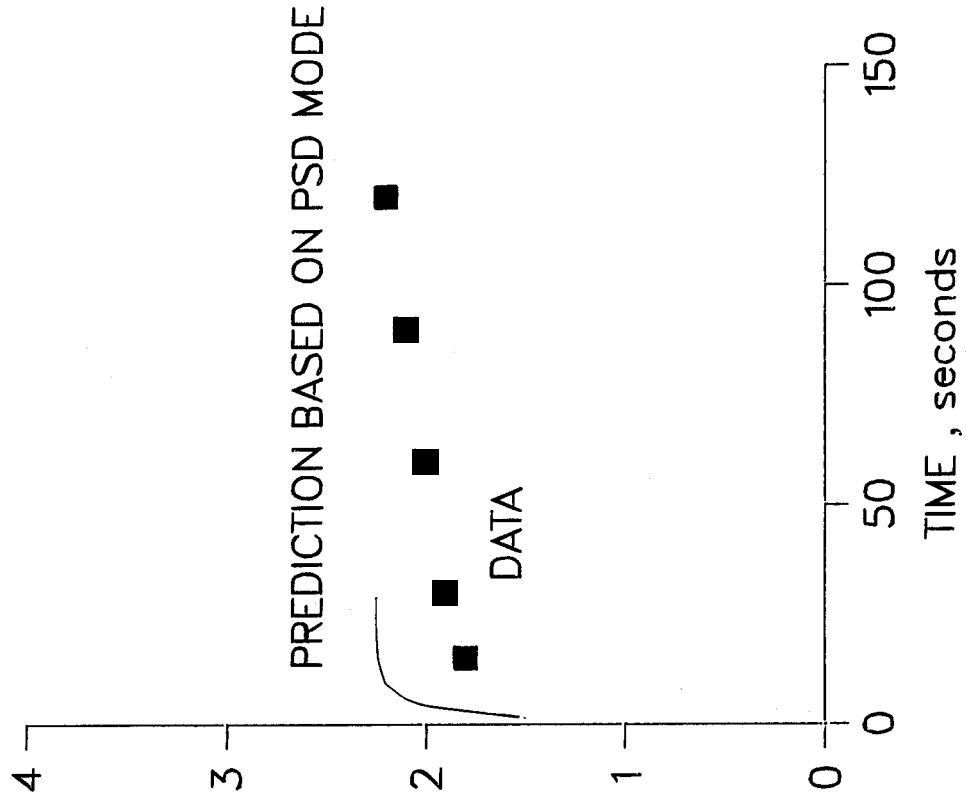


FIGURE 14. VERTICAL WICKING HEIGHT vs TIME
PAD 12

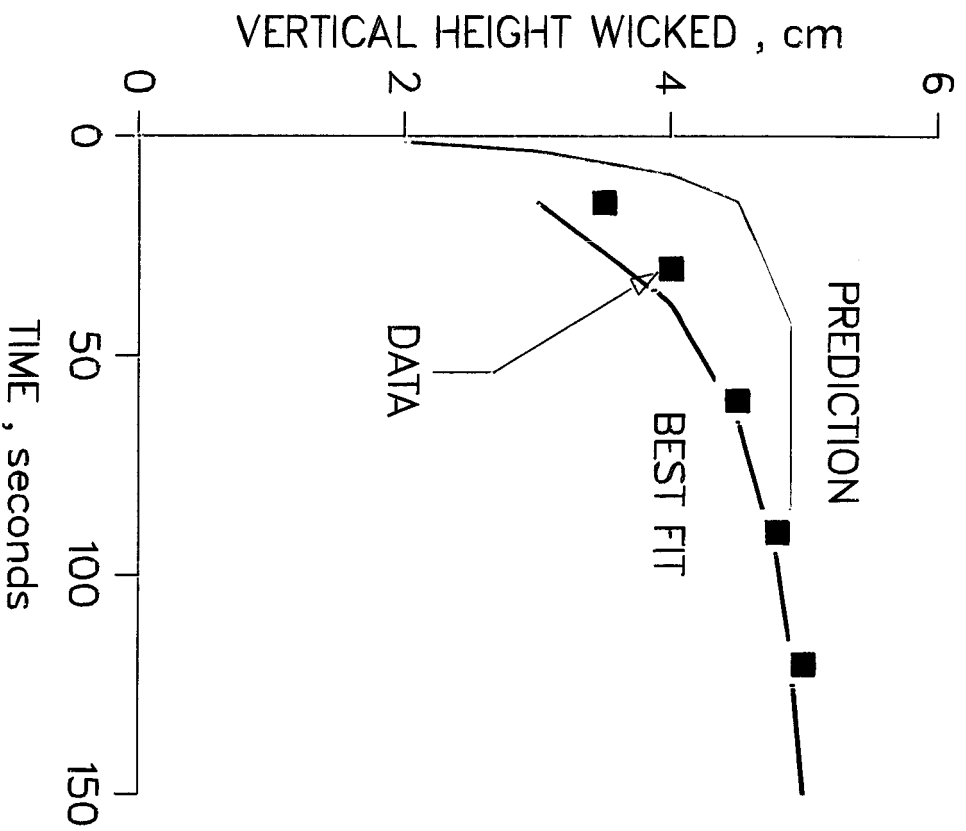


FIGURE 15. VERTICAL WICKING HEIGHT vs TIME
PAD 21

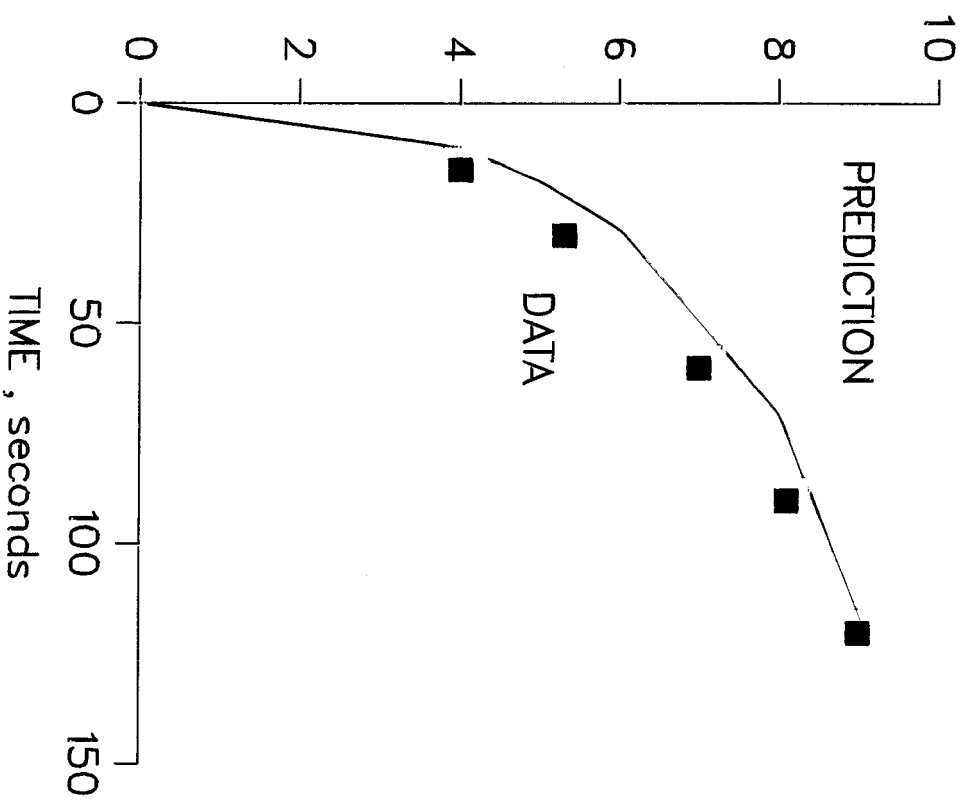


FIGURE 16. HORIZONTAL WICKING
PAD 2

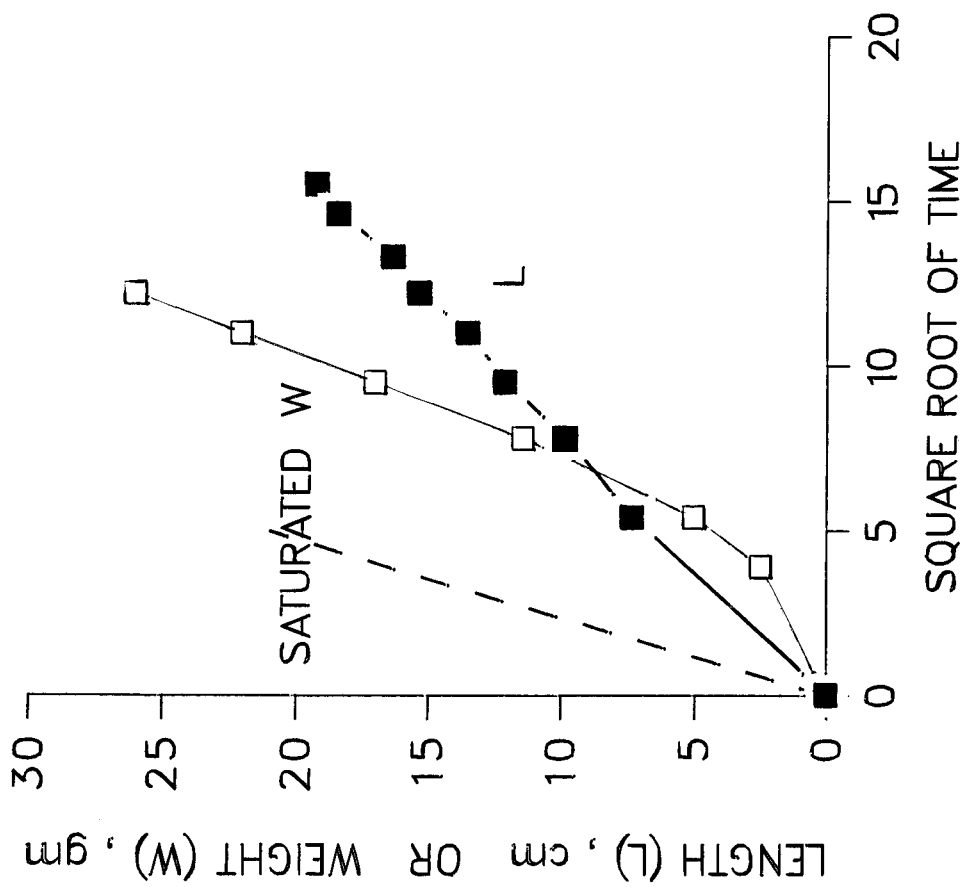


FIGURE 17. HORIZONTAL WICKING
PAD 14

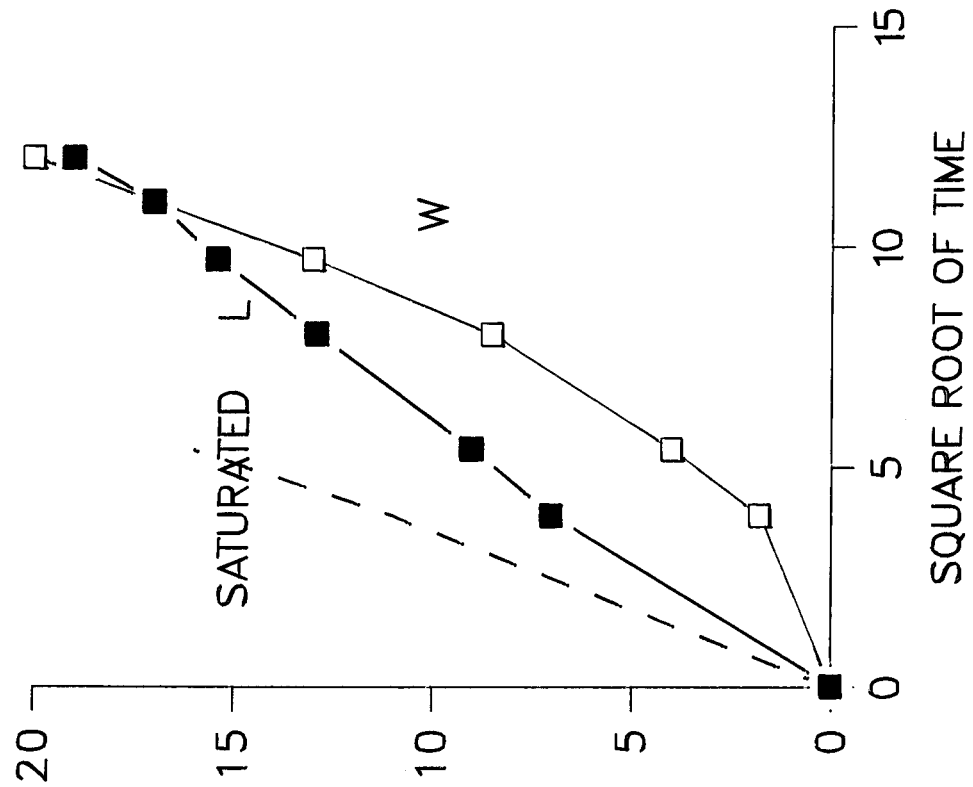


FIGURE 18. HORIZONTAL WICKING
PAD 17

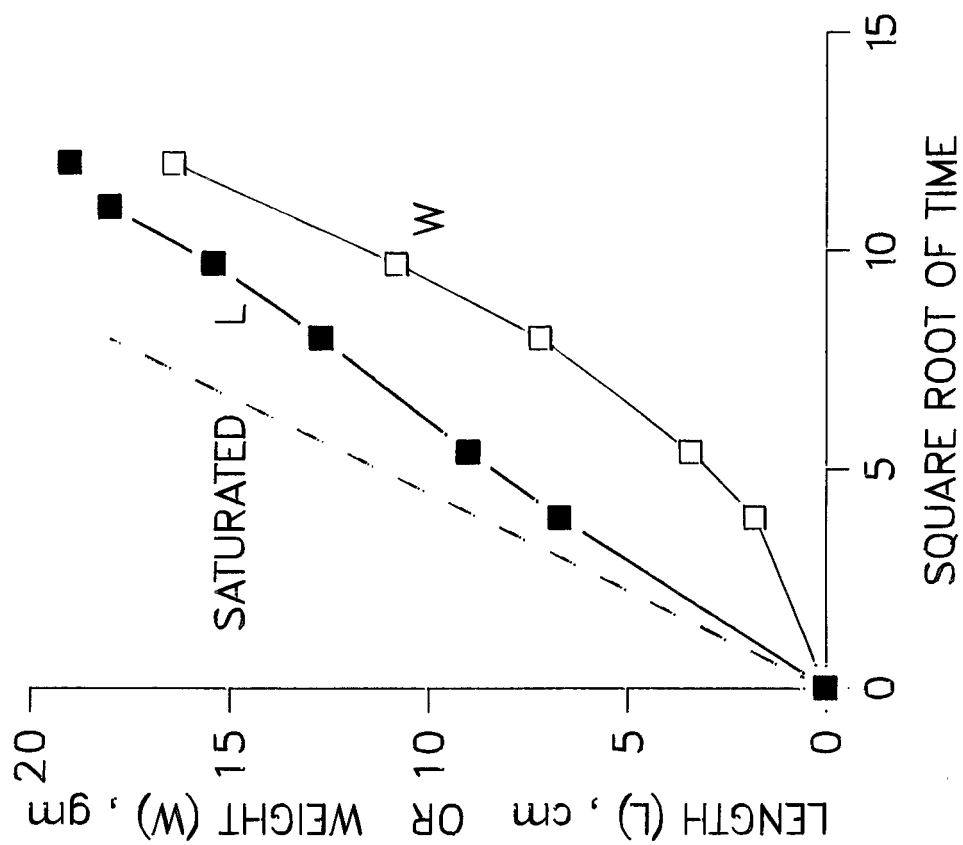


FIGURE 19. HORIZONTAL WICKING
PAD 21

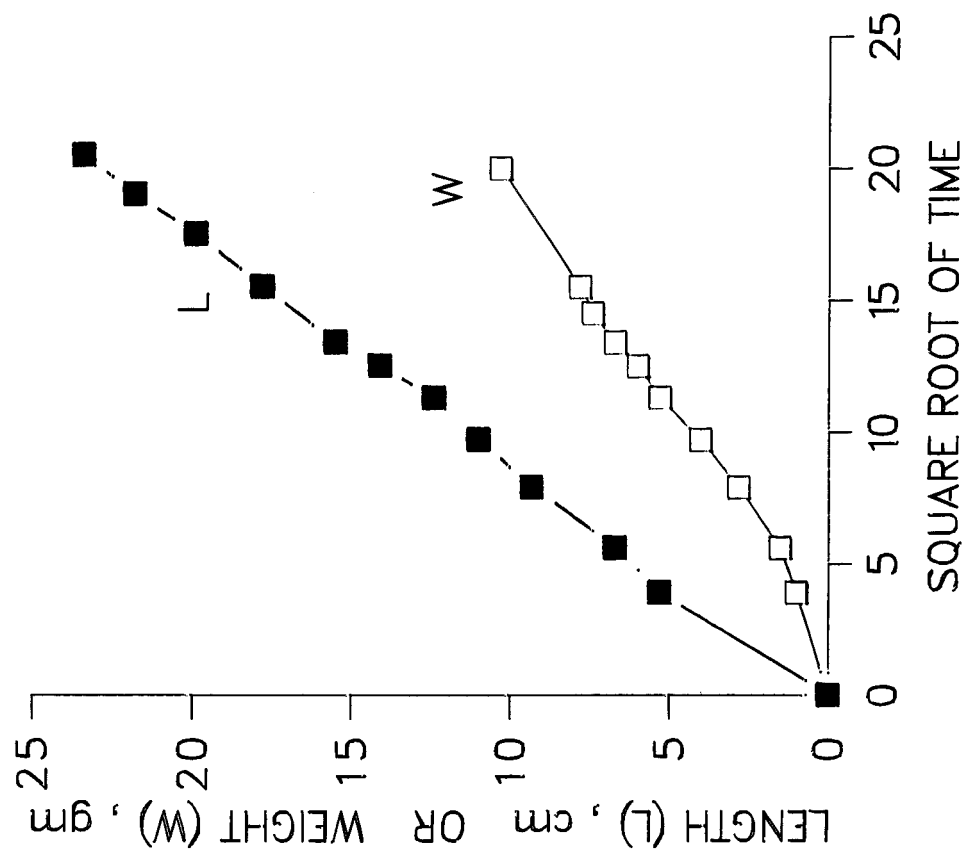


FIGURE 20. CALCULATED CONTACT ANGLE
BASED ON PSD MODE

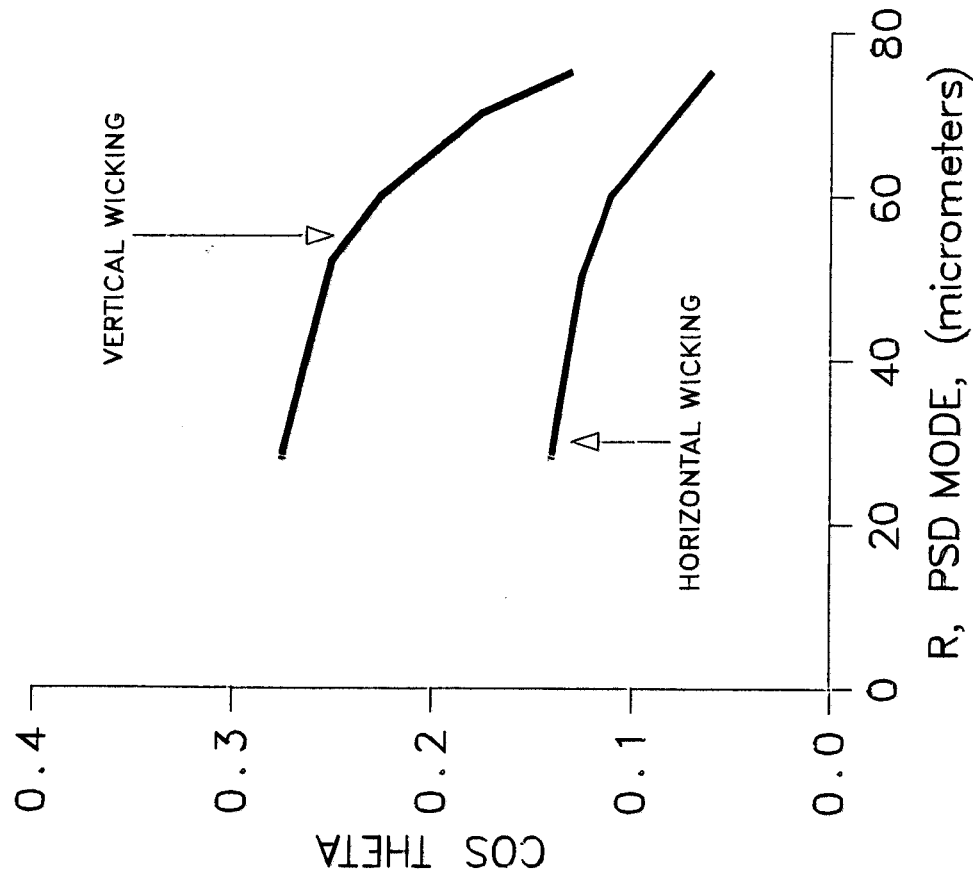


FIGURE 21. VERTICAL WICKING WITH VISCOUS FLUID
PAD 13

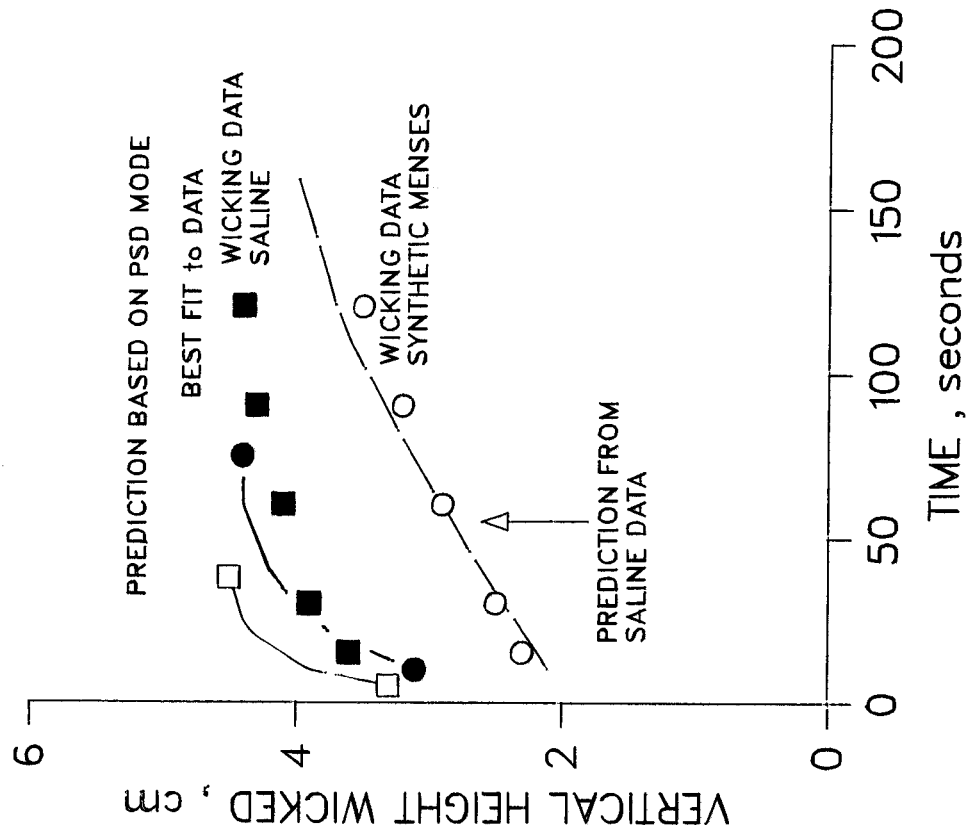


FIGURE 22. VERTICAL WICKING WITH VISCOUS FLUID
PAD 16

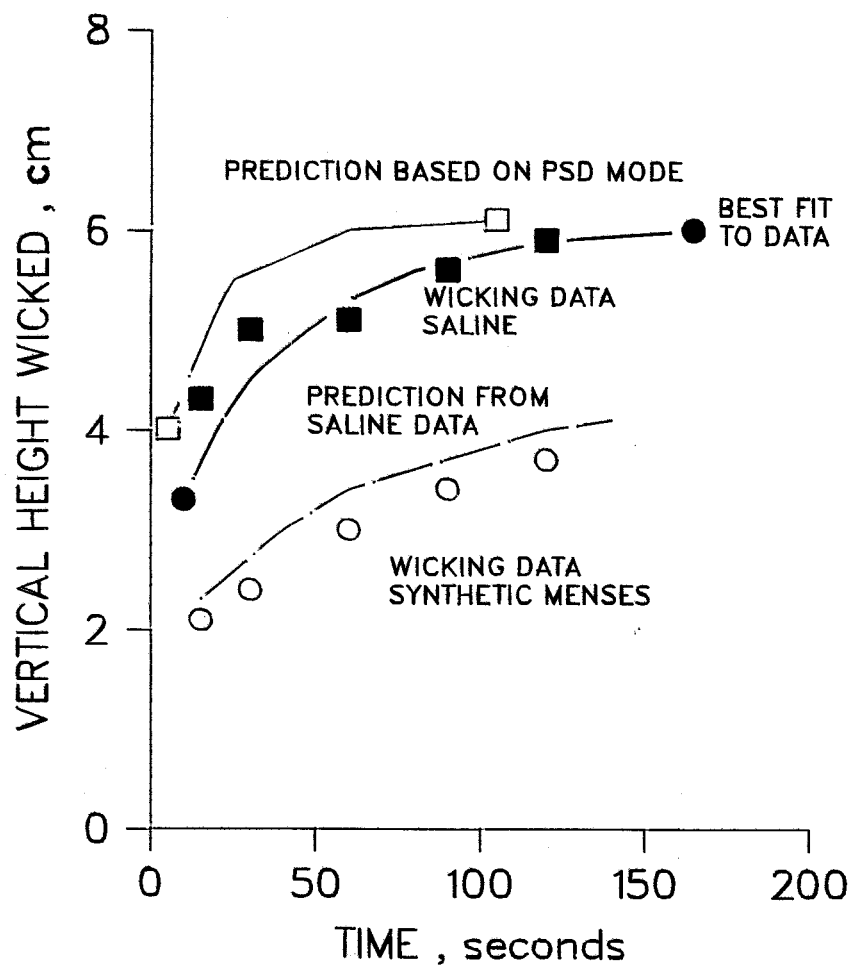


FIGURE 23. VERTICAL WICKING WITH VISCOUS FLUID
PAD 21

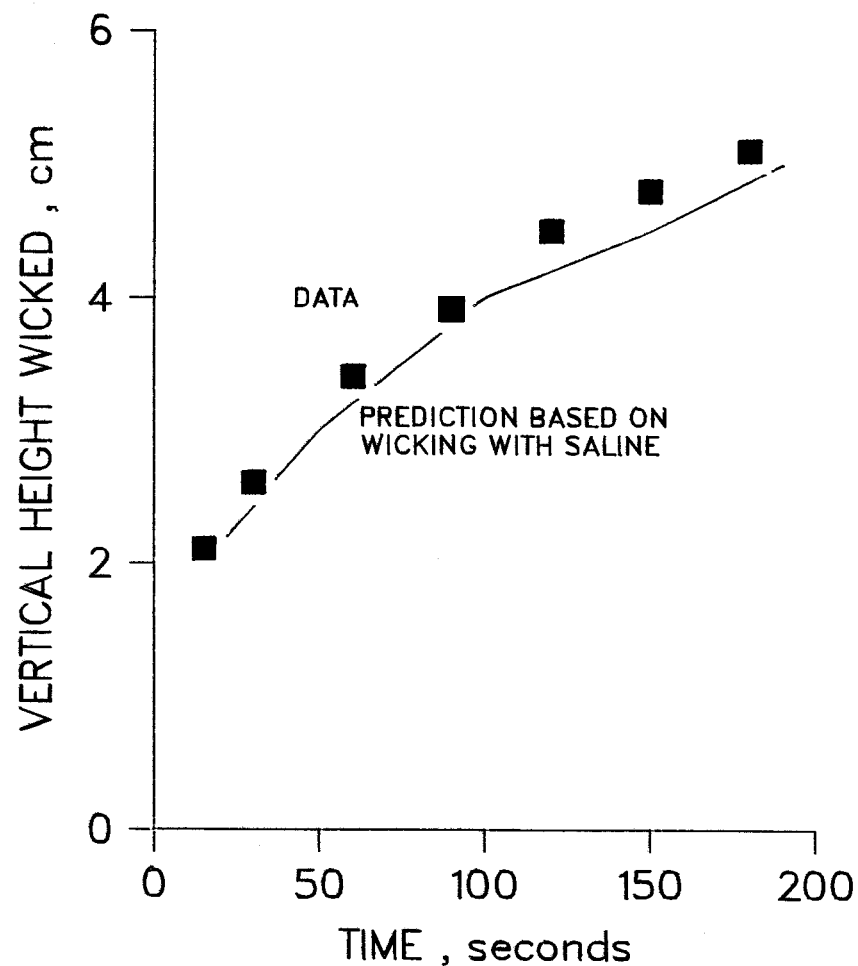


FIGURE 24. HORIZONTAL WICKING OF VISCOUS FLUID
PAD 2

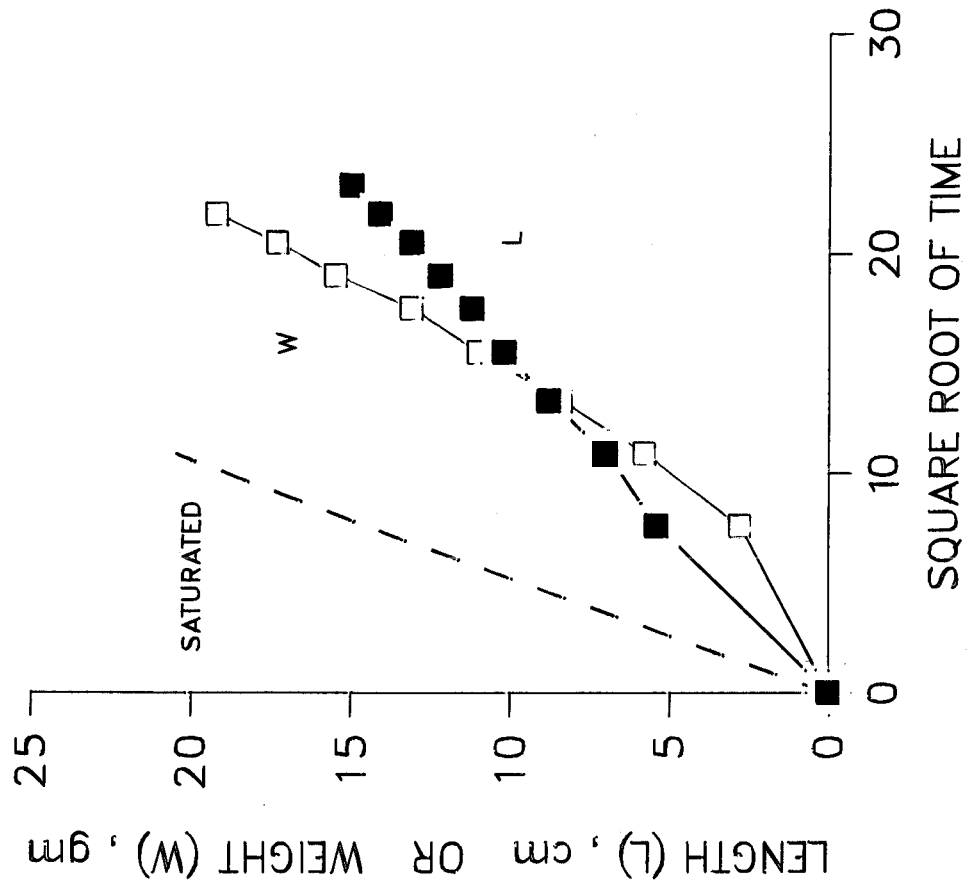
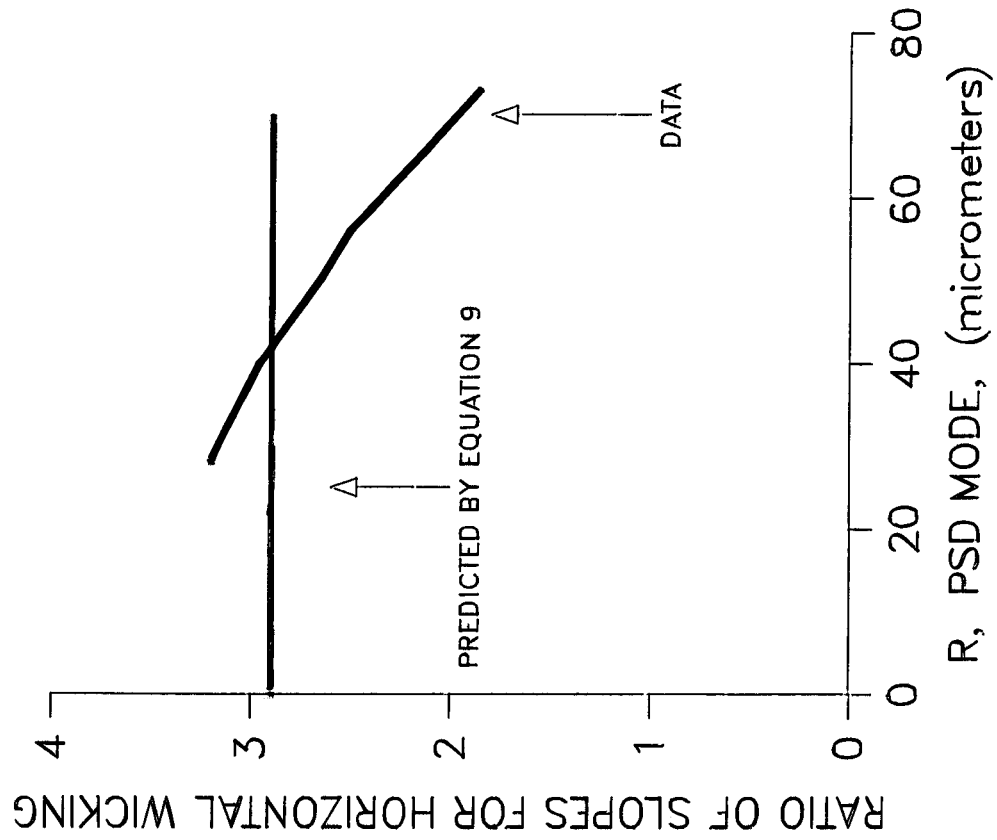


FIGURE 25. TEST FOR CONSTANT CONTACT ANGLE
HORIZONTAL WICKING WITH VISCOUS FLUID



FLUID MIGRATION IN NON-WOVEN CELLULOSIC ABSORBENTS
by Computed X-ray Tomography

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1.0 ABSTRACT

X-ray computed tomography is used to observe, in real time, the spatial distribution of water during injection at constant flow rate into non-woven porous absorbents. Examples given are diapers, tampons, feminine napkins with and without superabsorbent gel and simple filter paper. Results indicate the quantitative nature of the measurement and show regimes of displacement in which viscous or capillary forces dominate radial fluid displacement.

2.0 INTRODUCTION

