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Application of Ultrasound in Textile Wet Processing

Phase 1

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Application of Ultrasound in Textile Wet Processing

Phase 1

Ultrasound, or ultrasonic energy, promises significant benefits to the textile industry through improvements in two vital areas—cost of manufacturing and solutions to environmental impact problems. Cost of manufacturing would be reduced through the improved quality and speed of processing. Significant environmental benefits would be realized because of the reductions in requirements for energy, water, and processing chemicals.

INTEREST CATEGORY

Industrial

KEYWORDS

Textile
Electrification
Electrotechnology
Energy efficiency

BACKGROUND The U.S. textile industry is under tremendous pressure from international competition. New technologies are urgently needed to keep the industry competitive. Past research on the use of ultrasonics in textile dyeing has revealed possible applications in dye migration enhancement, better washing and scouring of textile material, and reduction in the total use of energy.

OBJECTIVES

- To develop a pilot plant–scale ultrasonic continuous yarn-dyeing system.
- To establish a solid foundation of knowledge, technical manpower, and facilities for the continuation of basic and applied research in the use of ultrasonic energy in textile processes.

APPROACH Researchers first reviewed the literature on ultrasound-enhanced textile wet processing. On the basis of the findings, they designed laboratory- and pilot-scale equipment as well as conducted studies using various dye and fiber combinations. In laboratory studies they used small vessels of approximately 1-liter capacity and ultrasound probes of 400–1200 W output. Pilot studies were conducted in a 40-gallon dye tank using ultrasound power of approximately 5 kW.

RESULTS Investigations on dye diffusion showed that ultrasound increased diffusion coefficients by typically 30% and permeability coefficients by more than 300%, thereby enhancing dye penetration. The apparent activation energy of diffusion was decreased by approximately 24%. The decrease in activation energy shows a potential for reducing dyeing temperatures. Ultrasound also increased the reactivity of fiber-reactive dyes. This knowledge of ultrasonic effects on fundamental processes leads to a better design for the dyeing process in pilot plant and commercial scale-up applications.

Researchers obtained the most promising pilot plant results in the dyeing of nylon with acid dyes. Results clearly indicate that both dyes and thermal energy can be saved in dyeing, and productivity can be increased.

Ultrasonic techniques may also benefit polyester dyed with disperse dyes. One hundred percent polyester dyed in the laboratory had approximately 150 percent increased depth. Increase in depth to this magnitude was not seen in pilot plant dyeing because of the low ultrasonic energy in the pilot plant unit.

EPRI PERSPECTIVE Textile wet processing is a major consumer of energy, most of which is fossil fuel driven. This study shows that the use of ultrasound for dyeing can replace expensive thermal energy and chemicals—which have to be treated in waste water—with electricity. It also considerably improves the quality of dyed fiber, thereby potentially enhancing the competitiveness of the U.S. textile industry.

PROJECT
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Application of Ultrasound in Textile Wet Processing

Phase 1

TR-101379, Phase 1
Research Project 2782-06

Final Report, November 1992

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ABSTRACT

The U.S. textile industry urgently needs new technologies to keep the industry competitive. Earlier research has revealed that ultrasound can be used to enhance dye migration, improve the washing and scouring of textile material, and reduce energy use.

This study was designed to develop a pilot plant–scale ultrasonic continuous yarn-dyeing system and to establish a foundation for continuing research in the use of ultrasonic energy in textile processes. On the basis of their findings in the literature on ultrasound-enhanced textile wet processing, researchers designed laboratory- and pilot-scale equipment and conducted studies using various dye and fiber combinations. In laboratory studies they used small vessels of approximately 1-liter capacity and ultrasound probes of 400–1200 W output. Pilot studies were conducted in a 40-gallon dye tank using ultrasound power of approximately 5 kW.

Investigations on dye diffusion showed that ultrasound increased diffusion coefficients by typically 30% and permeability coefficients by more than 300%, thereby enhancing dye penetration. The apparent activation energy of diffusion was decreased by approximately 24%. The decrease in activation energy shows a potential for reducing dyeing temperatures. Ultrasound also increased the reactivity of fiber-reactive dyes. Researchers obtained the most promising pilot plant results in the dyeing of nylon with acid dyes. Ultrasonic techniques may also benefit polyester dyed with disperse dyes. One hundred percent polyester dyed in the laboratory had approximately 150 percent increased depth. Increase in depth to this magnitude was not seen in pilot plant dyeing because of the low ultrasonic energy in the pilot plant unit.

This knowledge of ultrasonic effects on fundamental processes leads to a better design for the dyeing process in pilot plant and commercial scale-up applications. The study shows that the use of ultrasound for dyeing will replace expensive thermal energy and chemicals, which have to be treated in waste water, with electricity. It also improves the quality of dyed fiber, thereby potentially enhancing the competitiveness of the U.S. textile industry.

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Overview

The purpose of this project is the continuation of our investigation of the applications of ultrasound in textile wet processing. Previous to the project work performed in the past two years, laboratory studies had already shown that ultrasound had a pronounced effect on the rate of dyeing for several dye classes and various types of fibers. Our plan was to continue with additional laboratory work designed to further our understanding of the fundamental principles governing the effects of ultrasound on textile processes and to duplicate beneficial laboratory results in the pilot plant.

At the outset of this work, a complete literature search was done, and results were acquired from previous studies of other investigators. The literature review is in Section 2 of this report.

The laboratory bench studies were conducted in small vessels containing typically less than 1 liter of liquid and an ultrasound probe producing an estimated 400 to 1200 watts output. Laboratory investigations conducted during the course of this project tested the effects of ultrasound on:

- diffusion and permeability coefficients
- reactivity of fiber reactive dyes
- dyeing studies (fiber reactives on cotton, acid dyes on nylon, and disperse dye on polyester).

In the pilot plant, using a 40 gallon ultrasonic cleaning tank, our efforts focused on:

- construction of a prototype dyeing machine
- construction of a monitoring and control system for the prototype dyeing machine
- dyeing various substrates and evaluating performance of the prototype machine and process
- modification of the prototype to increase the ultrasound power level
- further evaluation and optimization of prototype machine, processes and dyed products

rate of washing-off of fiber reactive dyes and disperse dyes

Technical Summary of Experimental Work

Application of ultrasonic energy has been assessed in textile dyeing and washing processes. Investigations have been carried out on the laboratory and pilot plant scale. Prototype laboratory investigations were carried out with ultrasound transducers which operated at up to 1200 watts power from a 20 kHz generator. Pilot plant trials were conducted in a modified ultrasonic parts cleaner tank operated at 3200 watts power from a 43 kHz generator. A submersible transport system was constructed at NCSU to transport yarn and fabric through the tank. In the latter stages of this project the power density of ultrasound was supplemented in the pilot plant unit by the addition of an 1800 watt submersible transducer unit operating at 26 kHz. This brought the total ultrasound tank power up to 5.0 kW.

To better understand the effects of ultrasound on various stages of dyeing processes, fundamental laboratory studies of diffusion of dyes and reactivity of fiber reactive dyes were carried out. Investigations on dye diffusion showed that ultrasound increased diffusion coefficients by 30% and permeability coefficients by more than 300%. The apparent activation energy of diffusion was decreased by 24%. The decrease in activation energy confirms the previously-observed potential of reducing dyeing temperatures. Also, ultrasound increased the reactivity of fiber reactive dyes. These understandings of ultrasonic effects on fundamental processes will enable us to better design an ultrasonic dyeing process in pilot plant and future commercial scale-up applications.

Ultrasonic techniques seem potentially beneficial in dyeing polyester with disperse dyes. Polyester fabric dyed at atmospheric pressure in the laboratory had approximately 150% increase in color depth with the application of ultrasound compared with a sample dyed under the same condition but without ultrasound. This further increased to over 250% with booster horns. Increase in depth to this magnitude was not seen on pilot plant dyeings, apparently because of lower ultrasonic energy. Ultrasound marginally increased the color yield (13%) on cotton yarn with direct dyes in the pilot plant, again, we believe, due to lower ultrasound power levels. With reactive dyes this increase was in the range of 20 to 33 percent. Ultrasound power levels used in the laboratory studies were estimated at 15 to 20 watts of ultrasound power per square inch (watt/in^2) of exposed fabric surface. The initial pilot plant prototype tank had an estimated power density of 4 watts/in^2. This was later augmented by

additional submersible units to 6 watts/in^2. Thus the power of ultrasound applied in the laboratory studies was three times greater than in the pilot plant studies. It is also suggested that a means of increasing the ultrasound power level of the pilot plant tank be investigated to duplicate the results shown in the laboratory tests.

The most promising pilot plant results were obtained in the dyeing of nylon with acid dyes. Results clearly indicate benefits in terms of savings in dyes and thermal energy and an increase in productivity. The fastness properties of insonated dyeings were to comparable or better than control dyeings. For cotton yarn dyed with Direct Red 81, ultrasound increased the color depth by 13%. The fastness properties of knitted fabric dyed with and without ultrasound were comparable.

In the washing off of fiber reactive dyed cotton fabric, the washing time, ranging from 0.5 to 16 minutes, was reduced by over 60% by the use of ultrasound at power densities of 4 and 6 watts/inch^2. More detailed studies are necessary to evaluate washing fabric in a continuous manner for direct comparison to industrial washers to determine the efficiency of ultrasonic energy. Tests using the pilot plant tank with moving fabric are suggested.