The migration of fluid into a porous media which has been investigated since the time of D'Arcy, plays a major role in such diverse areas as nutrient delivery to plants, and recovery efficiencies of oil, gas or sometimes water from the porous reservoir rocks. Fibrous material, cellulosic, proteinous or synthetic, whether woven or not are porous structures. In that subgroup wherein fibrous materials are used for absorbency, the dynamics of fluid motion within the structure is the key to effectiveness. Chatterjee (1) has reviewed this subject. In this research we report on a new technique, X-ray computed tomography, for the quantitative observation of water absorption with non-woven absorbents.

Consider water flowing into a non-woven porous absorbent at a constant controlled rate. There are two dominant phenomena, the viscous two phase flow (air being displaced by water) and capillarity (cellulose absorbents are spontaneous wet by water). The physical packing of the fibers creates a porosity and an effective pore which has a distribution in size. This distribution dictates that capillary forces are distributed. In the small pores (where fibers are close together) imbibition is strong while in the more open regions, capillary effects may be small. In a single capillary, the Washburn equation describes fluid flow. Due to capillary effects alone Willemsen and others (2,3) have used invasion percolation models to describe fluid distributions in random porous networks. However, the interface between air and water is hydrodynamically stable (4). The relatively high viscosity water relative to air, creates viscous forces that tends to make the air-water fluid boundary planar and sharp, thus opposing the capillary dominated tendency for the interface to disperse. In addition to the porous structure itself, there is the swelling of the fibers and in some instances the localized swelling of super absorbent crosslinked polymers that significantly alters the migration of fluids.

Many classical studies have been made to describe the dynamics of water imbibition into cellulose by capillarity. A diffusion analogy has been used by Ruoff (5, 6), Fujita (7) and Gillespie(8) to describe the dynamics of this water sorption. A similar approach has been used by Gardner and Mayhugh (9) to correlate the migration of water in soil. In contrast to these studies, the present paper describes an experimental technique wherein the spatial distribution of fluid can be observed directly in a non-invasive and nondestructive manner. Gryte and coworkers (10-13) have demonstrated the application of CT to observation of oil-water displacement in porous reservoir rock such as sandstone and to the quantification of sedimentation of dilute solid suspensions (14).

Buy using as examples, a diaper, a tampon, feminine hygiene absorbents and simple filter paper, the range of application of computed tomography will be extended to the investigation of absorbency.

3.0 METHOD

3.1 CT Scanner

Experiments were carried out on a modified second generation EMI 5005 body scanner that is schematically represented in Fig. 1. A 120 kev, X-ray beam with a fan configuration falls on a detector containing 30 photomultipliers that record X-ray intensity. The source-detector assembly translates recording 761 sets of data as it moves across the image. The source-detector assembly then rotates 9° , the translation data are collected and this is repeated at 9° increments for a full 180° rotation of the gantry. A 320 by 320 data image is created for the 25

cm image area scanned. Nominally, the beam width is 8 mm and each pixel has dimensions 0.76 mm by 0.76 mm. The scan time is 20 s .

3.2 Flow Assembly

Flow experiments were carried out by mounting absorbent materials between two circular sheets of poly(methyl methacrylate), this being support, as shown in Fig.2, so that its position is within the tomographic plane of the CT scanner. Fluid is injected at constant rate as shown using a peristaltic pump (Rainin Instruments, Woburn, Mass.). During the mounting of the absorbent care was taken to ensure that the gap between the plates was constant, in all cases the absorbent was slightly compressed by the plates. Nylon bolts, transparent to X-rays secured these plates. After centering the absorbent, and recording the image of the absorbent in the absence of water, flow began and images were recorded at convenient intervals. After completion of an experiment, an absorbent was completely saturated with water and this image was also recorded. The local X-ray attenuation coefficient was recorded for each pixel position of the image.

3.3 Absorbent Materials

Standard commercial absorbent materials were investigated: a diaper, tampon, napkin and superabsorbent incontinency pad, together with the reference behavior of multiple sheets of Whatman Number 2 filter paper. Since only a few experiments were done on a limited number of samples, trade names have been omitted. In most cases distilled water was injected into the absorbent.

3.4 Analysis of the Data

The CT algorithm gave for each pixel, ij , an integer number proportional to the linear attenuation coefficient, μ_{ij} . From Fig. 2, the width of the beam, 8mm, dictates that each volume element attenuates X-rays because it contains a constant amount of poly(methylmethacrylate), and cellulose, and a variable amount of water. It is assumed that the air in the void spaces does not absorb radiation. Because the densities are similar and close to that of water for which the CT scanner is calibrated, the effect of beam hardening (the increase of X-ray average energy during the passage through a material which results from the larger attenuation of the low energy part of the beam) was shown to be negligible.

If μ_{ij} is the experimental attenuation at pixel ij , $\mu_{a,c}$ is the attenuation in the absence of water, and $\mu_{w,c}$ is the attenuation of the

absorbent when completely saturated with water, then the water fraction x_{ij} in the void spaces can be computed for each picture element.

$$x_{ij} = (\mu_{ij} - \mu_{a,c}) / (\mu_{w,c} - \mu_{a,c}) \quad (1)$$

Since the poly(methylmethacrylate) is constant, it does not affect the computation. For a quantitative determination of water content the void volume of the dry absorbent must be known and assumed constant over the absorbent.

4.0 DISCUSSION OF RESULTS

In Fig. 3(a)-(c), gray scale images are given for a diaper pressed to a 5 mm thickness and injected with water at $0.475 \text{ cm}^3 \text{ min}^{-1}$. In gray scale images the black intensity, proportional to the local water content. In Fig. 3 as the total volume injected increases the intensity and area of the dark x-ray attenuating, water-rich pixels increase from the central injection point. Although there is a gray shadow extending out from the injection point only the central black area contains significant water. The irregular pattern reflects the heterogeneity of the diaper absorbent (it contained a physical pattern in the non-woven mat). The fluid displacement is greater in the down direction indicating a significant gravity effect. The gray scale images give an immediate impression of the water distribution. The pixel X-ray attenuation data can be plotted directly as in Fig. 4 (a)-(b). In this manner, a quantitative picture of the water distribution around the injection point is presented. Please note that in Fig. 4 every third pixel is plotted, thus each grid point is 2.3 apart. At an injection rate of 0.48 cc/min, the water distribution is irregular and the void volume of the absorbent even in the center is not saturated with water. However, at the faster injection rate, a higher fraction of the void spaces are saturated with water near the injection point. In Fig. 4 (a)-(b) the raw data are plotted. The irregular base data outside the water regions is not smoothed. It contains some noise and information about the density distribution for dry fiber within the diaper. In subsequent plots in which Eqn. 1 is used to compute the pixel water fraction, the areas outside the water zones was averaged to eliminate this noise and thus appears flat.

In Figures 5 (a)-(c), gray scale images of water distribution in feminine napkins are given. In contrast to the diaper, the fluid migration is more regular. However, there appear to be distinct bands in the gray scale that map evidence of fluid motion in the different layers of the napkin absorbent. Since each layer has a different density, the capillary forces on the water-air interface are expected to vary from layer to layer. Imbibition will tend to be greatest in the regions with the smallest capillaries. In Fig. 6 (a)-(c), fluid migration into a feminine incontinence pad is observed. Here, the non-woven absorbent is non-cellulosic. In Fig. 5(a) there are evident dark alternate layers far removed from the injection point. These represent concentrations of

dry superabsorbent polymer that is concentrated in the folds of the absorbent mat. Instead of spreading uniformly into the absorbent, fluid absorption follows the zones of superabsorbent. The absorbent matrix swells and inhibits the dispersion of the fluid into the bulk of the absorbent pad. In Fig.7(a)-(b), water fraction distributions are given both for the napkin and pad. Clearly the napkin disperses the fluid almost isotropically while the flow in the pad appears localized in the plane of the superabsorbent. The water concentrations in the pad containing superabsorbent is more concentrated.

A tampon was mounted in the the assembly described in Fig. 2 and fluid was injected into to its tip at 0.3 cc/min. In this case 1 M KI (which has a very high specific X-ray attenuation) was injected into the tampon tip. The tampon contains superabsorbent and when dry is highly compressed. In Fig. 8(a), the EMI number (a medical relative attenuation scale defined as $500 (\mu_{ij} - \mu_{\text{water}}) / \mu_{\text{water}}$) is plotted for each pixel along the centerline of the tampon. Two processes are evident, the initial swelling in the region around the tip followed by the displacement of fluid along the stem of the tampon. In Fig. 8(b)-(d) gray scale images of the same experiment are given. The ridges in the dry tampon reflect the distribution of cellulose in the compressed dry tampon. Dark regions indicate the spread of water. Because of the superabsorbent present, high water saturations are found and the water propagates almost like plug flow.

It is very difficult to account for the physics of the displacement processes in the preceeding market place absorbents. The non-wovens are complex, containing regions of different density and pattern. In addition, they appear to be layered. Superabsorbent polymer dispersed in the absorbent actively immobilizes fluid and inhibits or alters the dispersion. Although difficult to quantify, without doubt this CT imaging technique yields without question the most complete picture of fluid displacement heretofore observed in non-woven absorbents. Most absorbent materials are hydrophilic and spontaneous imbibition of fluid due to capillarity is expected. However, the experiments reported herein have water injection rates that are constant.

In order to simplify the analysis, a simpler system was sought. A number of sheets of Whatman Number 2 filter paper (22 sheets) were stacked together and assembled in the apparatus described in Fig. 2. This was assumed to form a relatively homogeneous porous structure upon which a more fundamental analysis of the displacement process could be made. In Fig. 9 (a)-(c), gray scale images of the different stages of water injection into this filter paper mat are shown. Because of the regular character to the absorbent matrix, the fluid motion is radially uniform, describing almost a perfect circle. No gravity effect is observed because of the strength of capillary forces. In Fig 7(c) the water fraction has been plotted as a function of position. Though the spatial distribution of water is radially symmetric there is a regular decrease in the water fraction with radial distance from the injection point. In a series of experiments only a central traverse of pixels is considered to represent the total distribution. From a central traverse of Fig. 7(c) and that from approximately 22 scan taken in sequence during a fluid displacement process, the fluid migration can be

quantitatively tracked as it moves into the absorbent. Such is illustrated in Fig. 10(b) for a water injection rate of 0.216 cc/min.

It is not the purpose of this report to explain all of the observed phenomena. However, the spatial distributions of water in Fig. 10(a-c) are very rich in experimental information. At very high injection rates, (Fig. 10(a)), viscous forces predominate and capillary effects are not readily observed in the data. The void volume is almost totally filled with water. The leading air-water interface propagates from the injection point with a square root dependence on time (as it should in plug radial flow). At moderate injection rates, Fig. 10(b), the injection zone is saturated with water. However the average fluid velocity decreases with radial distance and as a result viscous forces decrease. It is observed that not all of the void spaces in the absorbent are saturated with water. Significant trapped air is observed. This results from the capillary driven imbibition flow which tends to exceed the fixed injection rate. As a result the regions containing the smallest capillaries control the water flow and the regions with large pores (small capillary forces) cannot retain water. The leading interface even here however follows the square root of time dependence on position. At the very smallest injection rates, Fig. 10(c), fluid distribution within the porous absorbent is totally controlled by capillarity. Void spaces containing the largest capillaries cannot hold the water as it is drawn off by the small capillaries. More comprehensive analysis of this data will be given elsewhere. Since there exists a distribution of pore size (or capillary forces) and since only a fraction of the void spaces are ultimately filled, simple percolation or simple dispersion models of this behavior cannot be applied. It is interesting to note however that because the air-water interface is hydrodynamically stable, the leading edge of the water front in all cases shows very little dispersion and is almost planar. One would expect enhanced dispersion if the viscosities were reversed as is commonly found in an oil displacement experiment.

5.0 CONCLUSIONS

Computed tomography proves to be a quantitative and nondestructive technique for tracking the migration of water in porous absorbents.

6.0 ACKNOWLEDGMENT

The authors thank the General Electric Company for their generous support of engineering research by computed tomography at Columbia University and Alonso J. Wilson for his technical advice and help.

7.0 BIBLIOGRAPHY

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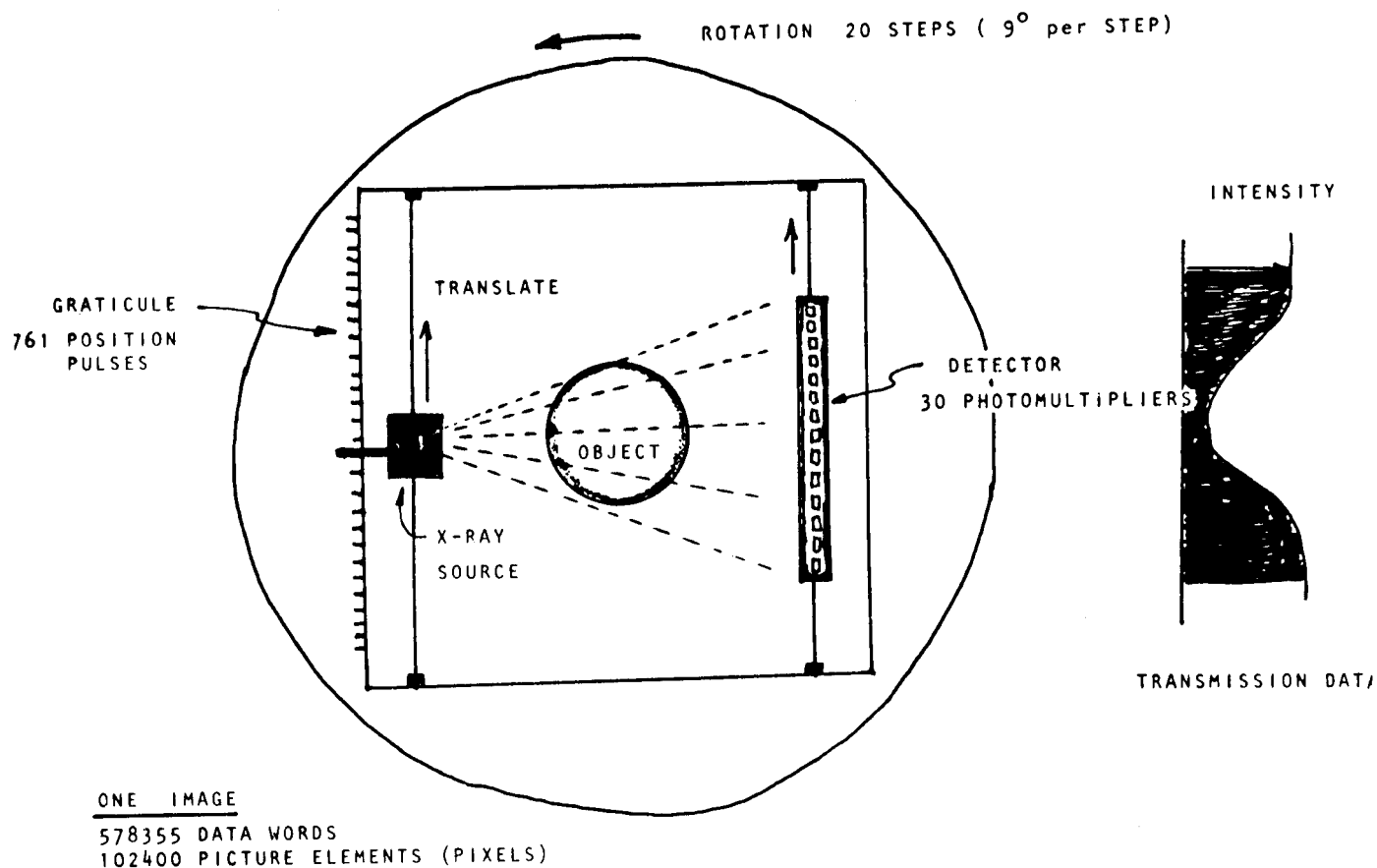


FIGURE 1 Scan Geometry Schematic for the EMI 5005 CT Scanner.

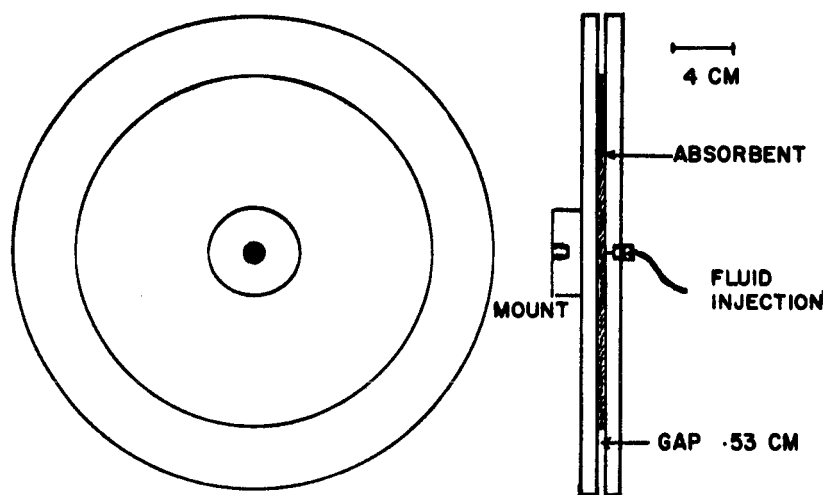
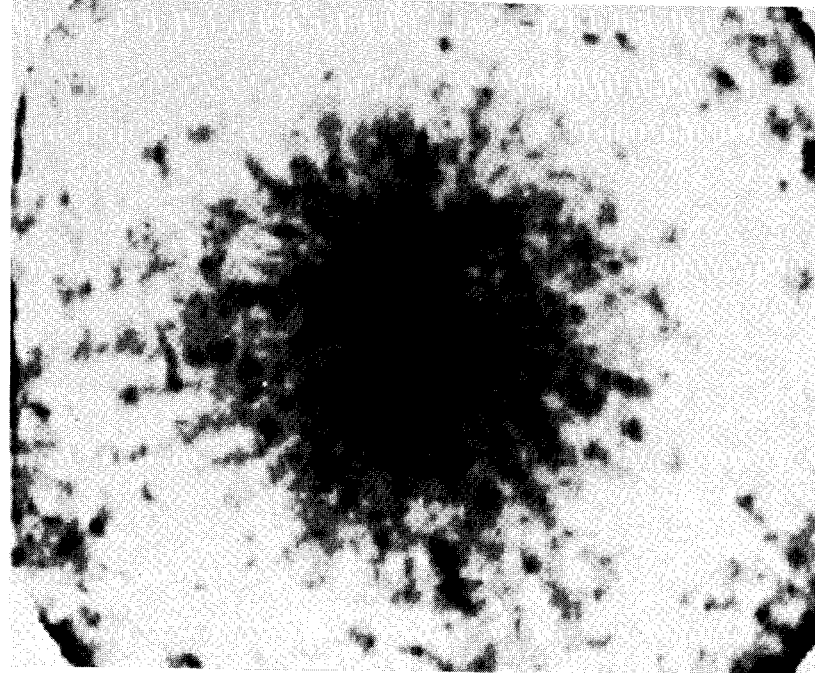
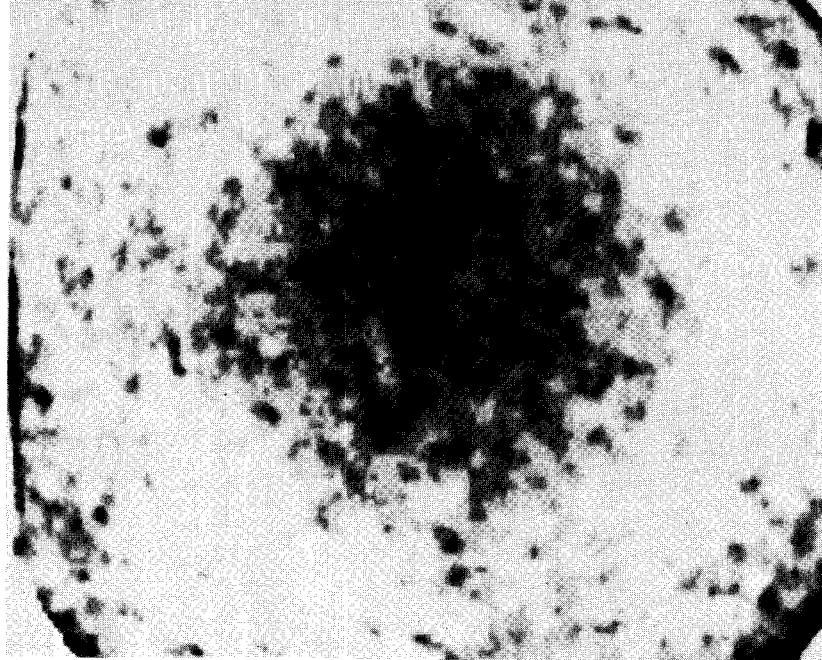


FIGURE 2 Absorbent Mounting Frame (dimensions in cm., material, poly(methylmethacrylate)).