Phase II of the research will: (1) analyze the amount of ultrasound energy in the laboratory and prototype ultrasound generators, (2) increase the ultrasound power level of the prototype to at least the laboratory intensity level, and (3) dye and wash materials in the tank to determine benefits of ultrasound in a pilot plant scale.

Section 1

LITERATURE REVIEW OF THE APPLICATION OF ULTRASOUND TO TEXTILE WET PROCESSING

Introduction

Wet processing of textiles uses large quantities of water, and electrical and thermal energy. Most of these processes involve the use of chemicals for assisting, accelerating, or retarding their rates and must be carried out at an elevated temperature to transfer mass from processing liquid medium across the surface of the textile material in a reasonable time. Like all chemical processes, these transport processes are time and temperature dependent, and compromising either could affect product quality. Ultrasonics may be employed to reduce processing time and energy consumption, and maintain or improve product quality. The idea of utilizing ultrasound in the textile processes is not a new one, and there is a considerable body of literature on the improvement and acceleration of textile processes using ultrasound [1-5]. In addition to its well established applications in forming stable dispersions and cleaning materials and machine parts, several new areas are being investigated. The widespread availability of cheap and reliable ultrasound generators, mainly in the form of cleaning baths, has promoted increased interest in the effects of ultrasonics in textiles. Growing interest in sonochemical reactions[6] is an example which does not require the use of specialized equipment.

This section reviews the theory of ultrasound and the work carried out so far in textiles, and identifies processes which could benefit with the addition of ultrasound.

Ultrasound - Its Propagation and Cavitation

Ultrasonic waves are sonic vibrations with a frequency above 17kHz - beyond the audible range of a human being. Like electromagnetic waves, ultrasonic waves can also be focused, reflected and refracted, but they require a medium with elastic properties for their propagation. In this respect, they differ from light and other forms of electromagnetic radiation which travel freely through a vacuum. When ultrasonic waves propagate, particles in the elastic medium oscillate and transfer energy through the medium in the direction of propagation. The marked effects of ultrasound actually arise from the way in which sound is propagated through the medium. The waves can be distinguished as:

- longitudinal waves in which the particles oscillate in a direction parallel to the direction of propagation of the wave.
- transverse or shearing waves in which the particles oscillate in a direction perpendicular to the direction of wave propagation.

In a solid, both longitudinal and transverse waves can be transmitted, but in gases and liquids only the longitudinal waves are transmitted [7]. In liquids, longitudinal vibration of molecules generates a series of compressions and rarefactions, i.e., areas of high and low local pressure. The low pressure in the rarefaction region can give rise to the formation of cavities or bubbles. These bubbles expand and finally, during the compression phase, collapse violently generating shock waves. The phenomenon of bubble formation and collapse is known as cavitation, and is generally considered responsible for most of the physical and chemical effects that are observed in solid/liquid or liquid/liquid systems.

The occurrence of cavitation depends upon several factors such as the frequency and intensity of waves[8], temperature of the liquid medium, and its vapor pressure. Very high frequencies are less favorable for cavitation and require large amounts of energy -- much of which is converted into thermal energy which heats the medium. At low frequencies, cavitation can be initiated at moderate intensities.

Intensity is a measure of the energy available per unit volume of the sample or material. Applications of ultrasonics can be divided into two categories -- low intensity and high intensity. In the low intensity applications, input power levels are low enough that there is never any change in the state of medium. Typical applications are non-destructive testing of materials and measurement of elastic properties of materials. High intensity applications are those where phase changes (i.e., vaporization, cavitation) occur. These have a more severe effect on the medium and are important for all wet processes. In most chemical reactions, the reaction rate is found to increase with intensity. Intensity of the waves is, however, greatly affected by the temperature of the medium. Investigations have shown that with the rise in the temperature of the liquid, the effects of intensity are reduced. In water, the effects of cavitation reach maximum at 50°C. The phase structure of the system is another criterion that determines whether the application of ultrasound will be beneficial or not. The effect of cavitation is several hundred times greater in a heterogenous system (e.g., like all textile wet processes) than in homogeneous systems.

Ultrasonic Equipment

Typical ultrasonic equipment usually consists of two parts: (1) the generator (power supply) and (2) the converter or cleaning bath. The generator converts conventional 50 or 60 Hz alternating current to high frequency electrical energy. This high frequency electrical energy is fed to the transducer(s) where it is transformed to mechanical vibration. Modern equipment uses piezoelectric crystals (e.g., lead zirconate titanate) which, when subjected to an alternating voltage, expand and contract. The transducer system vibrates longitudinally, transmitting waves into the liquid medium. As these waves propagate, cavitation and other previously described effects occur. Horn type converters are generally suitable for treating a small volume of liquid. These produce very high intensity, but energy distribution throughout the vessel may not be uniform. Non-uniform distribution of ultrasonic energy results in uneven treatment of textile substrates and uneven shades when dyeing.

Economic Considerations

Most reports claim beneficial effects of ultrasound in textile wet processing. However, few attempts have been made to use this technique on an industrial scale, probably due to the high cost of the equipment. With technological advances and widespread use of cleaning baths, the cost of ultrasonic equipment is decreasing. The cost of a bath is determined mainly by the cost of its power supply and the transducers.

Doubling the power by fitting twice as many transducers on the equipment generally doubles the prices. Calculations based on experiments and considerations on installation and depreciation costs show that the total cost of ultrasonic processing is between 9.5 and 33 cents/pound of material. Considering other potential benefits of sonochemistry such as lower operating temperature, reduced heating costs, less thermal degradation of products or shorter batch times, easier process control, etc., sonochemical reactors in practice may be economically justifiable.

Ultrasonics in the Textile Industry

The application of ultrasonics in the textile industry can be divided into two main categories:

- utilization in auxiliary processes
- the modification of textile wet processes

Utilization in Auxiliary Processes

These applications relate to preparation of auxiliary baths for processes, i.e., preparation of sizes, emulsions, dye dispersions and thickeners for print paste. Several such examples of ultrasonics are cited in the literature [9-17]. In the preparation of starch sizing agents, for instance, starch is mixed with water, heated to 95°C and held at that temperature for an hour before being used. By using ultrasonics, the operation can be conducted more rapidly and at a lower temperature. Experiments on the performance of sized yarns have shown that starch sizing agents prepared with ultrasonics are superior to conventionally prepared starch and to chemically modified starch. Ultrasonic homogenizers are used to prepare emulsions and solutions of fiber lubricants that are applied to reduce inter-fiber friction and static electricity. Ramaszeder [3,19] prepared water-oil emulsions using an ultrasonic device that remained stable for more that 212 hours, whereas those prepared by conventional mechanical stirring separated into phases after standing for only 12 hours. Lifchits [14] used ultrasonics in the preparation of paraffin/styrene emulsions to obtain a homogenous emulsion with reduced particle size of 1 μ as compared with 3 μ in the case of the conventional method.

All such applications make use of the dispersion effect of ultrasound. The dispersing action of ultrasound is widely used on commercial scale in the cleaning of machine parts[22]. For example, needles in knitting machines, spinnerets forming the chemical fibers, and open-end yarn spinning rotors are all reportedly cleaned by this method.

Modification of Textile Wet Processes

Applications in this group concern heterogeneous systems in which processing baths containing textile substrates are influenced by ultrasonic waves. There are a number of reports in which ultrasonic equipment has been used in preparation processes, for example, desizing, scouring and bleaching, dyeing and finishing, and washing. The objective of using ultrasound is different in each of these processes. In preparation and washing operations, the object is to remove natural material or impurities (soil) from the surface of the fiber, whereas in dyeing and finishing processes, it is to transport or diffuse dyes or chemicals into the fiber. Because of the complexity of these processes, and since in all these processes the rates reportedly are increased by ultrasound, it is difficult to understand exactly how ultrasound affects these widely diverse processes. Observed improvements are generally attributed to cavitation.

Desizing

In a study on the sizing and desizing of textiles with starch, size removal was effected by means of ultrasonically accelerated techniques [23]. It was found that the use of degraded starch followed by ultrasonic desizing could lead to considerable energy savings when compared to conventional starch sizing and desizing.

Valu et al [24], in their investigations of the use of ultrasonics in the desizing of woven cotton fabric, achieved a savings in chemicals and energy as well as reduced fiber degradation. The final whiteness and wettability of the fabrics were the same as those obtained without ultrasonics. These investigations were carried out on an industrial jig in which ultrasonic transducers were mounted on the walls. Power varied from 2kW to 4kW for different transducer positions.

Scouring and Bleaching

Garlinskaia, Dolgoponof, Mameykiy and Roubane reported that it was possible to scour wool in neutral or very slightly alkaline baths [2]. Fiber damage was less when ultrasound was used as compared to conventionally scoured wool fibers. Another study on the scouring of wool [25] also claims that fiber properties are improved and the rate of processing increased when ultrasonics are used. Satonov [26] used 20kHz frequency in the peroxide bleaching of cotton fabrics and observed an increase in the bleaching rate and reduction in required time. The whiteness of the fabric was improved over conventionally bleached fabrics. Investigations on the effect of ultrasonics on the processing of flax fibers indicate that whiteness is improved over conventionally scoured and bleached flax fibers [27,28].