(b).

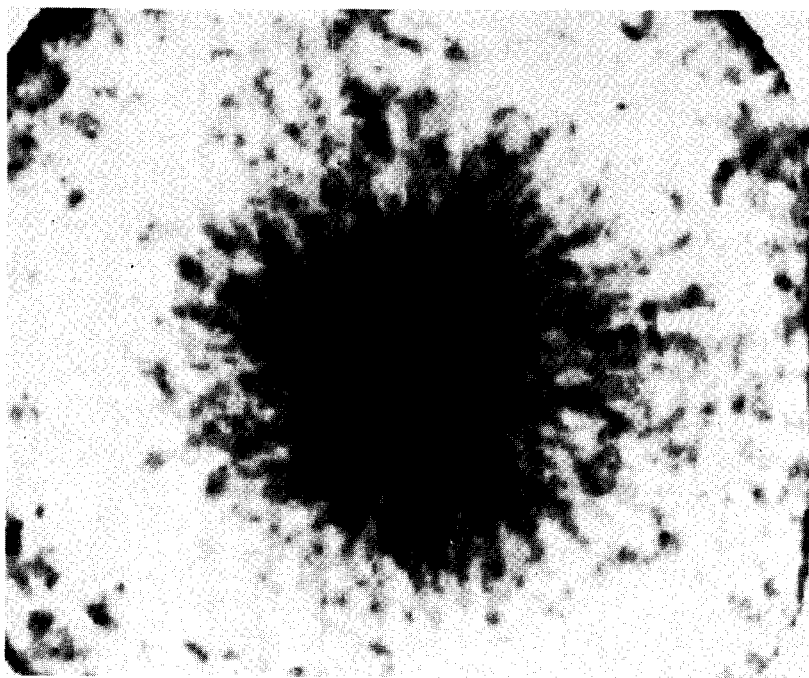


FIGURE 3

Water Injection into a
Diaper at 0.475 cc/min
(water-rich is dark);
volume injected-

(a). 0.28 cc

(b). 2.38 cc

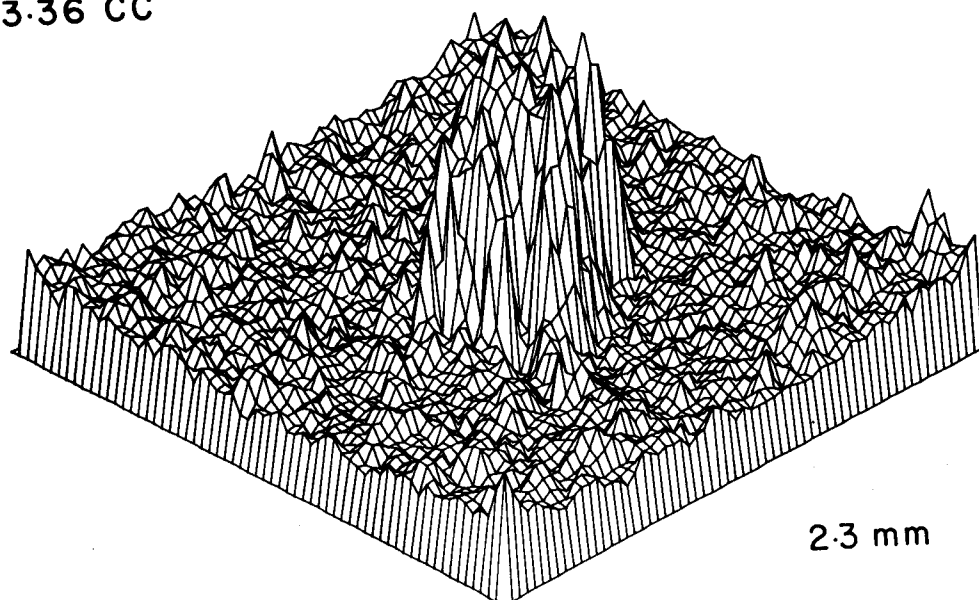
(c). 5.82 cc

(arrow- direction vertical,
length 2 cm)



2.95 CC/MIN

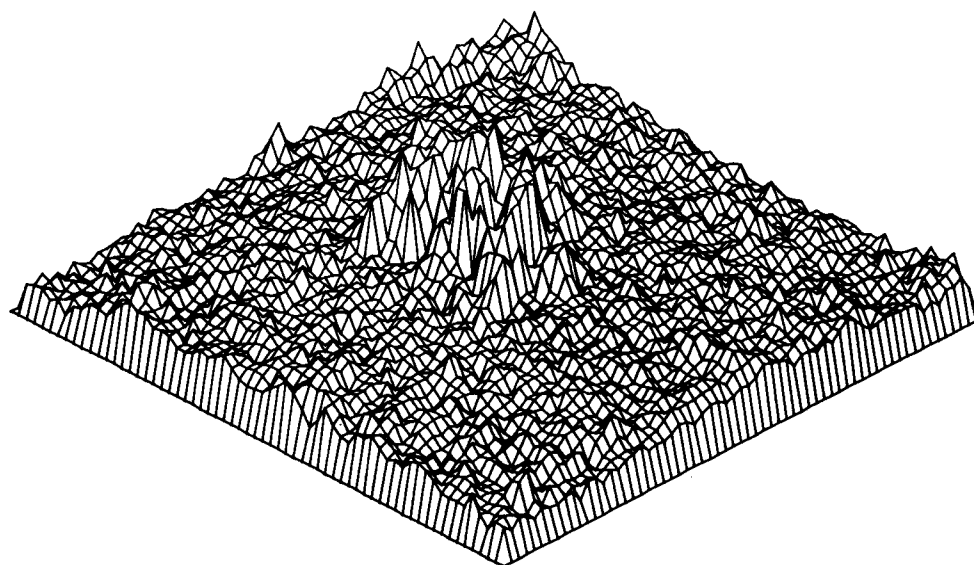
3.36 CC



(a).

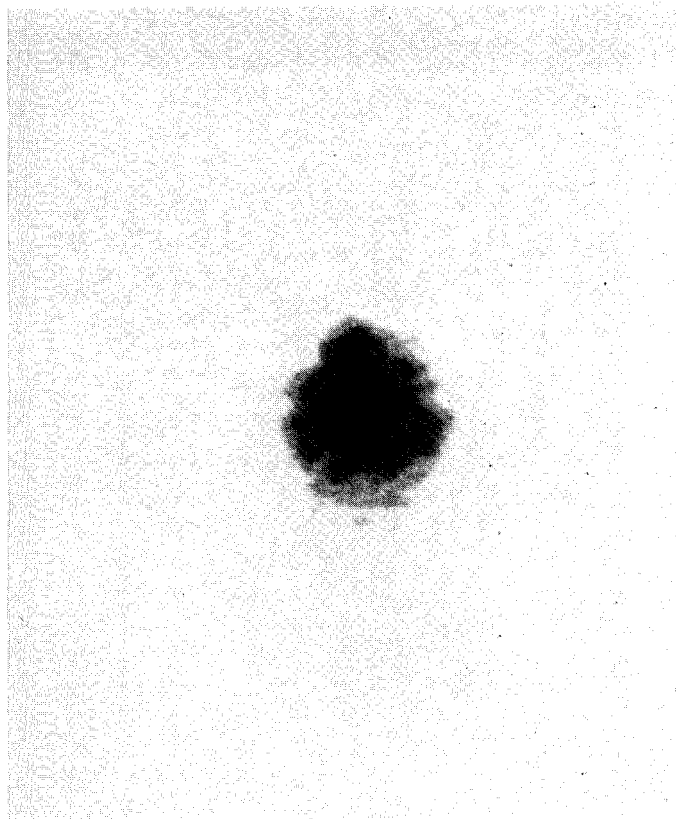
0.48 CC/MIN

1.66 CC

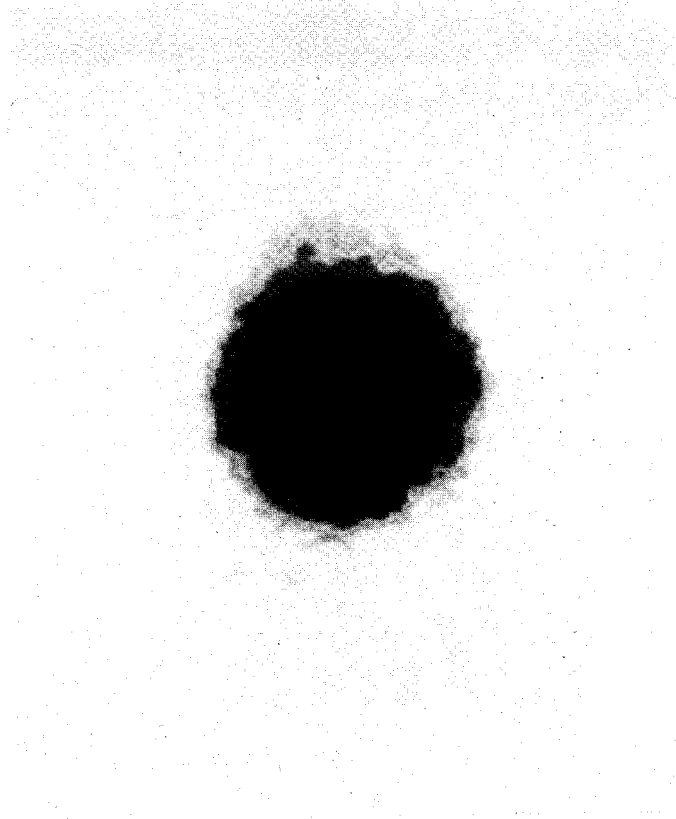


(b).

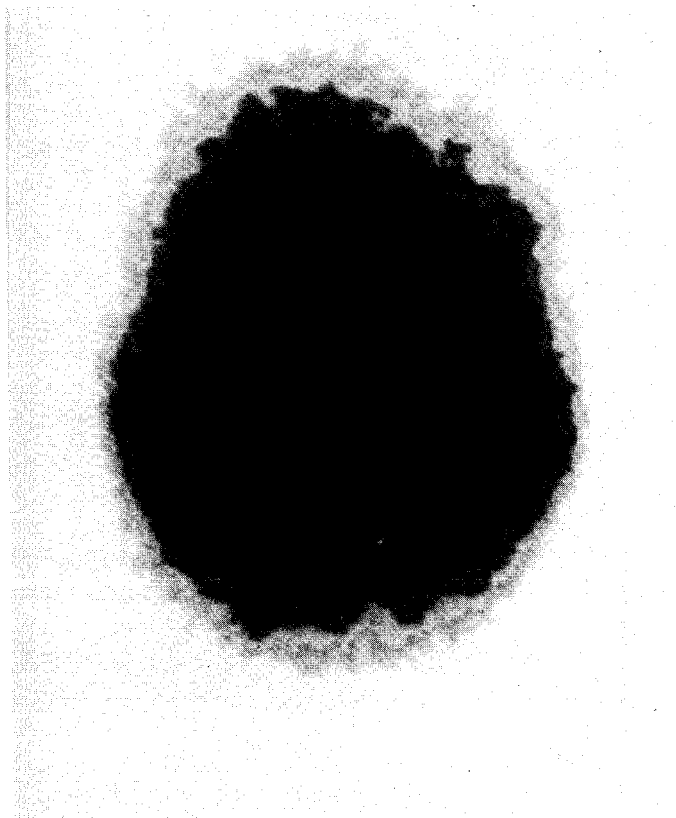
FIGURE 4. CT X-ray Attenuation Data for Every Third Pixel over Area Scanned. Absorbent Diaper, Injection Rate and Injection Volume Given.



(a).



(b).



(c).

FIGURE 5

Water Injection into a Feminine
Napkin at 0.44 cc/min.
(water rich areas are dark)
Volume Injected:

- (a). 0.44 cc
- (b). 3.08 cc
- (c). 6.60 cc

(arrow- direction vertical,
length 2 cm)



(a).



(b)



(c).

FIGURE 6.

Water Injection into a Feminine Incontinence Pad at 0.44 cc/min. (water-rich area is dark)

Volume Injected:

(a). 3.08 cc

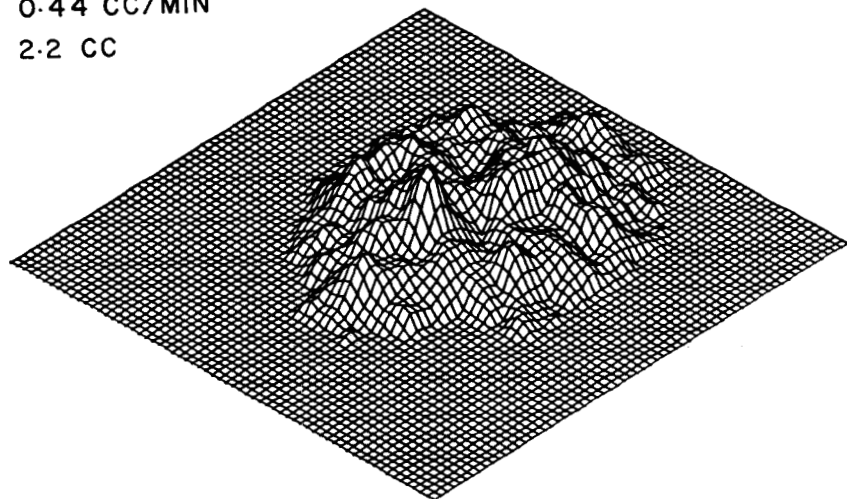
(b). 6.60 cc

(c). 10.12 cc

(arrow- direction vertical, length 2 cm)



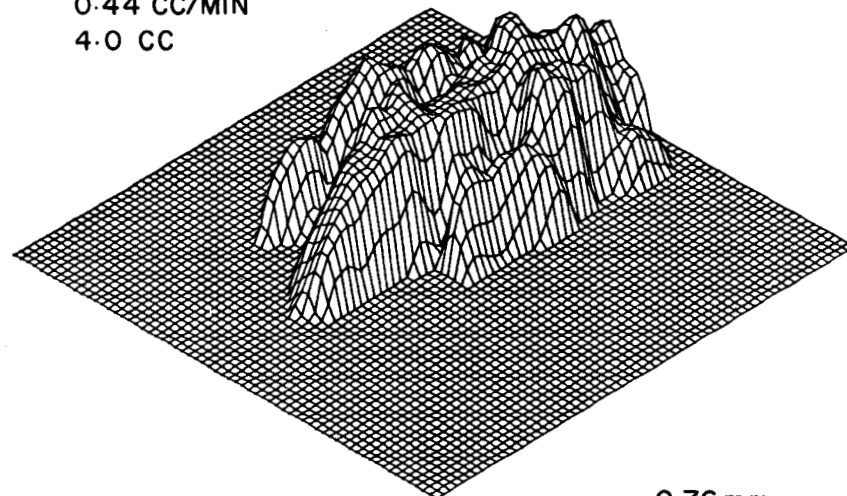
0.44 CC/MIN
2.2 CC



0.76 mm

(a).

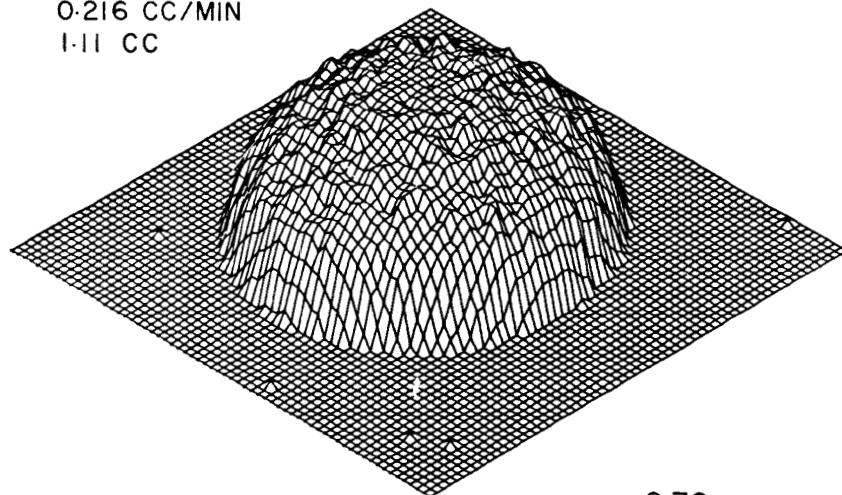
0.44 CC/MIN
4.0 CC



0.76 mm

(b).