Dyeing

Dyeing of textiles with ultrasound has been the subject of many studies [29-33]. Both low and high frequency sound waves have been used to study their effects on the quality of dispersion of dyes, change in solubility of water soluble dyes and dye absorption by textile materials.

Fredman, [29-33] using a 30kHz frequency, obtained a very fine dispersion of phthalocyanine pigments in distilled water and also had a longer life than that prepared by conventional stirring. The effect of ultrasound on the quality of dispersions was also studied by Simanvoich [35]. He prepared dispersions of vat and disperse dyes at 50°C using 9.4 kHz frequency for a period of 20 minutes and determined the change in particle size of the dispersion by electron microscopy.

About 93 percent of the dyes had particle sizes less than 1 μ as compared to only 50 percent in dispersions prepared by conventional method.

Kabilyus [36] studied the change in the solubility of direct dyes in cold water. He noticed a considerable increase in the solubility of two direct dyes (*e.g.* Blue M and Brown mX) when low frequency ultrasound was added to the bath, but there was no change in the solubility of Direct SkyBlue dye. Similar observations were made by Smirnova and Tynin [37] in their investigations. They concluded that the effectiveness of the treatment depends upon the nature of dyes and their physicochemical properties, in particular their solubility in water.

At the same time, attempts were also being made to study the effects of ultrasound on dyeing processes. Sokolov and Tumanski [36] produced vibratory waves of 9.5 kHz frequency and found a 2 to 3 fold increase in rate of dyeing with substantive dyes on all cotton fabric.

Soon after their studies, increased interest was developed in this area. Bruer [39] succeeded in reducing the dyeing time of vat dyes on cellulosic fabric by 75% as compared to conventional dyeing time. The depth of the shade using 175 kHz was much darker than that with 22 kHz. Their earlier work using audio frequency waves of 0.050 kHz and 3.0 kHz did not show improvement in the dye absorption. Rath and Merk [40] studied the effects of audio and ultrasound waves on dyeing of various substrates such as cotton, viscose and wool using direct and acid dyes respectively. In order to evaluate the true effect of sound waves on dyeing, they rotated fabric in a dyebath and varied frequency from 1 to 8 kHz in the audible range and 22, 30, 80, and 175 kHz in the ultrasound range. They found that low frequency waves had marginal effects on dyeing rates of direct dyes on cotton and viscose and considerable effect on the absorption of disperse dyes on cellulose acetate, and Perlitone dyes on Perlon. Investigations using higher frequencies, 22KHz to 175 kHz, showed increases in dye absorption. This effect was greatly dependent on the intensity of the waves. A noticeable difference in dye absorption was observed when the intensity was increased from 1 to 3 w/cm^2.

Researchers attributed acceleration in dyeing rates to cavitation, but the mechanism of the action was still not clear. Attempts are still underway to differentiate the effects of cavitation from those of mechanical stirring. Alexander and Meek [41] dyed cotton with direct dyes, wool with acid dyes, and nylon and acetate with disperse dyes with the addition of ultrasound at 17.3 kHz. In cotton and wool dyeings, ultrasound and mechanical stirrers produced similar dyeing results. With disperse dyes on nylon and acetate fibers, an increase in the rate of dyeing was obtained

as compared to the best rate of dyeing using mechanical stirring. From these observations, ultrasound was concluded to be more beneficial on hydrophobic fibers dyed with water insoluble dyes. Contrary to their findings that ultrasound is less effective for water soluble dyes, Chuz and Domorslov [42] obtained quality dyeings with water soluble systems on an industrial scale. They produced about 400 dyeings with vat dyes on cotton cloth in a dyeing and finishing range modified with ultrasound operating at a power of 1.5 kW power and a 22 kHz frequency. They claimed a 50 percent reduction in dyeing time, 10 percent reduction in the consumption of dye, water and steam, and 2% percent reduction in chemicals. Their hypothesis to explain increased dye absorpion and accelerated dyeing rate was an increase in dye diffusion inside the fabric. No quantitative data was presented.

The influences of ultrasound on the dyeing system are proposed to have three effects [48]:

(i) Dispersion: breaking up of micelles and high molecular weight aggregates into uniform dispersions in the dye bath

(ii) Degassing: expulsion of dissolved or entrapped gas or air molecules from fiber capillaries and interstices at the crossover points of fabric into liquid and removal by cavitation, thus facilitating dye-fiber contact

(iii) Diffusion: Accelerating the rate of diffusion of dye inside the fiber by piercing the insulating layer covering the fiber and accelerating the interaction or chemical reaction, if any, between dye and fiber.

While these effects seem acceptable, no detailed studies of microphysical fiber structure changes in the ultrasonic field were presented. Wisniewska, [49,50] on the basis of his previous work, stated that fibrous material is not always inert to ultrasonic waves of large amplitude and, therefore, the dyeing mechanism in the presence of ultrasound can be understood only by taking into account the action of waves on the fiber as well as the dyebath. He studied the kinetics of dyeing polyamide fabric with acid and disperse dyes in the presence of ultrasound at 0.6 MHz and 2.0 MHz frequency and at 50°C, 70°C, and 90°C and observed acceleration in dyeing rates and overall increase in the depth of dyeings in all cases compared to control dyeings. The maximum increase in absorption rate was observed at the lowest temperature at which the molecules do not possess sufficient kinetic energy to diffuse in the rigid macromolecule. He attributed this to deterioration in the orientation of crystallites in the insonated fibers so that more sites are accessible to the dye molecules. On the basis of quantitative data, he concluded that by using ultrasound, the total time, temperature and dye concentration can be reduced while still

achieving the same amount of dye in the fiber as compared to the control dyeings. However, the effects are dye specific and depend greatly on ultrasonic intensity.

Thakore [1-52(i,ii)] carried out a physicochemical study on the kinetics of dyeing of cotton fabrics with two pure direct dyes in an ultrasonic field of 40kHz. A significant rise in the temperature of the liquid mass in the tank was observed and the quantitative determination of the heat transfer process revealed that about 65% of energy is converted into heat. In such a case, the dyeing system is not influenced by cavitation only, but by the combination effects of cavitation and induced heat. Two approaches have been developed to determine the contributions of each effect in isolation and in combination in the dyeing process. In the first approach, the temperature rise due to insonation was nullified by circulating cold water around the liquor. Studies were carried out under variable dye and electrolyte concentration at constant temperatures (45, 60, and 80°C) with and without insonation. The acceleration of dyeing rate and increased dye absorption in this case were due to the effect of cavitation only.

The time-temperature profile of the insonated liquor was simulated by electrical heating of the dye liquor (without insonation) and dyeing experiments were conduced in both cases. The observed increase in the rate and absorption of dye were ascribed to be the combined effects of heat and cavitation. The diffusion coefficient of dyes was found to be increased by the ultrasonic field under the effect of cavitation. The dyeing activation energy [52-59] of C.I. Direct Blue 1 was approximately 9kcal/mole as compared to 13kcal/mole without ultrasound. X-ray diffraction studies, scanning electron microscopy and tensile strength testing of insonated fabrics revealed neither permanent loosening of the structure nor any morphological changes. It was also concluded that, by using ultrasonics, the same amount of dye can be exhausted on the fabric under one or more of the following: a) reduction in dyeing temperature, b) reduction in dyeing time, and c) reduction in dye and electrolyte concentrations.

Dye equilibrium studies were investigated by Smith et. al. in the dyeing of mercerized cotton yarn and various fabrics with a number of direct dyes. Using an ultrasonic probe operating at 20 kHz and 180 watts, true equilibrium was not reached even after the dye was insonated for four hours. An increase in the apparent standard affinity of all dyes studied was observed. However, the magnitude of this increase was dye specific. Dyeing isotherms obtained at different temperatures were found to be intensity dependent. Ultrasound accelerated the rate of dyeings, increased the color yield, and also improved the quality of the fabric by reducing the warp

streaks. Investigations on dyeing of polyester and acetate fibers showed marginal increase in color yield but substantial increases in dyeing rate.

Amorphous and unoriented nylon 6 films were dyed by Yoshio et. al. [54] at 20, 40, and 60°C temperatures in an ultrasonic field at a frequency of 27 kHz. The dye absorption of all dyes was increased and activation energy decreased with ultrasound. The amount of decrease was greatest for disperse dyes and smallest for fiber reactive dyes.

Trials on synthetic fibers have shown that high temperatures can be avoided during dyeing using ultrasound. For instance, acrylic and triacetate fibers have been dyed at 77°C in 30 seconds. A 30% increase in dyebath exhaustion has been achieved in the dyeing of viscose at 60°C.

Finishing

Finishing of textiles involving ultrasonics has been studied by several researches[59-64]. Scinhovich et. al., [65i-ii] applied ultrasound at 8 and 18 kHz frequency to formaldehyde resin treatment of cotton fabric and measured the change in physical properties before and after 60 washing cycles. The crease recovery angle, even after 60 washings, was much higher than without ultrasound but resulted in a small decrease in tensile strength. In the studies of Simkovich and Yastrebinski, [66] cotton fabric was treated with urea-formaldehyde resin in an ultrasonic field of 8 kHz frequency. The isonated fabric improved crease recovery properties. By electron microscopy, x-ray diffraction analysis and infrared spectroscopy, deeper penetration of resin inside the fiber and a change in their super molecular structure was reported.

A U.S. patent describes the method and apparatus for treating military fabrics with a liquid repellent fluorochemical finish in the presence of high frequency ultrasonic waves.[67] This method produced an increase in finish add-on. Another patent by Carpenter for applying a fluid treatment to textiles [68] describes a material that is passed through a chamber in which a dispersion of fluid treating medium is ultrasonically generated. The results are claimed to be fully comparable to padding and drying and are said to be capable of producing greater levels of treatment because this method can effectively handle treating media of higher solid contents.

Washing

Washing of textiles using ultrasound is another process studied by several workers, and positive results have also been reported [69-76]. Laboratory tests have shown that the washing time of wool can be reduced from 3 hours to 15 or 30 minutes for an equivalent whiteness [18-19]. Washing off of cotton and polyester fabrics soiled with carbon black, mineral oil, edible oil, and coffee using ultrasound were studied by Antonescu et. al [62]. Polyester fabrics washed quicker, and whiteness was improved for cotton fabrics. The efficiency of washing was intensity dependent and reached a maximum at 129 w/cm^2. According to the authors, the rapid cavitation is followed by powerful shocks which act on the fabric being washed. This weakens the molecular forces of adhesion between the particles of impurities and the fabric. Under these conditions, the foreign bodies which soil the fabric surface are detached and disperse into the washing solution.

Investigations on the use of ultrasonics for washing fabrics after dyeing and printing have been reported [77,78]. Kanazawa's [79] has a recent patent on an ultrasonic washing machine which can be used to wash sheets or strands of textiles. It features an ultrasonic generator contained at the bottom of a metallic trough open at its upper end with a bubble inlet on its floor. The frequency of the generator is adjustable between 10 and 60 kHz. According to the results of a statistically planned experiment, the washing of flax can be improved by ultrasonic vibration [80]. This method removes non-cellulosic material more effectively than mechanical agitation and improves whiteness of the flax fiber.

Summary

High energy ultrasonic waves accelerate the rates of textile wet processes. Studies made over the past fifty years indicate that this technique has enough promise for application to commercial scale processes. No significant commercial installations have been developed to date for the following reasons: 1) applying ultrasound to a bath is expensive, and environmental problems have not been a significant concern until recently; 2) the ultrasound waves may be less predictable with production size equipment; and 3) intensity of the wave could vary with production size equipment.

Benefits of ultrasound in textile processes may include savings in energy, reduction in consumption of chemicals and/or dyes, and reduction in process time. Improvement in quality and easier process control are other criteria that merit consideration while determining the

economical viability of this technique. Among all wet processes, application to the dyeing process seems to be most advantageous, followed by finishing and preparation. Washing being the least expensive process, the use of ultrasound may or may not be advantageous in terms of saving money.

Another important aspect of today's concern is the possibility of reducing the pollution load on effluent water. Increased mass transfer from liquid to textile substrate leads to two possibilities: (i) increase in add-on, or (ii) less dosage of dyes or chemicals to the bath to obtain equivalent add-on to the substrate. In either case, the concentrations of pollutants in water would decrease. A final benefit might be the potential for controlling process rates with ultrasound as a substitute for chemical accelerators and retarders.

Most of the reviewed papers contain little information on the type of equipment used in the investigations. The most important factor is that the distance between the transducers and the substrate be uniform from end-to-end, side-to-side, and front-to-back.

Section 2
LABORATORY EXPERIMENTAL DETAILS

Investigations of ultrasound to improve the efficiency of dyeing processes have been carried out on the laboratory and pilot plant scale. Efforts were also made to study fundamental effects and technological aspects of applying ultrasonic energy to the dyeing process. A variety of dye/fiber systems were studied in which dyeing conditions and ultrasound related parameters were varied.

Diffusion

Diffusion of dye inside the fiber is an extremely slow process and is highly temperature dependent. Earlier work on dyeing kinetics has shown an accelerating effect on dyeing rates, but no information was available on the dye diffusion phenomena. The effects of ultrasound on the diffusion of direct dyes in cellulose film were studied using commercial samples of Pyrazol Fast Red 8 BL (C.I. Direct Red 81 obtained from Sandoz) and Pargasol Orange 2R (C.I. Direct Orange 15 of Ciba-Geigy Corp.). These dyes were used without further purification.

Cellulose film was supplied by Flexcel Corporation, and it was used after washing with 1% Na_2CO_3 solution, followed by thorough washing. The film was equilibrated to pH conditions for several hours. The thin layer chromatography (TLC) of dye solutions did not show other components.

Diffusion experiments with Red 81 dye were carried out in a standard electrochemical H-cell, model C-600, purchased from Electrosynthesis Company. The glass apparatus had two parts, A and B, which were clamped together to hold a cellophane film between them and ensuring a leak proof joint (Figure 2-1). Part A was filled with 60 mL of a 100 mg/L solution of C.I. Direct Red 81 prepared in a buffer of pH 9.0. The concentration of the solution was kept constant by circulation through an expansion reservoir of dye solution. Both the glass apparatus and the reservoir were placed in a constant temperature bath set at 60°C. Part B was filled with a buffer solution of pH 9 and was circulated to a flow-through cell positioned in a Diano Match-Scan II double beam spectrophotometer. The dye solution in Part A was well stirred with a

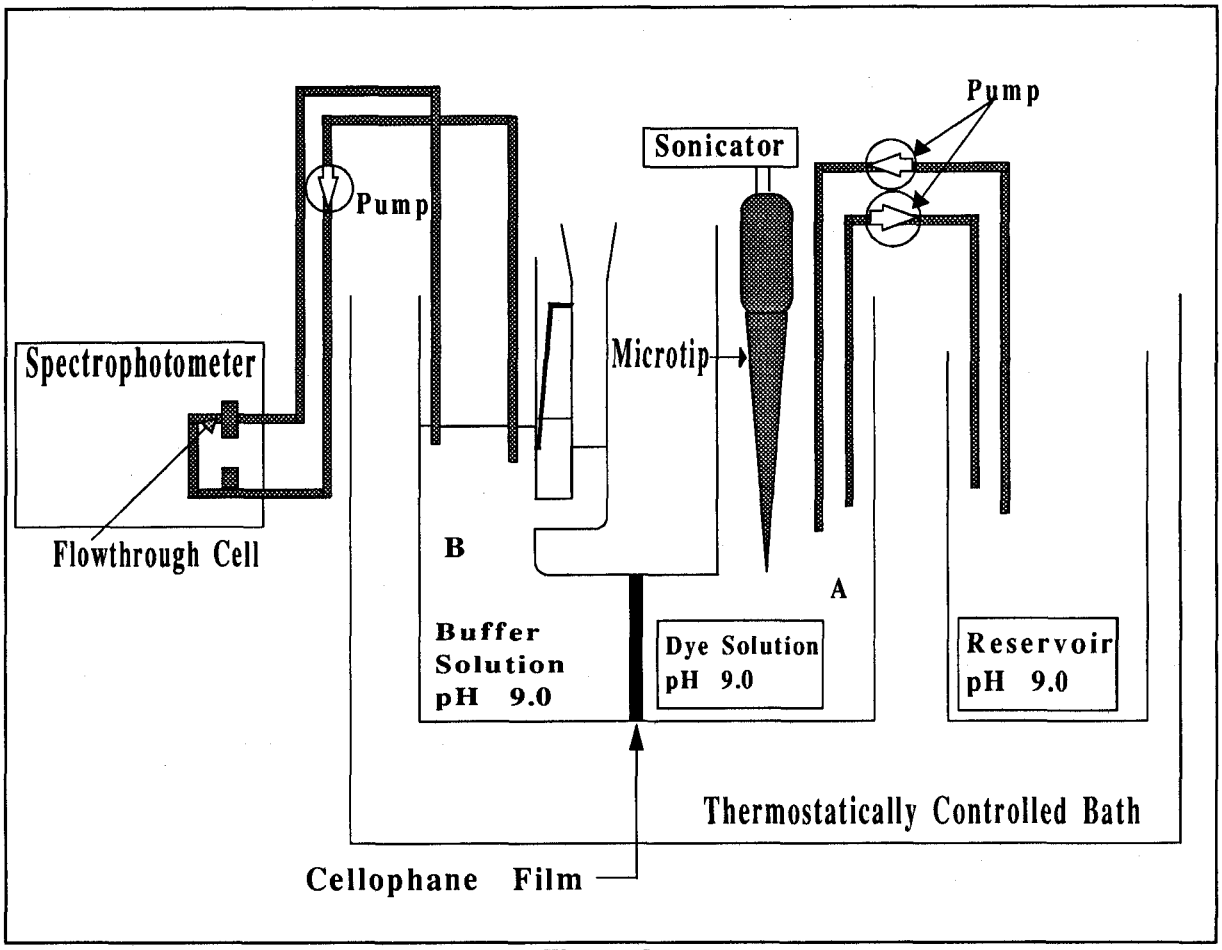


Figure 2-1
Determination of Steady State Diffusion of C.I. Direct Red 81 through Cellophane Film in the Ultrasonic Field

mechanical stirrer. In insonated dyeings, a microtip transducer was connected to a 1200 watt sonicator operated at 20 kHz frequency. The microtip was supplied with 40% and 50% available power (approximately 600 watts).