0.216 CC/MIN
1.11 CC



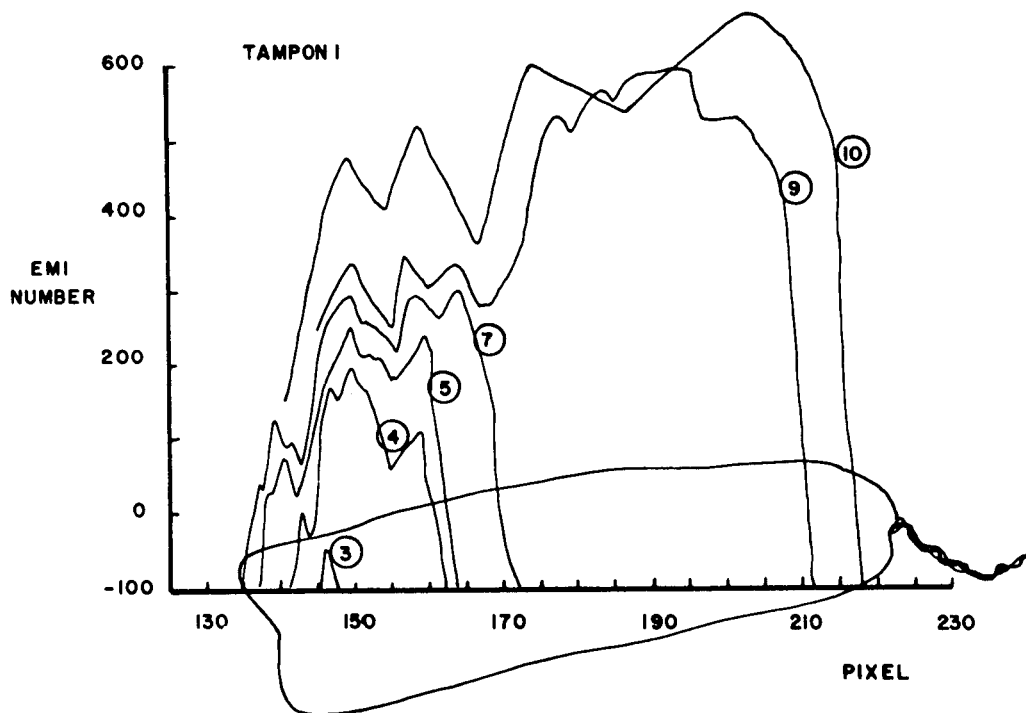
0.76 mm

(c).

FIGURE 7

Water Fraction Computed from Pixel
Attenuation at Indicated Water
Injection Rate and Injection Volume.
(grid spacing 0.76 mm)

- (a). Feminine Napkin
- (b). Incontinence Pad
- (c). Filter Paper Mat



(a).



(b)

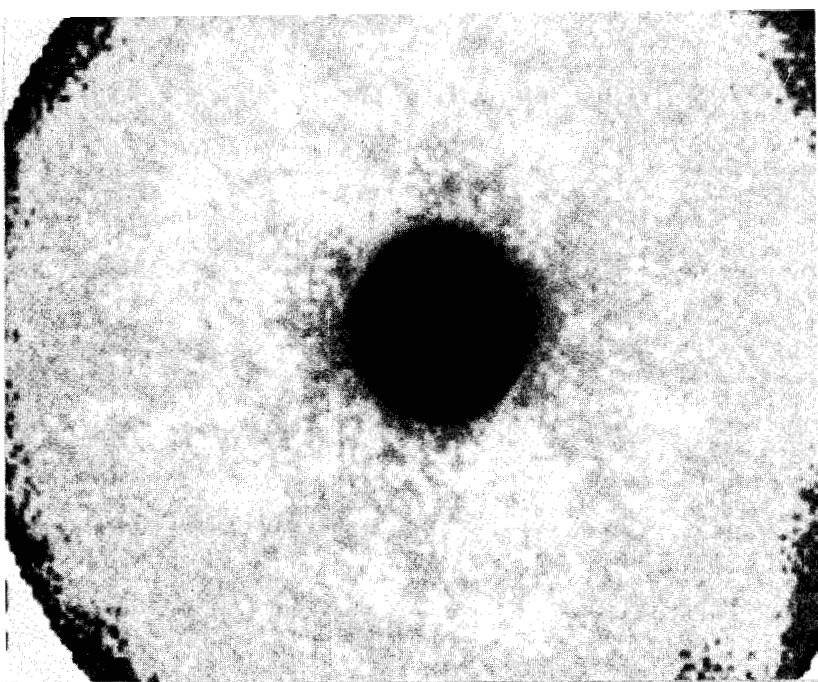


(c).

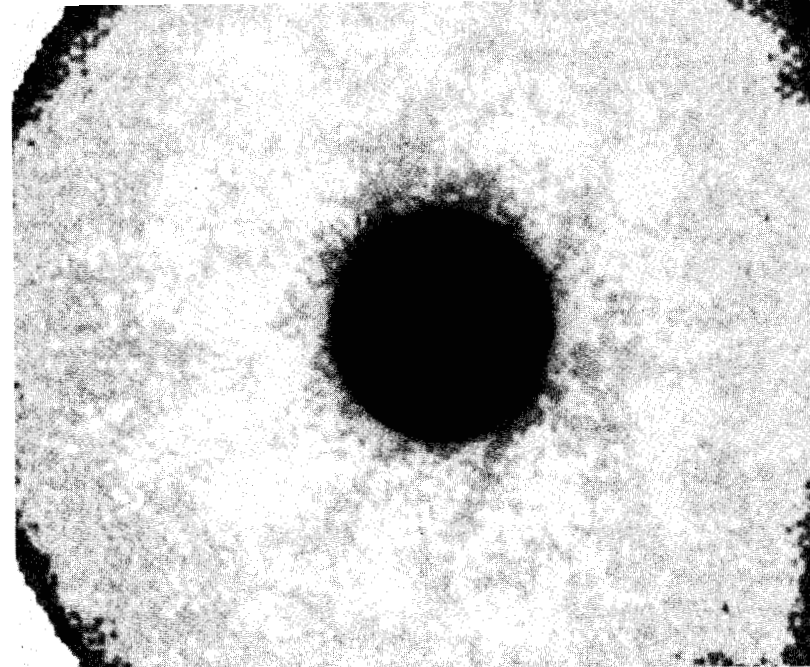


(d).

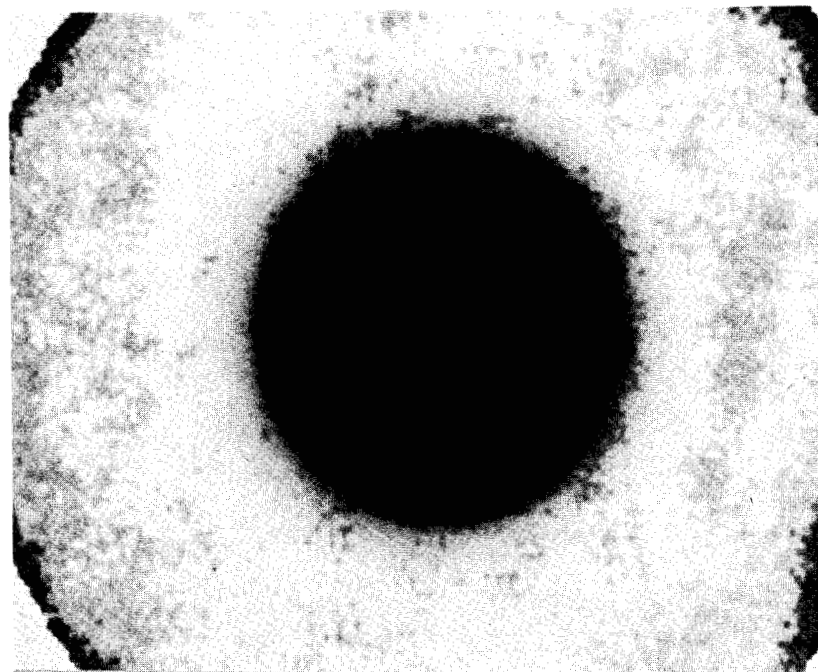
FIGURE 8. Injection of 1 Molar Potassium Iodide into a Tampon at 0.3 cc/min. (a). Trace of EMI Attenuation Number along Tampon Centerline as a Function of Volume Injected: 3 - 0.9 cc; 4 - 1.5 cc; 5 - 2.4 cc; 7 - 4.3 cc; 9 - 14.1 cc; 10 - 19.5 cc. (b) volume 0.9 cc; (c) volume 1.5 cc; (d) volume 4.8 cc.



(a).



(b).

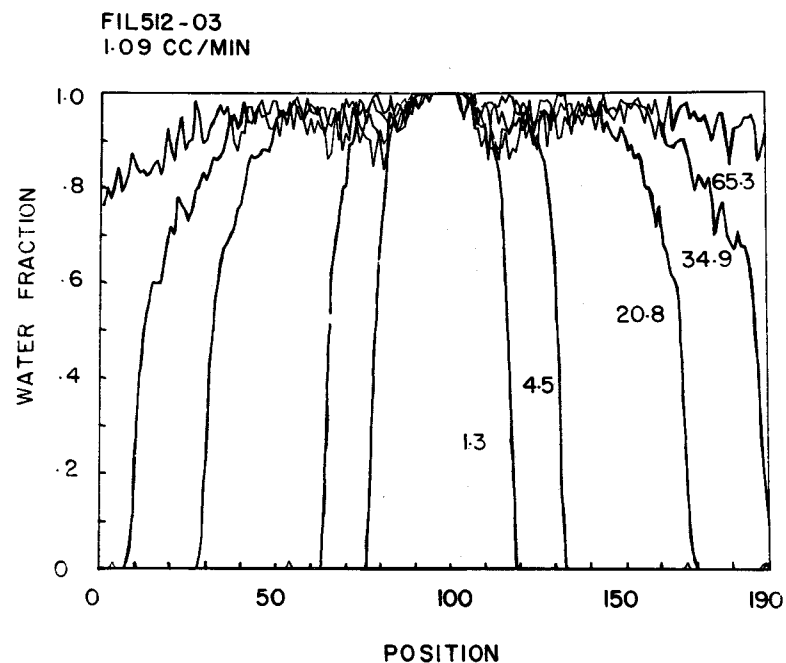


(c).

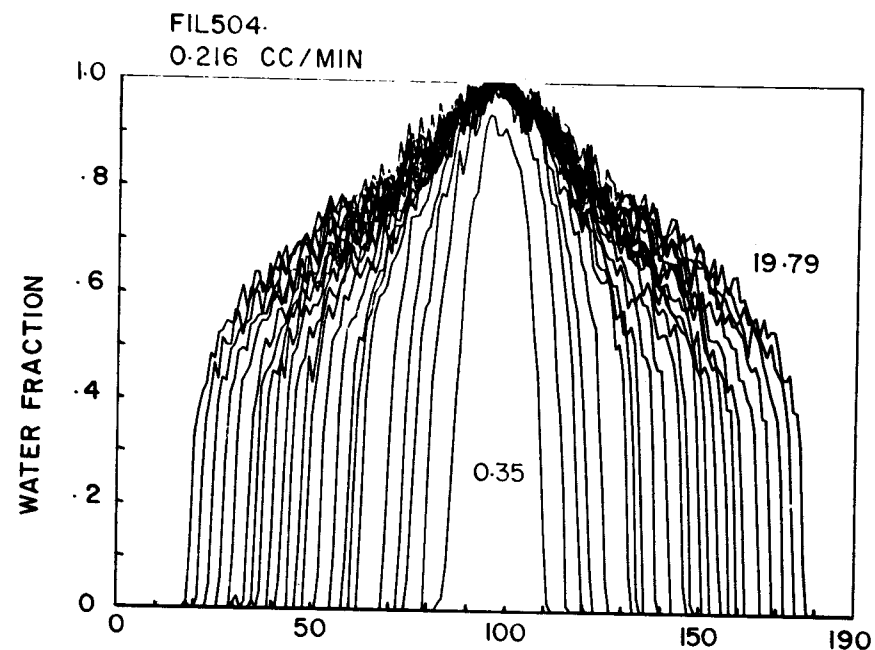
FIGURE 9

Gray Scale Image of Water Injected into
22 sheet Stack of Whatman Filter Paper
at 1.09 cc/min; volume injected:

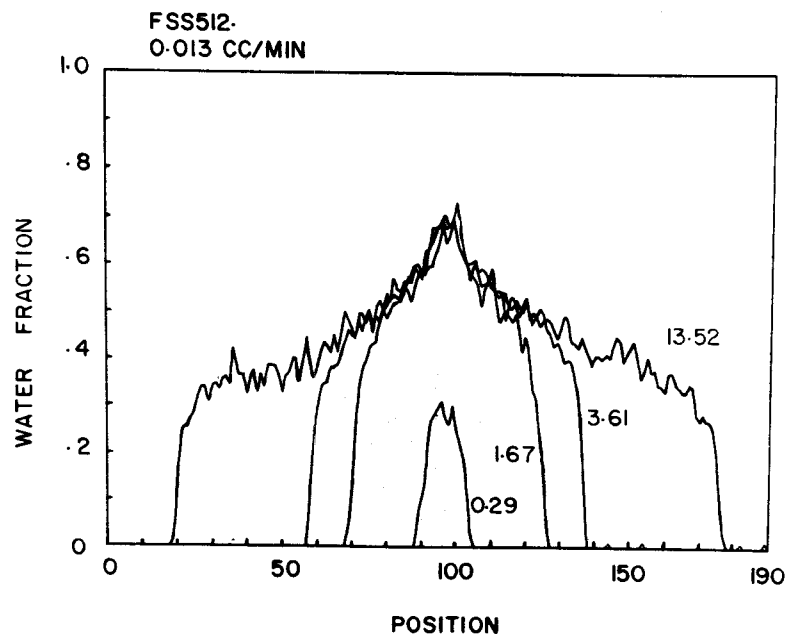
- (a) 2.8 cc
- (b) 4.5 cc
- (c) 14.3 cc



(a).



(b).



(c).

FIGURE 10

Water Fraction for Pixels along Diameter as a function of indicated water injection rates for Whatman Filter Paper. (numbers on figures indicate volume of water injected).

IMPROVED METHOD FOR MONITORING LIQUID ENTRY AND BREAKTHROUGH UNDER HYDROSTATIC PRESSURES

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ABSTRACT

A new test procedure is detailed for evaluating the resistance of a fibrous material to the forced passage of a liquid. The driving pressure can be controlled at a constant value or else at a known rate of increase. If the material has any appreciable resistance to the liquid, the volume transport can be monitored continuously with sufficient sensitivity so that the rate of penetration and the total uptake can be determined at any instant after first contact. The moment of breakthrough can be identified easily and automatically, so that breakthrough time (at constant pressure) or critical breakthrough pressure can be determined in a routine manner.

The method is based on simple fundamental principles and requires only conventional laboratory instrumentation. It has been found to be a distinct improvement over the standard methods currently recommended for hydrostatic pressure testing.

Background

There are a variety of applications for fabrics and other fibrous or porous materials that require resistance to liquid breakthrough. In some cases this is accomplished by using inherently resistant fibers, that is, those that form an advancing contact angle greater than 90° with the liquid. Alternatively, fibers can be surface-modified by coating or chemical reaction to produce the required reduction in wettability. If resistance to breakthrough is needed only for a limited time, a porous barrier may serve the purpose even if its surfaces do not have contact angles greater than 90° . Since the volume rate of flow into a capillary network, which we term penetration, depends on the wettability function $\gamma \cos \theta_a$ and on the ⁵4th power of the capillary radius, a material with very small pores and a high contact angle (e.g., $>70^\circ$) may be able to inhibit liquid penetration to such a degree that it will be an adequate barrier for certain end-uses. However, it should be kept in mind that breakthrough, in contrast to penetration, does not occur as the result of capillary forces – a porous network that takes up liquid spontaneously will not release it out the other side unless there is some external driving pressure. This pressure is usually produced by a hydrostatic head of the liquid, though other sources of driving force are possible. Every nonwetting ($\theta > 90^\circ$) porous barrier-liquid combination should have a critical breakthrough pressure defined as the pressure just high enough to produce breakthrough. Materials that are only slightly wettable may also exhibit an apparent critical breakthrough pressure, if the time scale of the test is not too long.

The determination of a critical breakthrough threshold is considered an important requirement for ranking fabrics as barriers to the passage of liquids. Two types of test procedures have been recommended as standards. AATCC Method 127-1980 is typical of the dynamic hydrostatic head method. The fabric

specimen is placed as a "seal" at the bottom of a chamber and water is added to the chamber so as to increase the head at a rate of one centimeter per second. An operator, with the aid of a mirror, observes the bottom face of the specimen and determines the moment when liquid has broken through at three different places. The head at that moment is recorded as the critical breakthrough pressure.

An example of a static head test is INDA Standard Test 80.7-70 (R82) for saline repellency of nonwovens. This procedure uses a Mason jar with a hole drilled in its bottom. With the hole closed temporarily, a fixed amount of solution is added to the jar. The test fabric is placed over the mouth of the jar and sealed in with a gasket and a threaded cover ring. The jar is then inverted and the operator times how long it takes for the first indication of breakthrough (called "penetration" in the written procedure, which is misleading, unfortunately).

Both the above types of test have been judged unsatisfactory and inadequate for several reasons and are used with considerable reluctance by most laboratories concerned with breakthrough phenomena. From the practical point of view, the need to have an operator watch the down face of the fabric to detect the moment of breakthrough is clumsy and imprecise. The endpoint involving three different locations in the AATCC test will not be clearly identifiable in many instances. The Mason jar test, in addition to being time-consuming, requires an arbitrary choice of a single pressure head. This can mean that a fabric judged to be completely resistant by this test could be totally nonresistant under a slightly greater pressure head.

A more fundamental objection to both types of test is that they focus only on the moment of breakthrough. Therefore, it is possible for a fabric that takes up liquid until it is saturated before showing breakthrough and one that is nonwetting but has a pin-hole defect to be judged equivalent because they test as having the same breakthrough pressure.

This paper presents a new method for monitoring both penetration and breakthrough that eliminates the aforementioned practical and technical objections. It permits automatic recording of the necessary data and is based on a simple principle that has been accepted for over two thousand years.

Measurement Principle

The measurement depends on the application of the classic Archimedes buoyancy force principle: "A body submerged wholly or partially in a fluid is buoyed up by a force equal to the weight of fluid displaced by the body." In the context of liquid-fabric penetration and breakthrough processes, this means that if one side of a fabric is placed in contact with a liquid in a reservoir, the buoyancy force will decrease systematically as liquid displaces air within the fabric pores. If the liquid has a density of 1.0 g/ml (e.g., water), this means that the displacement of one microliter of air by liquid will produce a change in buoyancy force of one milligram. Therefore, one can follow very small amounts of liquid intrusion using a buoyancy force detector that reads in the milligram range.

Because most reference sources describe the buoyancy force concept in terms of an upward force on a partially submerged object, it is not commonly realized that there will be an equal and opposite force exerted by the object. If the liquid container is mounted on a top-loading balance, this force will be recorded by the balance (see B. Miller, Buoyancy Measurements for Teaching and Research, J. Chem. Education, in press).