The experimental arrangement had the cellophane film parallel to the microtip at a distance of one inch. The diffusion of dye through the film in Part B was measured by the change in absorbance (at the wavelength of maximum absorption, *i.e.* λmax = 520 nm) recorded as a function of time. The amount of dye diffused in Part B was plotted against time to give the graphs shown in Figures 2-2 through 2-4. Three conditions are plotted: 1) Figure 2-2 was without ultrasound, 2) Figure 2-3 was with 40% ultrasound and 3) Figure 2-4 was with 50% ultrasound.

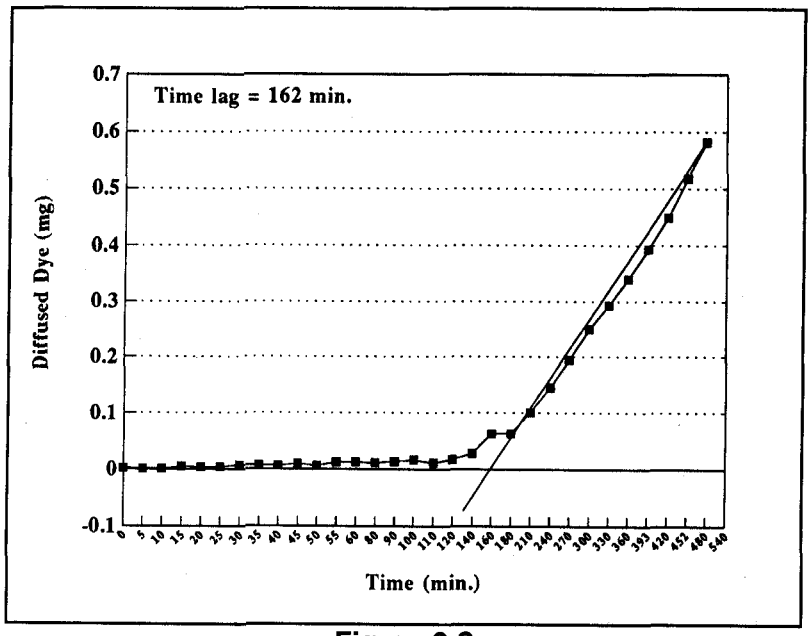


Figure 2-2
Steady State Diffusion — No Ultrasound
C.I. Direct Red 81 — Cellophane

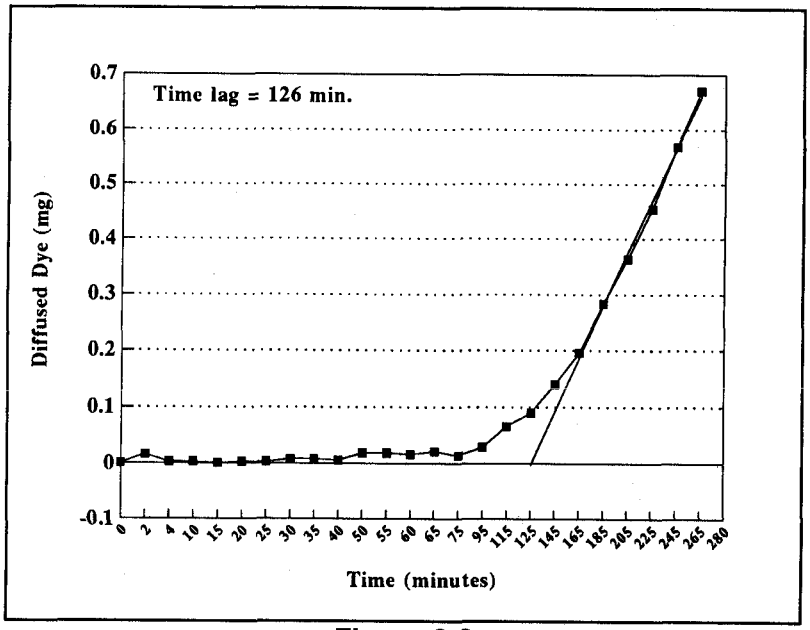


Figure 2-3
Steady State Diffusion of C.I. Direct
Red 81 through Cellophane;
40% Ultrasound

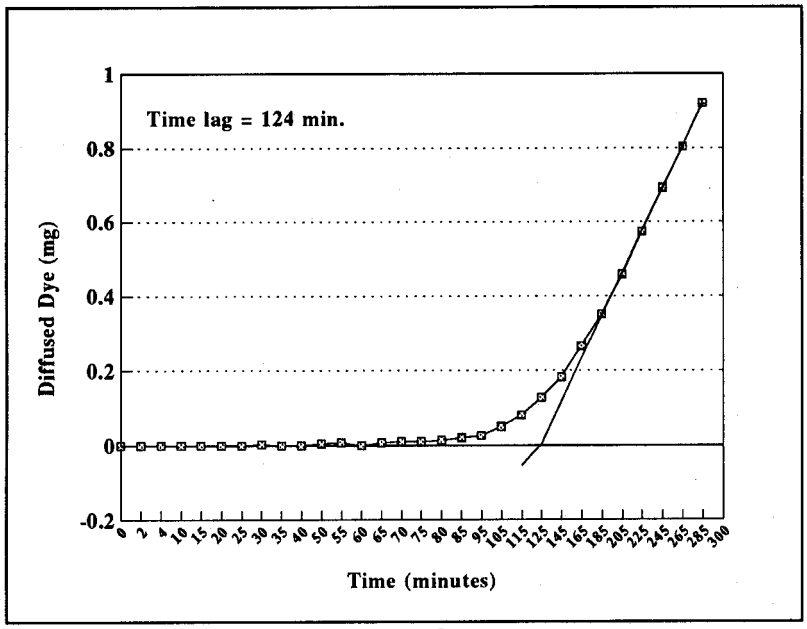


Figure 2-4
Steady State Diffusion of C.I. Direct Red 81 through Cellophane; 50% Ultrasound

The diffusion coefficient was determined from steady state diffusion experiments using Dayne's time lag method. Diffusion being a slow process, dye appears in B after a certain time lag, corresponding to the time required for diffusion through the film. When a steady concentration gradient has been established, the concentration of dye in B increases at a constant rate with time. Extrapolation of the steady state portion of this curve gives an estimate of the time lag required for diffusion through the film. Since the film thickness is known, diffusion coefficients can be deduced from the following equation:

$$D = \frac{l^2}{6L} \qquad (1)$$

where D = diffusion coefficient

l = thickness of film (0.005 cm)

and L = time lag.

The quantity of dye diffusing through the film in unit time per unit area is referred to as the permeability coefficient; it depends on the diffusion coefficient and the concentration gradient

in the film. Once a steady state has been established in a system, the permeability coefficient is the slope of the linear portion of the curves shown in Figures 2-2 through 2-4, i.e.,

$$p = \frac{ds}{dt} \qquad (2)$$

where p is the permeability coefficient and ds/dt is the slope of the line. The results of experiments carried out with and without ultrasound are given in Figure 2-5.

The data show an approximate increase of 30% in diffusion and 310% in permeability by using ultrasound in this configuration. It can be noted that (1) the direction of propagation of

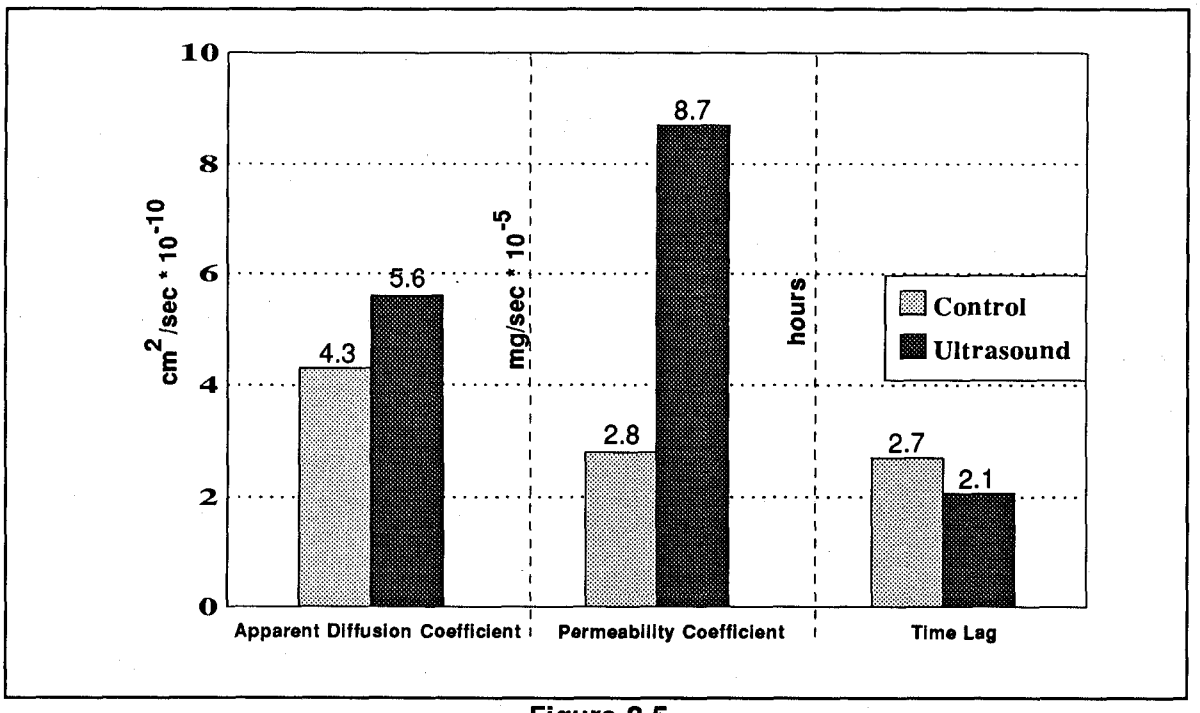


Figure 2-5
Time Lag, Diffusion and Permeability Coefficient Data for C.I. Direct Red 81

waves was parallel to the position of the film and (2) the microtip was operated at 50% power due to power handling limitations.