Experimental Details

The test chamber, associated plumbing, and detectors used for the measurement are shown in Figure 1. The brass specimen holder has an outside diameter of 7 cm and a height of 4 cm. It is held in place by a sleeve clamp that attaches its

brass air passage tube to the plastic cover. The fabric specimen sits on a rigid perforated plate that keeps it from being deformed by the applied pressure. The fabric is sealed in by the combination of an O-ring, a compression ring that does not turn, and a threaded sealing ring. The underside of the compression ring is beveled outward slightly so that the O-ring, when compressed, does not exert an inward buckling force on the fabric. The pressure below the fabric is reduced and controlled by connecting that part of the test chamber to a previously evacuated reservoir, as shown.

Enough water is placed in the lightweight container on the balance pan so that it will cover the test chamber when the latter is put in position. This provides a starting pressure head of about 3 cm. If the fabric resists penetration under such conditions, the balance reading remains constant at a value that represents the combined effects of the weight of water plus its container and the buoyancy forces produced by the submerged test chamber. When the connecting valve to the evacuated reservoir is opened, the pressure below the fabric drops at a rate controlled by the setting of the needle valve. Because the reservoir volume is much larger than the chamber, this rate will be constant for most cases. The pressure change is detected by the pressure transducer and transmitted to one channel of the recorder. As liquid penetrates into the fabric, the balance reading drops according to how much air has been displaced by liquid. The fabric specimen has an exposed surface area of 7 cm^2 . If it had a nominal thickness of 0.05 cm, this would give a volume of 0.35 ml, which means that, with a typical woven fabric having a porosity of about 0.8, 0.28 ml is void volume. If this space were completely occupied by water, the buoyancy force change would be 280 mg. For thicker or less dense fabrics the change would be proportionately greater. When breakthrough occurs, the rate of change of the balance reading (transmitted to the second recorder channel) increases noticeably, and the critical breakthrough pressure can be found from the simultaneous plots of the two channels of the recorder.

Figure 2 is a representation of the way the recorded data may appear when a fabric takes up significant liquid before breakthrough. Up to time t_1 , the pressure on the fabric was only 3 cm (H_2O) and the balance remained constant because (in this case) no liquid entered the porous network. At t_1 , the pressure below the fabric started to drop and liquid began to intrude. At t_b , breakthrough occurred at a pressure gradient of $\Delta P_b + 3$ cm. The amount of liquid that entered the fabric structure before breakthrough is ΔW_a .

Alternatively, the experiment can be carried out on a time basis at constant pressure, which can be as low as the initial head (3 cm), as long as the influx of liquid does not start immediately, that is, before the balance reading becomes stabilized after the test chamber is immersed. This usually takes about five seconds, so that only very rapidly absorbing materials are excluded.

Examples of Results

Two polytetrafluoroethylene/polypropylene laminate materials (Goretex[®]) were examined using the buoyancy change technique. These materials were described by the manufacturer's brochure as having pore sizes of 3 and 5 μm , respectively; however, it was not clear whether these dimensions were radii or diameters, and whether the designations were meant to represent maximum or mean pore dimensions. The results of our breakthrough tests are shown in Table I, along with "minimum water entry pressure" values given in the brochure.

TABLE I
BREAKTHROUGH DATA: GORETEX[®] LAMINATES

<u>"Pore size"</u>	<u>Buoyancy monitor*</u>		<u>Entry pressure, cm (H_2O)</u> <u>(Goretex specification)</u>
	<u>ΔP_B, cm (H_2O)</u>	<u>ΔW_a, mg</u>	
3 μm	91.0	235	140
5 μm	31.3	160	35

*Pressure increase rate = 0.4 cm (H_2O)/s.

A nonwoven polyester/rayon surgical drape fabric which had been given a chemical treatment to make it resistant to liquid breakthrough was tested by the buoyancy method and by a semistatic conventional hydrostatic head test in which the pressure above the material was increased in steps of 5 cm until breakthrough was observed. The two faces of this material were different in appearance and surface characteristics. The results (Table II) show that breakthrough pressure was dependent on which side of the material was in contact with the liquid.

TABLE II
BREAKTHROUGH DATA FOR VARIOUS MATERIALS

<u>Material</u>	<u>Buoyancy Monitor*</u>		<u>Hydrostatic ΔP_B, cm (H₂O)</u>
	<u>ΔP_B, cm (H₂O)</u>	<u>ΔW_a, mg</u>	
Surgical drape			
Contact face-blue	138, 131	240, 250	80
" " -white	147, 154	260, 210	105
Manila paperboard	137, 131, 136	225, 290, 240	--
Stormshed [®] fabric			
Contact face-F	57.3	845	50
Contact face-S	92.5	845	50

*Pressure increase rate = 0.4 cm (H₂O)/s.

Table II includes results for a coated paperboard (Manila) and for a water-proofed polyester/cotton fabric (Stormshed[®]) that had been treated differently on each side. One side (labeled S) was treated with a silicone finish, the other side (labeled F) with a fluorocarbon finish. Both surfaces were hydrophobic, but a drop of aqueous surfactant solution ($\gamma = 30$ mN/m) placed gently on the S face was gradually absorbed, while the same drop on the F side stayed undisturbed on that surface indefinitely. Therefore the F face was considered to be less wettable. The TRI experiment also showed breakthrough pressures that were dependent on which side faced the liquid.

All of the above data were obtained at the same rate of pressure increase (0.4 cm/s). When the Stormshed fabric was run at a much slower rate (0.06 cm/s), breakthrough pressures obtained in this way were lower, as shown in Table III.

TABLE III
EFFECT OF RATE OF PRESSURE INCREASE ON
BREAKTHROUGH PRESSURE

Pressure Increase rate, cm/s →	ΔP_B , cm (H ₂ O)	
	<u>0.4</u>	<u>0.06</u>
Stormshed (F-face)	57.3	51.8
Stormshed (S-face)	92.5	70.3

An example of results for a breakthrough time experiment at constant pressure is shown in Figure 3. One, two and three layer barriers of a polypropylene fabric were tested with water at 3 cm hydrostatic head. The amount of water taken up by the material before breakthrough (ΔV_a) was directly proportional to the number of layers. However, the breakthrough time increases at an increasing rate as the number of layers increased.

Conclusions

The buoyancy monitoring technique is a sensitive and convenient method for following penetration and breakthrough with liquid-resistant materials. Its automatic readout feature alone makes it preferable to the conventional direct observation methods that have been used. Based on the limited results shown in Table III, ΔP_B can increase with the pressure increase rate. This is consistent with the fact that the dynamic experiment always gives a higher breakthrough pressure than the semistatic water column method. The reproducibility of the

method is very good when the material being tested is uniform (e.g., the Manila paperboard); when the sample is grossly nonuniform, the replicate data will indicate it.

If the two faces of a fabric are not the same, the measured breakthrough pressures can be different, depending on which side faces the liquid. This does not necessarily mean that the contacting surface wettability controls the breakthrough process. One possibility is that asymmetric flow resistance is due to conical shaped pore cross sections such as those depicted in Figure 4. Since resistance to flow is inversely dependent on the radius of the meniscus, it should be more difficult to force liquid through an expanding cone than through one that is contracting. The pore passages through a fabric have an hourglass shape. Therefore, if one side of a fabric has been made less wettable (e.g., the F side of the Stormshed fabric) it should be a more effective barrier when it is facing away from the advancing liquid front.

The results for breakthrough at constant pressure imply that breakthrough time is related to thickness in a nonlinear manner. Since the pressure head for this experiment was low, it may be that the upper layers were closer enough to the surface to make a difference in the actual driving pressure.

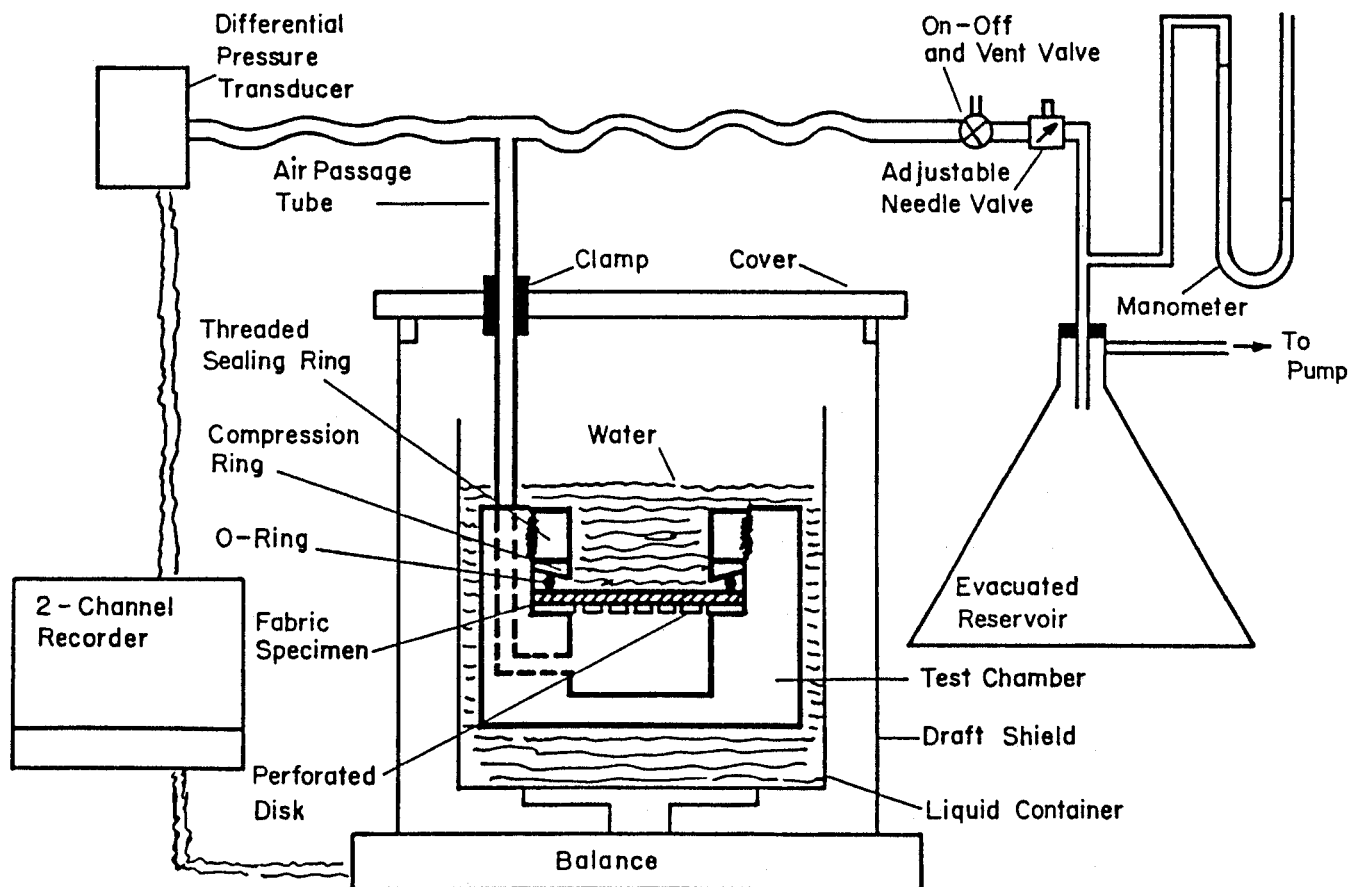


Figure 1. Design of Liquid/Air Displacement Analyzer (LADA).

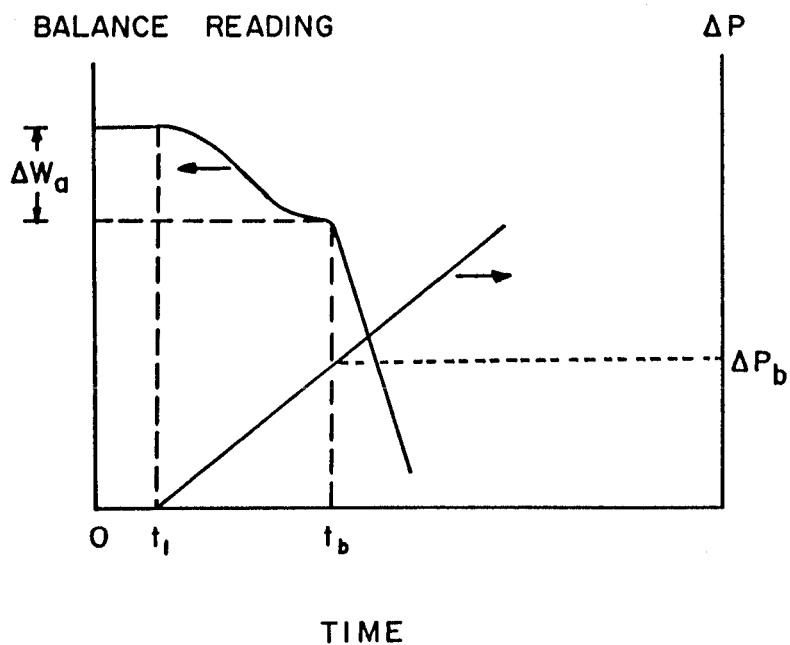


Figure 2. Form of responses recorded during a breakthrough experiment.

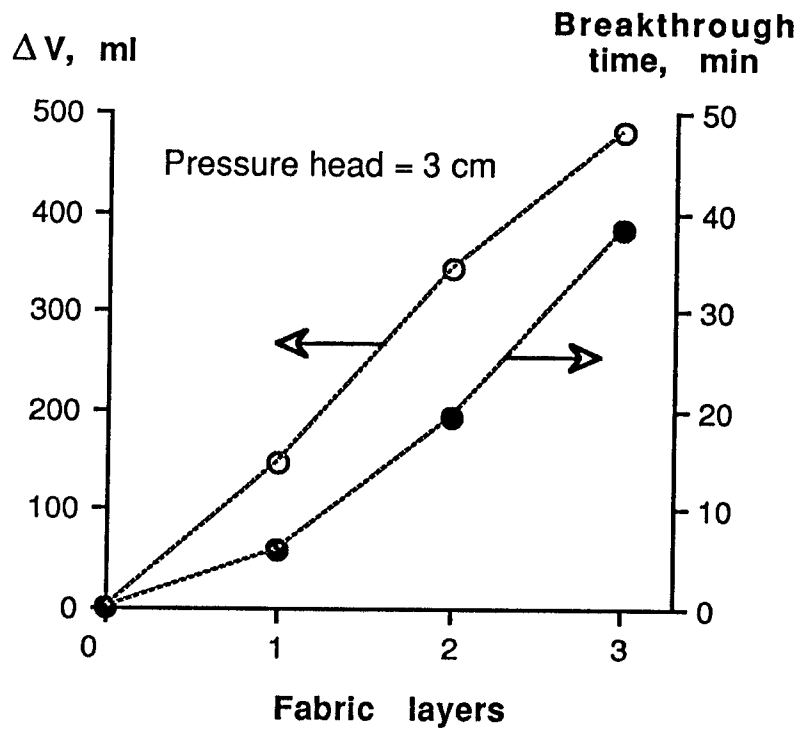


Figure 3. Water penetration and breakthrough for polypropylene fabric layers at a constant pressure head.

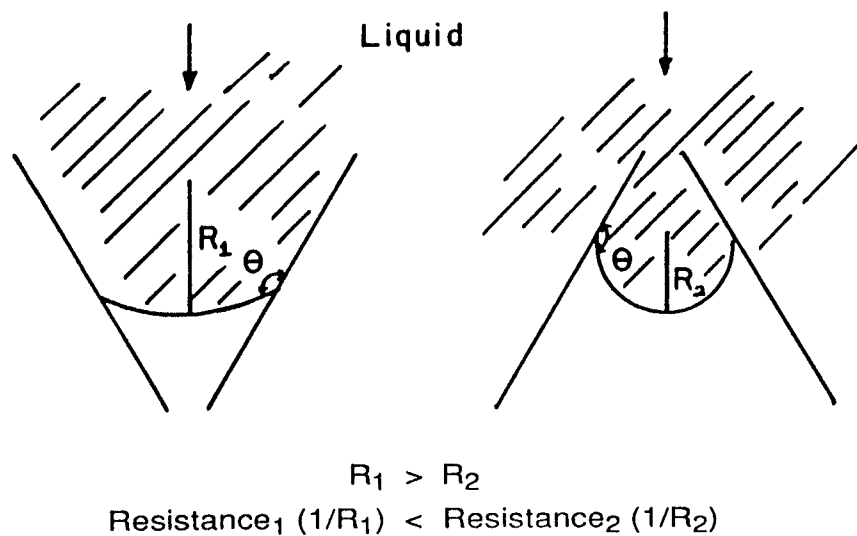


Figure 4. Illustration of how a conical pore can produce different resistances to breakthrough, depending on its orientation. The contact angles are the same, but the menisci have different radii, and resistance is proportional to $1/R$.

Absorbency and Wear Characteristics of Nonwoven and Woven Fabrics Containing Crosslinked Polyethylene Glycols

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Nonwoven substrates, comprising diverse fiber types, were treated with three different molecular weights of polyol/crosslinking agent (DMDHEU) under conditions that insolubilized the polyol on the substrates and resulted in improvement of several functional properties. These included: thermal storage and release over a given temperature range, improvement in flat abrasion and absorption/exsorption of large amounts of water. Initial sorption of water, relative to untreated substrates, was faster for treated hydrophobic surfaces than it was for treated hydrophilic surfaces. Water capacity, regain and imbibition values were appreciably higher for all types of nonwovens containing bound polyols and well within the range of commercial superabsorbent fibers. Wet shrinkage of the treated nonwovens was relatively unaffected in contrast to wovens and knits containing bound polyols.

Previous studies have demonstrated that polyethylene glycols with M_n from 600-20,000 can be bound to a variety of fibrous substrates (cellulosics, wool, synthetic fibers, glass fibers, blends) by crosslinking with polyfunctional methylolamide reagents (1, 2, 3). This bonding has been achieved on all types of fabric/substrate constructions (woven, knit, nonwoven) to impart a variety of functional properties (e.g., thermal storage/release; improved resistance to flex/flat abrasion, static charge, oily soils and pilling; and greater absorbency). A preliminary study (4) utilizing only M_n of 1,000 of the polyol (PEG) with a crosslinking agent (DMDHEU) has also shown that nonwoven substrates containing the bound polyol have desirable thermal, mechanical and water-absorbent properties and experienced little or no wet shrinkage in contrast to wet shrinkage observed with woven fabrics containing the bound polyols.