Further laboratory work using the same method has been done with various mechanical configurations in which the ultrasound has been presented to the substrate (Figure 2-6). In this

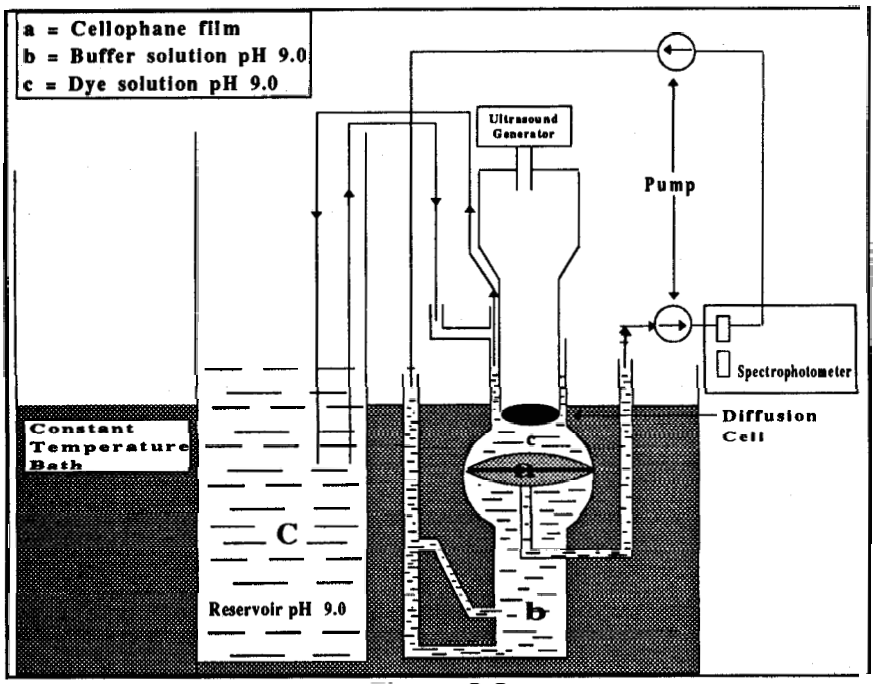


Figure 2-6
Steady State Diffusion Coefficient
of C.I. Direct Orange 15

work, a low molecular weight dye, C.I. Direct Orange 15 was studied at different temperatures. Results are shown in Table 2-1.

Table 2-1
Time Lag, Diffusion and Permeability Coefficient
Data for C.I. Direct Orange 15

Temperature (°C)	Time Lag (seconds)	Apparent Diffusion Coefficient ($cm^2/sec \times 10^{-10}$)	Permeability Coefficient ($mg/sec \times 10^{-5}$)
45 C	10974	2.80	6.69
UL	7704	5.41	8.59
60 C	7855	5.30	5.94
UL	6330	6.58	9.14
75 C	6714	6.21	7.17
UL	5423	7.68	12.29

In the first configuration, the direction of ultrasound waves was parallel to the position of the film. In this study, the film was positioned perpendicular to the direction of waves so that they impinged directly on the film. In this configuration, the increase in the diffusion coefficient was found to be temperature dependent and showed an increase from 30% to 200%. The greater increases occurred at lower temperature. This was a general trend in all this work, *i.e.*, when a process is very slow, (*e.g.*, diffusion of dye through a polymer film at low temperature) the application of ultrasound makes a greater difference. The effects of ultrasound are much less pronounced when the process is rapid.

The activation energy of dyeing was calculated from the diffusion data for C.I. Direct Orange 15. The relationship between the rate constant and the activation energy is given by the Arrhenius equation:

$$\log D = \log A - \frac{E_a}{RT} \qquad (3)$$

where D = rate constant
A = frequency factor
E_a = activation energy of dyeing
R = gas constant
and T = absolute temperature.

Apparent activation energy "E_a" was determined from the plot of log D vs. 1/T, and values are given in Figure 2-7 for the films treated with and without ultrasound. Activation energy decreased 24% during insonated dyeings.

Reactivity of Fiber Reactive Dyes

Fiber reactive dyes are the most commonly used class of dye applied to cellulosic fibers. In an alkaline medium, the dye reacts with cellulose, forming a strong bond, but it also tends to react with water forming hydrolyzed dye. Hydrolyzed dye goes to waste, and it is necessary to know the rate of formulation and the amount of hydrolyzed dye in a given process. A critical factor in the design of the fiber reactive dyeing process is the reactivity of the dye. Consequently, the knowledge of the effect of ultrasound on the reactivity and hydrolysis rates is essential for developing an ultrasound assisted dyeing process.

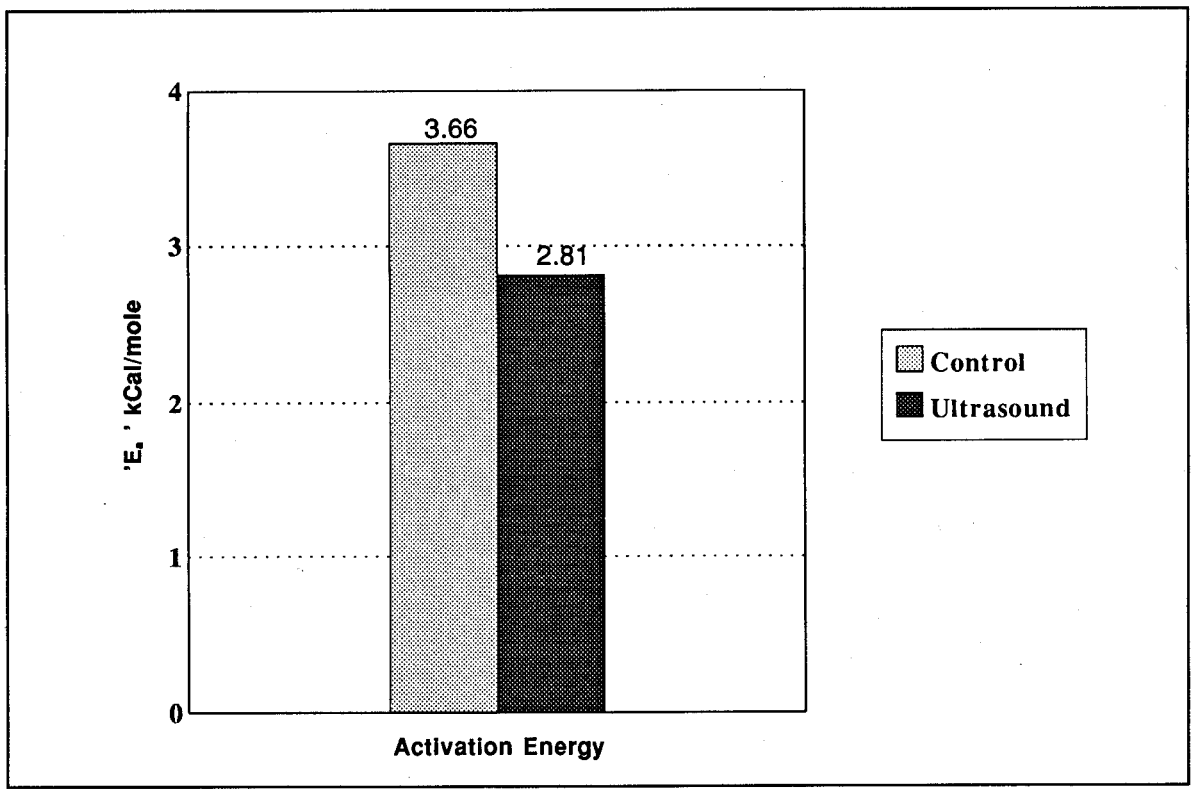


Figure 2-7
Activation Energy of C.I. Direct Orange 15
for Cellulose Film

Investigations were carried out to determine the effects of ultrasound on the kinetics of vinyl sulfone fiber reactive dye hydrolysis, in particular the hydrolysis of C.I. Reactive Violet 5. This set of experiments was a screening test to determine what effect ultrasound would have on the reactivity of fiber reactive dyes. Some of the factors which influence the efficiency and rate of fixation or fiber reactive dyes on textile substrates include:

- standard affinity ($-\Delta\mu^{\circ}$),
- hydrolysis reaction rate constant (k'_{OH}),
- diffusion coefficient (D), and
- ratio of reaction rates with water (hydrolysis) compared to cellulose (fixation) (R_f).

In general, these are factors not under the dyer's control. It is known that higher affinity dyes have lower diffusion coefficients. Ultrasound appears to be able to increase the diffusion coefficient without affecting affinity. This is fundamentally different than the effect of raising the temperature, by which the diffusion coefficient is increased but affinity is lowered. This is

important in terms of fiber reactive dye fixation, for which it is desirable to have both high diffusion coefficient and high affinity. Having direct control of this is an important new degree of control for a dyer.

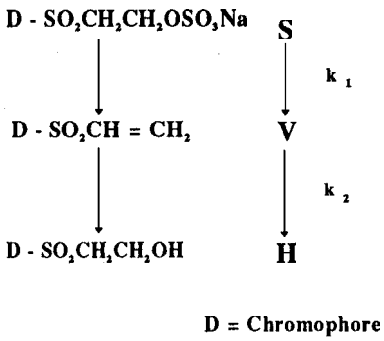
The reaction rate of dyes increased when ultrasound was introduced, but did not determine what happened to the ratio of reaction rates with cellulose and water (R_f).

$$R_f = \frac{k_{Cell^-}}{k_{OH^-}} \qquad (4)$$

To study this, one would react fiber reactive dyes with homogeneous water/alcohol mixtures to determine R_f for several alcohols, which are analogous to the polysaccharide cellulose. Time did not permit us to do these experiments, but this is an interesting topic for future work (if any).

In the preliminary study of reactivity, the dye used was a commercial sample of C.I. Reactive Violet 5 used without further purification. Reactions were performed in a one-liter glass beaker with and without ultrasound. Ultrasound energy was produced by a Heat Systems Ultrasonic Generator Model W-1200, delivered through a ¾-inch diameter standard tip liquid processor immersed in the solution. The frequency of waves was 40 kHz and the power supplied to the tip was approximately 480 watts. To avoid bulk heating from the ultrasound, the temperature was maintained by circulating water through a copper coil placed in a constant temperature bath set at 60°C. The reaction mixture was analyzed on a Waters HPLC using a 990 photodiode array detector. This instrument uses a 600E multisolvent delivery system and Waters C18 column as the stationary phase. Dye solutions were prepared by dissolving 0.1 gram of dye in one liter of buffer solution of 0.05*M* Na_2CO_3 + 0.1*M* NaOH (pH 10.5 at 60°C).

Solutions were brought to 60°C and mixed, resulting in the following reaction sequence:



Aliquots were withdrawn from the reaction mixture at various times and immediately mixed with stoichiometricly equivalent amounts of ice cold HCl to halt the reaction. These neutralized samples (pH 6.8) were eluted as soon as possible using the gradient system shown in Table 2-2.

Table 2-2
Gradient System

Time (min)	Flow (mL/min)	A	B
Initial	1.5	30	70
5.0	1.5	50	50
7.0	1.5	30	70

(Helium sparge)

The mobile phase of the system consisted of Solvents A and B. Solvent A was 100% acetonitrile containing 0.025*M* tetrabutyl ammonium bromide (TBAB) as an ion pairing agent. Solvent B was a 30/70 mixture of acetonitrile containing 0.025 *M* TBAB and deionized water containing 0.05*M* ammonium dihydrogen phosphate. The above gradient system gave excellent separation of vinyl and ß-sulfatoethyl peaks. Eluting the samples as described above gave chromatograms as shown in Figures 2-8 and 2-9. Retention times for the various components of the mixture are given in Table 2-3. The first peak, a minor component in the dye (which does not participate

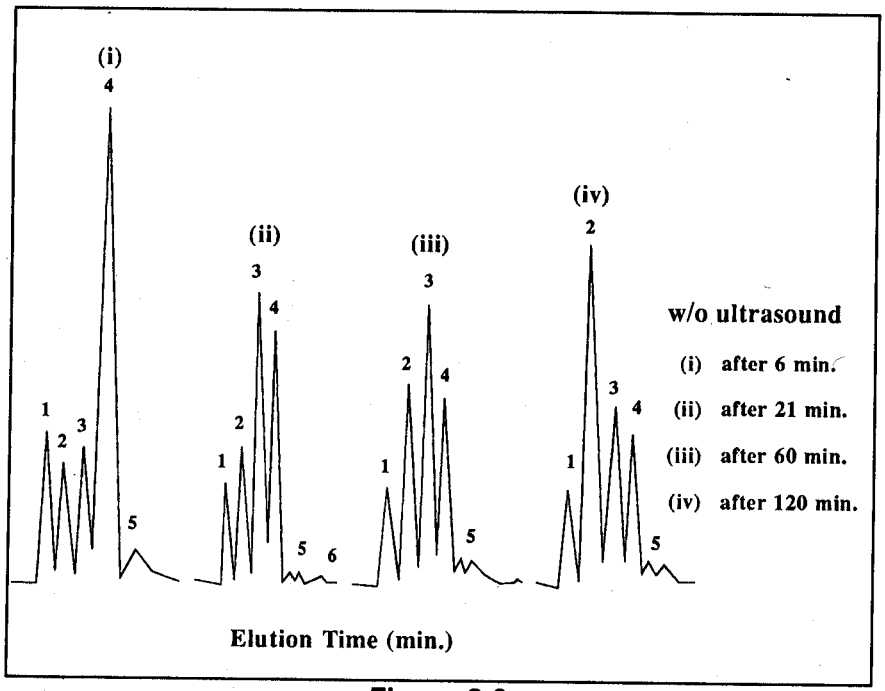


Figure 2-8
Chromatogram of C.I. Reactive Violet 5
Reaction Carried out at 60C and at pH 10.5 without Ultrasound

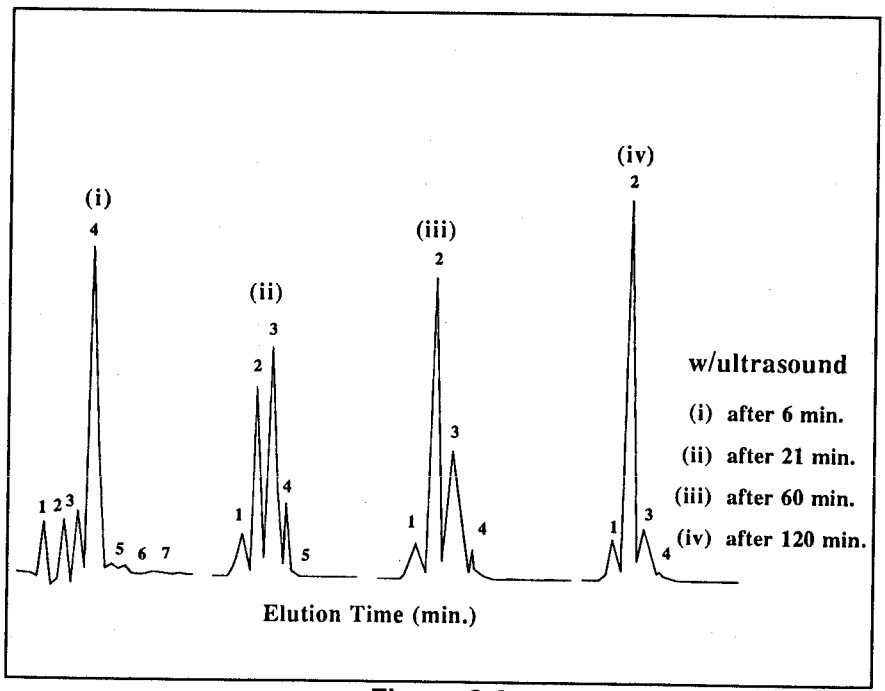


Figure 2-9
Chromatogram of C.I. Reactive Violet 5
Reaction Carried out at 60C and at pH 10.5 with Ultrasound

in the reaction), was monitored as an internal reference to verify the consistency and uniformity of data.

Table 2-3
HPLC Elution Data for Vinyl Sulfone Dyes

Peak Number	Identification	Retention Time (min)
1	A Minor nonreactive component	0.98
2	ß-hydroxyethyl	1.45
3	Vinyl	1.75
4	ß-sulfatoethyl	2.26
5, etc.	Other non participating minor components	3.43