This investigation evaluates three different types of nonwoven substrates (paper, thermally bonded cotton/polyester and melt-blown polypropylene) and the corresponding woven substrates (cotton, cotton/polyester and polypropylene) treated with three polyol (M_n 600, 1,000 and 1,450)/DMDHEU solutions. Thermal, mechanical and water-absorbent properties of the treated and untreated substrates were determined. Various types of sorbent-related evaluations (wicking, drop absorbency, sorptive capacity, wet shrinkage, regain and imbibition) were conducted to assess the potential of the modified nonwovens for a variety of end uses relevant to marked increases in the retention and sorption of water and moisture.

Experimental

The fabrics or substrates consisted of three pairs (woven and nonwoven) of fiber types or blends: 100% cellulosic (woven cotton printcloth--125 g/m² and a nonwoven polyamide-reinforced paper towel--71 g/m²); 50/50 cotton/polyester (woven cotton/polyester sheeting--139 g/m² and a thermally point bonded nonwoven--160 g/m²); and 100% polypropylene (woven--Testfabrics Style #976--157 g/m² and nonwoven melt-blown--34 g/m²). The thermally-point bonded 50/50 cotton/polyester and melt-blown polypropylene nonwoven fabrics were prepared in the Textile Processing Laboratory at the University of Tennessee, Knoxville. Commercial grade polyethylene glycols (PEG's) of M_n 600, 1,000 and 1,450 were obtained from Union Carbide and used as received. The crosslinking agent was Hylite LF [obtained as a 40% aq. soln. of dimethyloldihydroxyethyleneurea (DMDHEU) from National Starch Co.]. The mixed acid catalyst (a 5/1 molar ratio of $MgCl_2 \cdot 6H_2O$ /citric acid) and the NaCl were both reagent grade (J. T. Baker and Mallinckrodt Co., respectively). Deionized water was used to make up various solutions containing the above polyols, resin, acid catalysts and salt. The following solutions were used to treat fabrics/substrates: 50% aq. PEG-600/10% DMDHEU/3.0% mixed catalyst/0% NaCl and 5% NaCl; 50% aq. PEG-1,000/11% DMDHEU/3.3% mixed catalyst/0% NaCl and 5% NaCl; and 50% aq. PEG-1,450/13% DMDHEU/3.9% mixed catalyst/0% NaCl and 5% NaCl. All fabrics/substrates were immersed in the aqueous PEG solutions containing the above compositions, put through squeeze rolls under 30 lb pressure to obtain pickups ranging from 85 to 375% of dry fabric/substrate weights. All fabrics were then dried for 7 min. at 85°C, cured at 120 and 140°C for 2 min., washed in hot tap water (60°C) containing 1% by weight of a commercially-available phosphate-based detergent, rinsed in tap water at 25°C, then tumble or oven-dried to constant weight. Percent efficiency of polymerization of the polyol on the substrate was calculated by dividing the increase in the conditioned dry weight of the treated material by the theoretical weight possible [(% PEG + % DMDHEU) x increase in wt. after padding] x 100.

A Perkin-Elmer DSC-2 calorimeter equipped with a 3600 Data Base Station was used to determine the melting (T_m) and

crystallization (T_c) temperatures, the corresponding heats of fusion (H_f) and crystallization (H_c) and the total heat content (Q) of untreated and treated woven and nonwoven substrates. Q measurements over a given temperature range are only C_p (specific heat) for untreated surfaces; however, Q for treated surfaces over a range are cumulative values (C_p for the untreated fiber + H_f or H_c of crosslinked polyol bound to modified surfaces).

Mechanical tests conducted were: breaking strength, elongation-at-break and energy-to-rupture by ASTM D1682-64 and bursting strength by ASTM D-3786-80a. Tests related to water sorption and behavior included: drop absorbency by AATCC 79-1986, wicking by a modified capillary action test described by Skinkle (5), absorbency time and absorptive capacity by INDA 10.0-70 (R82), and water of imbibition (4) and moisture regain values at 65% RH/20°C (4); each of the latter two tests were modified by oven-drying 16 hrs. at 110°C for treated surfaces containing crosslinked polyols due to difficulty in removing all water from the hydrophilic fibers. Water-retention value (WRV) or capacity was also measured by a modification of the procedure of Preston and Nimkar (6), in which substrates were wet by immersing them in running tap water at 25°C for 30 min., centrifuging for 5 min. at 1,000 g (equivalent to ca. 3,000 rpm), drying for 1.5 hr. in air at 110°C, conditioning overnight, and calculating the % WRV as the difference of the wet wt. after centrifugation - wt. after oven drying and conditioning/ wt. after oven drying and conditioning. % Wet shrinkage was arbitrarily determined by immersing fabrics or substrates in tap water at 25°C for 15 min., then measuring the dimensions to the nearest 0.1 in.

Results and Discussion

It was known and determined from our previous studies (3, 7) that a very broad range of curing temperatures (100-170°C) could be used to insolubilize polyols on a variety of substrates. However, a better overall balance of properties was achieved with curing temperatures in the range of 120-140°C. It has also recently been determined (8) that the addition of up to 5% NaCl in PEG-1,000-resin solutions improves the efficiency of the polymerization and concomitant insolubilization on fibers, particularly on woven cellulosic fabrics such as cotton. Thus, formulations containing added salt were utilized for both cellulosic and cellulosic blend substrates (both woven and nonwoven) at suitable cure temperatures (120 and 140°C) for all three molecular weights. Solutions without added salt were used to treat both woven and nonwoven polypropylenes, since solutions containing salt were very viscous and did not adequately wet these hydrophobic substrates. Table I shows typical weight gains and efficiencies for the six substrates treated under a variety of conditions. Insolubilization of the polyols on the paper towel was much more efficient when solutions containing salt were employed. Less improvement in efficiency was observed with cellulosic blends and particularly with hydrophobic fibers when

TABLE I
Weight Gains and Efficiencies of Nonwoven and
Woven Substrates Treated with PEG-DMDHEU Solutions ^a

Surface and/or Fiber Type	M _n PEG	%NaCl	Cure Temp. (°)C	% Wet Weight	%Wt. Gain	% Efficiency
Polyamide-reinforced paper towel	600	5	120	157	50.0	53
	1,000	5	120	156	79.0	83
	1,450	5	120	198	127.9	100
Cotton printcloth	600	5	120	97	42.2	72
	1,000	5	120	92	49.0	87
	1,450	5	120	101	61.6	97
Thermal bonded 50/50 cotton/polyester	600	5	120	165	83.9	85
	1,000	5	120	177	98.0	91
	1,450	5	120	190	121.0	100
Woven 50/50 cotton/polyester	600	5	120	87	44.4	85
	1,000	5	120	88	47.5	89
	1,450	5	120	93	60.0	100
Melt-blown polypropylene	600	0	120	233	75.1	54
	1,000	0	120	246	124.4	83
	1,450	0	120	345	206.7	95
Woven polypropylene	600	0	120	103	53.8	87
	1,000	0	120	103	54.3	86
	1,450	0	120	117	67.4	92

^a All fabrics/substrates treated as described in experimental section.

salt was present in the polyol solutions. The PEG-1,450 solutions exhibited very high efficiencies relative to lower molecular weight polyol solutions; this was true for all fiber types. For comparative purposes, the woven and nonwoven substrates chosen for evaluation of their thermal, mechanical and water-related properties were cured at 120°C, with cellulosic and cellulosic fiber blends treated with solutions containing salt and polypropylenes treated with solutions without added salt.

Thermal Properties

The thermal properties of untreated and treated nonwoven substrates are given in Table II. The same type of thermal behavior was generally exhibited with the nonwoven substrates as was observed with woven fabrics: heats of fusion and crystallization and overall heat content usually increase with increasing amounts of and increasing molecular weights of the bound polyol. The T_m of the crosslinked polyols varied somewhat from one fiber type to another, but increased with molecular weight of the bound polyol from an average of about 15°C for the PEG-600 to about 26°C for the PEG-1,450. Corresponding increases in T_c ranged from an average of about -22°C for the PEG-600 to about 2°C for the PEG-1,450. Overall heat contents (Q) on heating and cooling varied from only marginal increases (e.g., PEG-600 with melt-blown polypropylene) to more than double (e.g., paper towels or cotton/polyester nonwovens treated with the PEG-1,450) that of the corresponding untreated substrates in the same temperature range. Figure 1 shows comparable heat contents of untreated and treated paper towels (PEG-1,000), while Figure 2 shows comparable data for untreated and treated cotton/polyester nonwovens (PEG-1,450). In each instance, the heat content of the untreated substrate is linear and due only to the specific heat (C_p), while the heat content of the treated substrate additionally exhibits an endotherm or endotherms (heat absorption) due to the T_m of the bound polyol and possibly also due to evaporative cooling caused by loss of water in the hydrophilic polyol. Further evaluation is needed to determine the role of water in the heat absorption behavior of the treated fabrics.

The mechanical properties of each of the nonwovens containing PEG-600, PEG-1,000 and PEG-1,450 were measured relative to the corresponding untreated materials. It is difficult to note any meaningful trends in most mechanical properties of the treated relative to the untreated substrates. However, the resistance to flat abrasion was improved in treated substrates (compared to untreated), particularly with increasing molecular weight of the polyol and for the cellulosic substrates. The paper towel treated with the PEG-1,000 or PEG-1,450 had almost an order of magnitude improvement in cycles to failure for flat abrasion compared to the untreated paper towel. The thermally bonded cotton/polyester nonwoven had similar (a four to fivefold improvement) flat abrasion characteristics after treatment with

TABLE II

Thermal Properties of Nonwoven Substrates
Containing Crosslinked Polyols (M_n 600-1,450) ^a

Surface and/or Fiber Type ^b	M_n PEG	% Wt. Gain	T_m (C)	H_f (cal/g)	T_c (C)	H_c (cal/g)	Q(heat) (cal/g)	Q(cool) (cal/g)
Polyamide-reinforced paper towel	600	50	19	2.2	-18	2.6	15.9	14.4
	1,000	79	21	3.4	-1	3.6	17.9	18.6
	1,450	128	27	5.9	1	6.3	20.8	20.0
UT control	-----	---	---	---	---	---	12-10	11.6-10
Thermal bonded 50/50 cotton/polyester	600	84	14	3.9	-27	0.8	15.0	14.4
	1,000	98	19	3.9	-15	2.0	16.2	14.0
	1,450	134	29	5.7	-1	6.3	20.7	21.4
UT control	-----	---	---	---	---	---	12.2-10.3	11-9.6
Melt-blown polypropylene	600	75	13	4.8	-22	1.0	17.7	17.7
	1,000	125	27	7.6	-6	5.9	29.3	25.1
	1,450	207	32	9.1	6	8.8	27.0	25.5
UT control	-----	---	---	---	---	---	17.3-15.1	15-13.3

^a All substrates were cured for 2 min. at 120°C and wt. gains given in Table I; ranges for Q (heating/cooling in °C): PEG-600 (-13 to +27/-3 to -43); PEG-1000 (-3 to +37/+27 to -13); PEG-1450 (+7 to +47/+27 to -13). All samples heated/cooled at 20°C/min.

^b UT control is corresponding untreated fabric.

^c Paper towel and cotton/polyester nonwovens all treated with solutions containing 5% NaCl; polypropylene treated with solutions containing no salt.

these two polyols. Breaking strength of each of the treated substrates was generally equal to or greater than that of the corresponding untreated substrate. More random results were obtained for other properties such as elongation, energy-to-rupture and bursting strength. However, these properties were usually better when higher molecular polyols were bound to the nonwovens.

TABLE III

Mechanical Properties of Nonwovens
Containing Crosslinked Polyethylene Glycols ^a

Surface and/or Fiber Type	M _n PEG	Break. ^b Strength (lb)	Elong. at-break %	Energy-to rupture (lb. in.)	Bursting strength (lb/in ²)	Stoll flat abrasion (cycles)
Polyamide-reinforced paper towel	600	4.6	27.7	1.69	6.3	94
	1,000	3.7	8.0	0.73	5.8	334
	1,450	6.6	25.0	2.31	4.7	336
	---	3.2	11.0	1.03	5.8	44
Thermal bonded 50/50 cotton/polyester	600	7.7	9.4	1.57	9.0	857
	1,000	14.7	8.5	2.32	8.8	2414
	1,450	13.3	10.0	2.44	24.8	3088
	---	9.1	19.9	4.76	23.5	597
Melt-blown polypropylene	600	2.9	28.0	1.94	3.5	23
	1,000	2.5	30.5	1.96	3.8	39
	1,450	2.9	32.2	2.25	5.3	44
	---	2.4	34.3	2.37	2.2	14

^a First set for each treated and untreated substrate corresponds to samples and weight gains in Table II.

^b Breaking strength, elongation and energy-to-rupture measured in machine direction; other tests are nondirectional.

The absorbency of water and other liquids even in simple fiber assemblies and structures is quite complex (9); however, there are several types of tests that can assist in evaluation of highly absorbent materials. Table IV shows the behavior of untreated and treated nonwoven and woven substrates with regard to water of imbibition, moisture regain, wicking, drop absorbency and wet shrinkage. As noted in earlier studies (3, 4), drying times of four hours, usually sufficient for obtaining weight of the dry sample for determining water of imbibition and moisture regain for fibers, were not adequate for the highly hydrophilic fibers containing the crosslinked polyols. Figures 3 and 4 illustrate that about 16 hours drying time is the minimum needed to obtain values of moisture regain for substrates containing the crosslinked polyols that are useful for comparing them to untreated substrates.

Moisture regain for the treated substrates (woven and nonwoven) is highest for those containing the crosslinked PEG-

nonwoven) is highest for those containing the crosslinked PEG-1,450. Since weight gains are also higher than those for substrates treated with lower molecular weights, this may be more a function of total add-on rather than any inherent advantage of a particular molecular weight polyol. Increases in the percent moisture regain for treated relative to untreated material were predictably more dramatic for hydrophobic fibers than for hydrophilic fibers. For example, untreated melt-blown polypropylene had a moisture regain value of only 0.4%, while the same nonwoven treated with PEG-1,450/DMDHEU had a value of 57.2%, a 14,200% increase. Substantial, but proportionally less, increases in percent moisture regain were observed for untreated paper towels (8.2% moisture regain for untreated and 59.4% for paper towels treated with PEG-1,450/DMDHEU; this represented an increase of 625%. Similar increases in moisture regain were observed for the other nonwoven and all woven fabrics containing bound polyols.

The same trends observed for the increases in moisture regain of substrates containing the crosslinked polyols were observed for the water of imbibition values. The water is so tenaciously bound that most treated substrates lost less than 6% by weight when they were dried in a vacuum oven (30 mm Hg pressure) for 16 hr. at 60°C. Thus, it was necessary to dry for 16 hr. at higher temperatures to determine imbibed water in these treated samples. Again, the hydrophobic substrates (such as melt-blown polypropylene) exhibited the greatest increase in water of imbibition relative to the untreated control (thirty to thirty-five times). All nonwovens treated with the PEG-1,450/DMDHEU had imbibition values (ca. 200-400%) equal to or greater than that of commercially available superabsorbent fibers (10). Although woven fabrics containing the polyols had somewhat lower imbibition values than the nonwovens, the woven polypropylene had 161% water of imbibition, a value 73 times greater than that of the corresponding untreated fabric.

Wicking and drop absorbency characteristics of treated fabrics and substrates (compared to untreated controls) were primarily dependent on fiber type. Unmodified hydrophobic fibers (both woven and nonwoven polypropylenes) do not wick at all and have a contact angle of 180° with water. However, due to the presence of the very hydrophilic crosslinked polyol on these types of fibers, dramatic improvements in drop absorbency and wicking were observed (Table IV). In contrast, untreated hydrophilic fibers (such as the paper towel, cotton printcloth and thermally bonded cotton/polyester nonwoven) wick rapidly and have drop absorbency times of 1-5 seconds. When these substrates contain the crosslinked polyol, the high degree of swelling of the polyol (and possibly greater swelling of the hydrophilic fiber) slows the rate of surface adsorption of water, and thus slows the rate of wicking. This is particularly illustrated by a marked increase in wicking time (at 3 cm) for cotton treated with PEG-1,450/DMDHEU (> 1,200 seconds) relative to untreated cotton printcloth (65 seconds). The woven cotton/polyester

TABLE IV

Absorbency and Moisture-Related Properties of Nonwoven and Woven
Substrates Containing Crosslinked Polyethylene Glycols ^a

Surface and/or Fiber type	M _n PEG	%H ₂ O Imbibition ^b	% Moisture Regain ^b	Drop Absorbency (sec)	Wicking (sec)		% Wet Shrinkage ^c		Area
					2cm	3cm	W or M	F or C	
Polyamide-reinforced paper towel	600	147.4	23.8	4	8	17	+2.4	+5.0	+10.8
	1,000	164.9	38.6	4	22	46	0	+10	+10.0
	1,450	257.8	59.4	4	52	90	+6.7	+3.3	+10.2
UT control	---	66.1	8.2	<1	5	16	0	0	0
Cotton printcloth	600	84.0	26.7	9	80	126	-16.7	-10	-25.0
	1,000	78.3	29.5	35	123	334	-6.7	-10	-16.0
	1,450	119.7	34.0	>1200	571	>1200	-6.7	-3.3	-9.8
UT control	---	33.7	7.2	4	25	65	-2	0	-2.0
Thermal bonded 50/50 cotton/polyester	600	216.7	39.9	7	51	125	0	0	0
	1,000	168.7	47.6	1	23	60	0	0	0
	1,450	215.7	57.4	3	27	86	0	+3.3	+3.3
UT control	---	16.8	3.3	<1	8	19	0	0	0
Woven 50/50 cotton/polyester	600	86.3	24.3	11	65	154	-13.3	-13.3	-25.0
	1,000	80.8	29.7	16	72	240	-6.7	-10	-16.0
	1,450	103.4	33.1	30	74	328	-6.7	-3.3	-9.8
UT control	---	16.3	3.5	65	100	158	0	0	0
Melt-blown polypropylene	600	356.9	19.3	544	836	>1200	+6.7	0	+6.7
	1,000	345.1	57.1	11	47	107	-3.3	0	-3.3
	1,450	409.1	57.2	101	94	209	0	0	0
UT control	---	12.5	0.4	>1200	>1200	>1200	0	0	0
Woven polypropylene	600	136.8	25.8	20	64	147	-13.3	-6.7	-19.0
	1,000	117.5	28.6	24	88	175	-10.7	-3.3	-13.7
	1,450	160.7	36.5	13	119	268	-10	-3.3	-13.0
UT control	---	2.2	0.5	>1200	>1200	>1200	0	0	0

^a Corresponds to weight gains and samples in Table I. Wicking evaluated in warp or machine direction.

^b Calculated after 16 hours drying time.

^c All materials immersed for 15 min. in tap water at 20°C, then dimensions measured to nearest 0.1 in.
(+ = growth; - = shrinkage). W = warp, F = fill for wovens; M = machine direction, C = cross direction for nonwovens.

fabric containing the crosslinked polyols generally exhibited improved wicking and drop absorbency behavior relative to the untreated fabric, and thus behaved more like the hydrophobic substrates.

Wet shrinkage of wovens and particularly of high twist knits containing the crosslinked polyols is known to be appreciable (10-30%); however, on drying the fabrics completely recover to their original dimensions (4). In contrast, nonwovens, fibrous assemblies that have no twist and are of random orientation, experience no wet shrinkage if untreated and little or no wet shrinkage even when they contain the crosslinked polyols. A comparison of treated nonwovens with the corresponding treated wovens (Table IV) substantiates this observation. Only the treated paper towels had any appreciable dimensional changes when wet, but this was characterized by growth or dimensional increases rather than by shrinkage. The other two nonwoven substrates (thermally bonded cotton/polyester and melt-blown polypropylene) were either completely unaffected in their dimensions when wet, or had very slight amounts of expansion or shrinkage. In contrast, both the woven cotton/polyester and polypropylene fabrics containing the crosslinked polyols had decreases in their area of 10-25% after wetting. Microscopic evaluations are in progress to relate the degree of yarn twist in a fibrous structure to the wet shrinkage that occurs when it contains bound polyols.

TABLE V

Sorptive Capacity Evaluation of Nonwovens
Containing Crosslinked Polyethylene Glycols

Surface and/or Fiber type	M _n PEG	Absorbency ^a Time (sec)	Absorptive ^a Capacity (%)	% H ₂ O Retention Value by Centrifugation ^b
Thermal bonded 50/50 cotton/polyester	600 1,000 1,450	2.5 2.0 2.5	1,385 1,375 1,390	175 127 116
UT control	---	21.8	688	23
Melt-blown polypropylene	600 1,000 1,450	413 137 98	696 580 555	275 191 231
UT control	---	>1200	65	17
UT cotton printcloth	---	c	c	44

^a Determined by INDA Test 10.0-70 (R82).

^b Centrifuged for 5 min. at 1,000 g; calculated by method of ref. (6).

^c Served as control for centrifuge test only; untreated cotton values typically range from 35-60% under these conditions.

The sorptive capacity or amount of liquid retained by a substrate after a drainage process (exsorption rather than absorption) has been measured by many different techniques, including retention of water after drainage by gravity, centrifugation and hydrostatic pressure (9). The first two techniques were utilized to evaluate untreated and treated thermally bonded cotton/polyester and melt-blown polypropylenes (Table V). In the immersion and gravity drainage test, both time (for sample to become completely wet) and capacity were measured. Since the unmodified polypropylene would not remain submerged, it theoretically had an infinite time for wetting; treated polypropylenes had wetting times that became shorter with increasing molecular weight of the bound polyol. The treated cotton/polyester sample became wet ten times faster than the untreated sample irrespective of molecular weight of bound polyol. The sorptive capacities of the treated thermally bonded cotton/polyesters were twice that of the untreated material, and ten times greater for treated polypropylenes relative to the unmodified control.

When the same untreated and treated nonwovens were evaluated for their liquid retention by centrifugation, trends observed were the same as those observed in the gravity drainage test. Treated cotton/polyester nonwovens had five to eight times the sorptive capacity of untreated nonwovens, and treated melt-blown polypropylenes 10-15 times the sorptive capacity of the untreated control.

Evaluation and optimization of the very high water sorbency and capacity of various nonwoven substrates containing cross-linked polyols are continuing with regard to mechanisms of absorption/exsorption and utilization of these properties in many applications such as filtration, diapers, wipes and other end uses.

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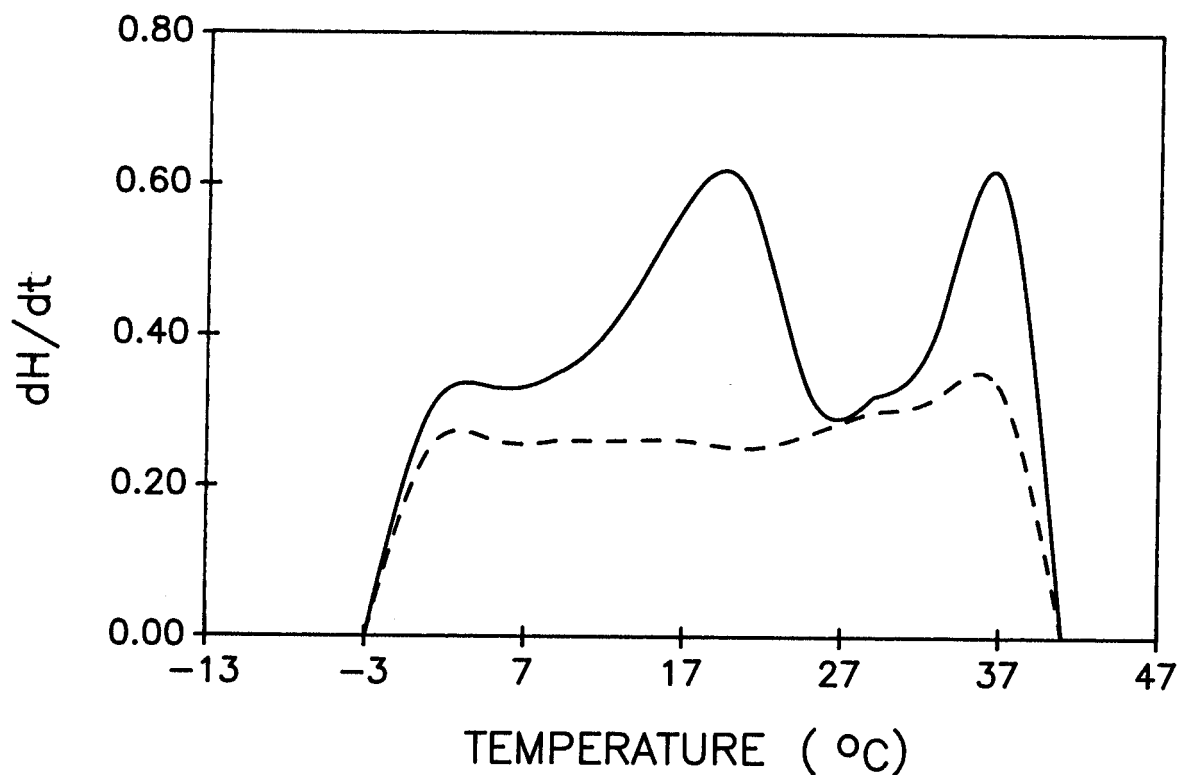


Figure 1. Heat content (Q) from -3 to +37 $^{\circ}C$ for untreated (---) and PEG-1,000/DMDHEU-treated (____) paper towels.

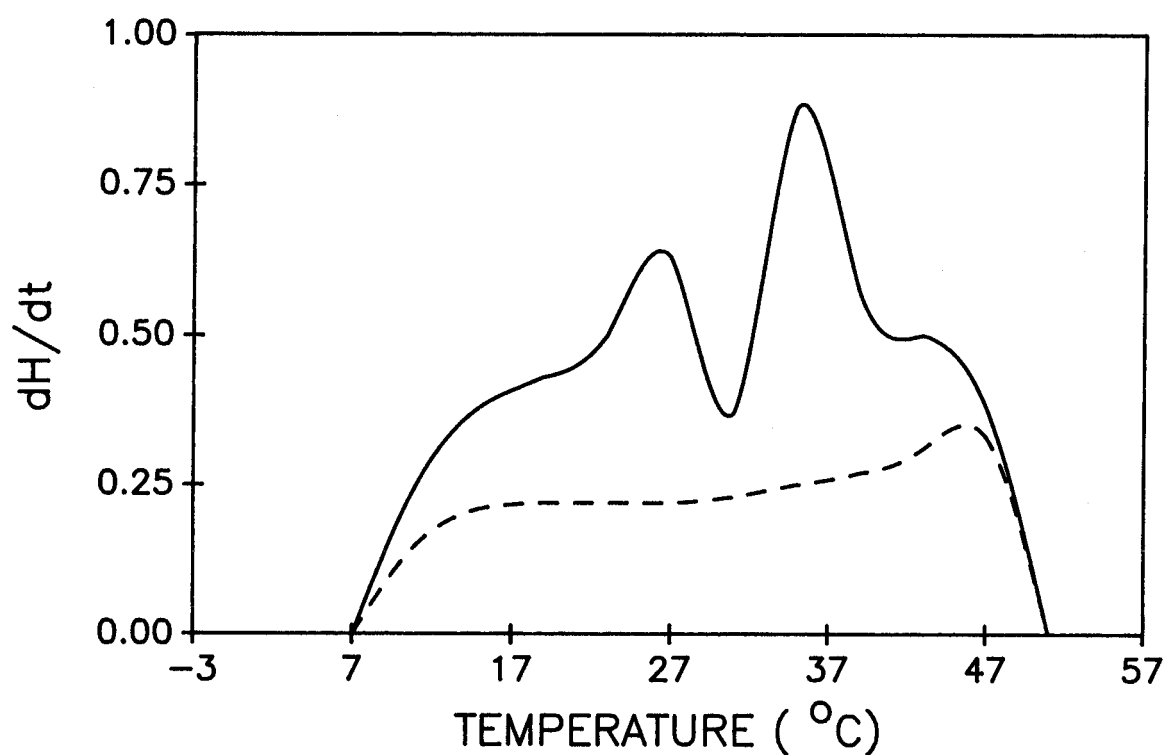


Figure 2. Heat content (Q) from +7 to +47 $^{\circ}C$ for untreated (---) and PEG-1,450/DMDHEU-treated (____) thermally bonded cotton/polyester nonwovens.

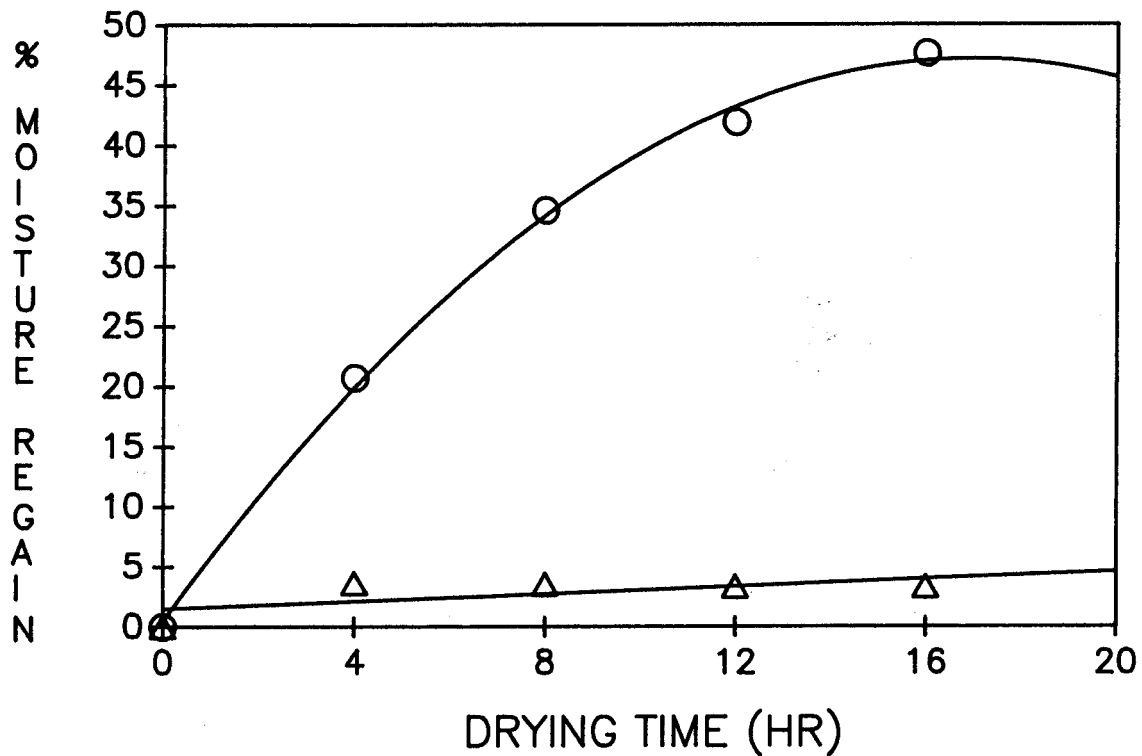


Figure 3. % Calculated moisture regain vs hrs. previously dried at 110°C for untreated (Δ) and PEG-1,000/DMDHEU-treated ($\circ$) thermally bonded cotton/polyester nonwovens.

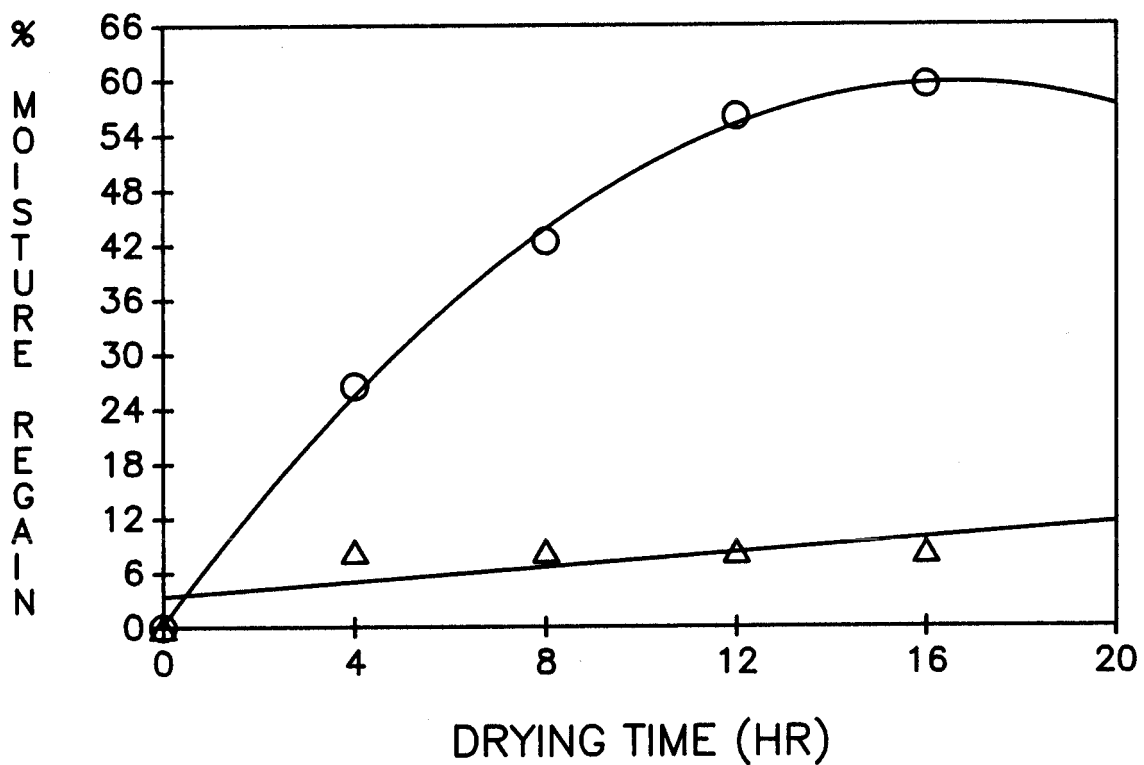


Figure 4. % Calculated moisture regain vs hrs. previously dried at 110°C for untreated (Δ) and PEG-1,450/DMDHEU-treated ($\circ$) paper towels.

CONVERSION FACTORS FOR SI UNITS

Quantity or Test	Value in Trade or Customary Unit	X	Conversion Factor	=	Value in SI Unit	Symbol
Area	square inches		6.45		square centimeters	cm ²
	square feet		0.0929		square meters	m ²
	square yards		0.836		square meters	m ²
	acres		0.405		hectares	ha
Basis Weight* or Substance (500-sheet ream) or Grammage* when expressed in g/m ²	17 x 22		3.760		grams per square meter	g/m ²
	24 x 36		1.627		" " " "	"
	25 x 38		1.480		" " " "	"
	25 x 40		1.406		" " " "	"
	pounds per 1000 square feet (Paperboard)		4.882		" " " "	"
Breaking Length	meters		0.001		kilometers	km
Burst Index	$\frac{\text{g/cm}^2}{\text{g/m}^2}$		0.0981		$\frac{\text{kPa}}{\text{g/m}^2}$	
Bursting Strength	pounds per square inch		6.89		kilopascals	kPa
Caliper	mils		0.0254		millimeters	mm
Concora Crush	pounds		4.45		newtons	N
Edge Crush	pounds per inch		0.175		kilonewtons per meter	kN/m
Energy	British thermal units (Btu)	1055			joules	J
Flat Crush	pounds per square inch		6.89		kilopascals	kPa
Force	kilograms		9.81		newtons	N
	pounds		4.45		newtons	N
Length	angstroms		0.1		nanometers	nm
	microns		1		micrometers	um
	mils		0.0254		millimeters	mm
	feet		0.305		meters	m
Mass	tons (2000 lb)		0.907		metric tons	t
	pounds		0.454		kilograms	kg
	ounces (avd p)		28.3		grams	g
Mass per Unit Volume	ounces per gallon		7.49		kilograms per cubic meter	kg/m ³
	pounds per cubic foot		1.60		kilograms per cubic meter	kg/m ³
Puncture Resistance	foot pounds		1.36		joules	J
Ring Crush	pounds (for a 6-in length)		0.0292		kilonewtons per meter	kN
Stiffness (Taber)	gram centimeters (Taber Units)		0.0981		millinewton meters	mN.m
Tear Strength	grams		9.81		millinewtons	mN
Tensile Breaking Load	pounds per inch		0.175		kilonewtons per meter	kN/m
	kilograms per 15 millimeters		0.654		kilonewtons per meter	kN/m
Volume, Fluid	ounces (US fluid)		29.6		milliliters	mL
	gallons		3.79		liters	L
Volume, Solid	cubic inches		16.4		cubic centimeters	cm ³
	cubic feet		0.0283		cubic meters	m ³
	cubic yards		0.765		cubic meters	m ³

*See TAPPI Technical Information Sheet 0800-01.