Figure 2-10 shows the data obtained from the outset of reaction ($t \rightarrow 0$), at which time the reaction rate law can be written:

$$\frac{d[S]}{dt} = -k'_1[S] \qquad (5)$$

where [S] is the concentration of the ß-sulfatoethyl form of the dye, k'_1 is the pseudo first order reaction rate constant for conversion of sulfatoethyl to vinyl and *t* is time. At later times, the two reactions which affect the concentration of the vinyl form are:

$$S \xrightarrow{k'_1} V \xrightarrow{k'_2} H$$

where *S* is the ß-sulfatoethyl form, *V* is the vinyl form and *H* is the ß-hydroxyethyl (hydrolyzed) form. This gives a rate law:

$$\frac{d[V]}{dt} = k'_1[S] - k'_2[V] \qquad (6)$$

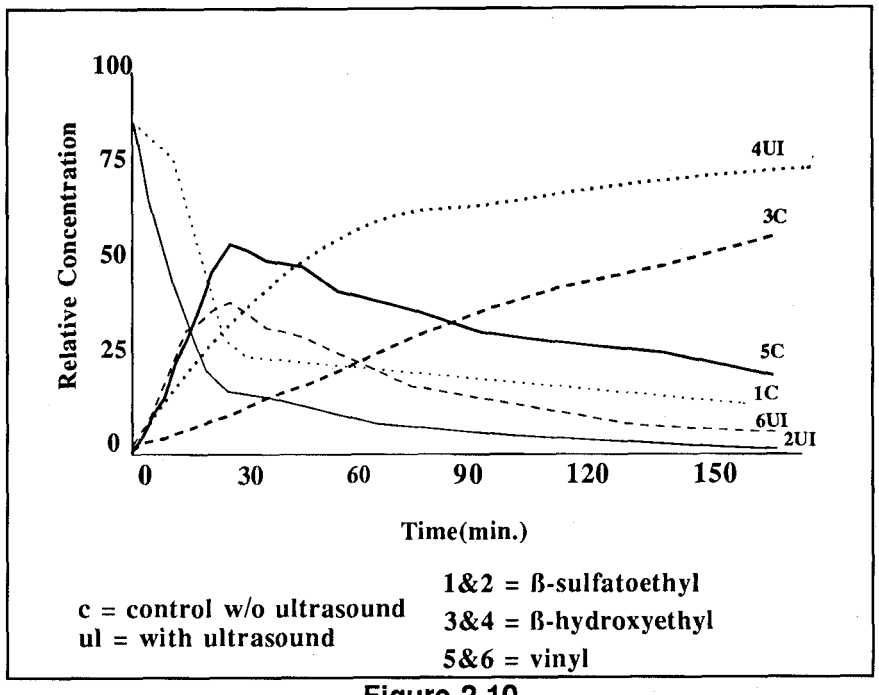


Figure 2-10
Hydrolysis Kinetics of C.I. Reactive Violet 5

where k'_2 is the pseudo first order reaction rate constant for conversion of vinyl to ß-hydroxyethyl form. Due to the stoichiometry involved (*i.e.*, low concentration of $SO_4^=$ and high concentration of OH^- and H_2O) the reverse reaction:

$$S \xrightarrow{k'_{-1}} V$$

and

$$V \xrightarrow{k'_{-2}} H$$

are considered negligible in this analysis. To determine k'_1, the above equation can be integrated to produce:

$$\ln\left(\frac{S_o}{[S]}\right) = k'_1 t \qquad (t \to 0) \qquad (7)$$

where S_o is the concentration of the ß-sulfatoethyl form at time zero, and t is time. Thus plots or regressions of $\ln(S_o/[S])$ *versus* time give values of k'_1 as the limiting slope as $t \to 0$ (Figure 2-11). Values of k'_1 were determined by ordinary linear regression to be 0.0490 ± 0.003 min^{-1} without ultrasound and 0.0746 ± 0.004 min^{-1} with ultrasound. The regression has r-values of 0.99 in each case.

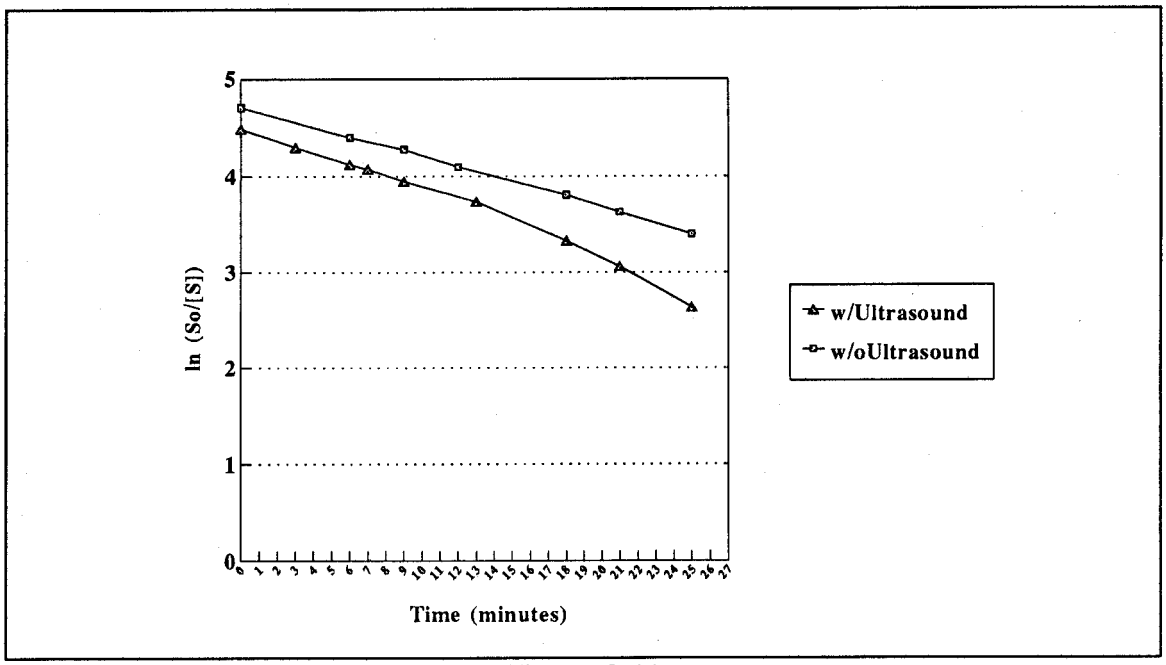


Figure 2-11
Plots of ln (S_o/[S]) Versus Time

To determine k'_2, the rate equation was used in its differential form when d[V]/dt = 0, *i.e.* at about 30 minutes. At that time, the intermediate vinyl form is at a maximum where

$$\frac{d[V]}{dt} = 0 = k'_1[S] - k'_2[V] \qquad (8)$$

The value of k'_2 can be found to be

$$k'_2 = \frac{[S]}{[V]} k' \qquad (9)$$

This gives values for k'_2 of 0.0112 ± 0.00195 min^{-1} without ultrasound and $0.0622 \pm .00105$ min^{-1} with ultrasound.

The results show that ultrasound increases the reaction rate both for conversion of ß-sulfatoethyl to vinyl and for hydrolysis of vinyl to ß-hydroxyethyl form of C.I. Reactive Violet 5. The rate constant for vinyl formation is increased by about 50% whereas the rate constant for hydrolysis is increased by well over 500%. This increase was observed in a homogenous system.

In the dyeing of cellulosic fibers in an alkaline medium, the formation of vinyl sulfone occurs so rapidly that this reaction has no effect on the dyeing speed. The reaction with cellulose, on the other hand, takes place much more slowly. The vinyl form of the dye reacts preferentially with the hydroxyl group of cellulose to that of water. However, hydrolysis of dye does take place. The dye-cellulose reaction may be speeded up by ultrasound as the latter is known to have marked accelerating effects at the interphases in heterogeneous systems. [6]

This study suggests that the optimization of fiber reactive dyeing procedures in terms of time, temperature and chemical additions may be quite different when ultrasound is used compared to conventional methods. The role of temperature and perhaps salt will certainly be different. Dyers will undoubtedly gain a new degree of control over the fixation of fiber reactive dyes by the use of ultrasound.

Dyeing Studies

Fiber Reactives on Cotton

100% cotton bleached fabric was dyed with Remaxol Blue BB (C.I. Reactive Blue 17) by the following method and conditions:

M:L:		1:50
Sodium chloride:		10 g/L
Alkali-	Sodium Carbonate:	10 g/L
	Sodium Hydroxide:	2 g/L
Volume:		0.5 L
Final Dyeing Temperature:		60°C
% Shade owf		1 and 4.5

The fabric was introduced into a dyebath at 40°C and dyed for 5 minutes. Then salt was added in two portions and dyeing was continued for 10 minutes with slow heating to 60°C. Then alkali was added and dyeing was continued for about 30 minutes. These dyeings were done with and without ultrasound and in one dyeing a booster horn (2.5 : 1) was used. The dyed samples were rinsed with cold water followed by hot and cold washings.

Color measurement was done on Macbeth 2020+ spectrophotometer. Results are shown in Table 2-4.

Table 2-4
Color Strength Analysis of Fiber Reactive Dyes on Cotton With and Without the Use of Ultrasound

Exp. No.	% Shade	λmax (nm)	k/s		Relative % Difference (gain due to ultrasound)
			Control	Ultrasound	
1	1	620	0.91	1.55	170
2	1 (Booster Horn 2.5 : 1)	620	.91	1.89	208
3	4.5:1	620	6.35	8.46	133

These results show a substantial increase in the color strength and is further increased by booster horn. The power density with the standard tip used in all experiments was about 18 watts/inch^2.

Nylon Acid Dyes

The most significant increase in dye absorption with ultrasound was noticed on nylon yarn dyed with Acid dyes. The color strength difference (Table 2-5) indicates an increase in the depth of the shade by as high as 300% at 75°C with ultrasound. These data show that for the equivalent amount of dye on the fiber, either the dyeing time can be reduced by 66% or the temperature could be lowered by 20°C using ultrasound at the 60 watts/in^2 power level. The light fastness ratings were the same for dyeings carried out with and without ultrasound. However, the yarn dyed without ultrasound showed a change in color on fastness testing. In this respect, the ultrasound dyeing was rated better.

Table 2-5
Color Strength Analysis for C.I. Acid Red 1
on Nylon (λmax 540 nm)

Exp. No.	Temperature (°C)	Dwell Time (seconds)	K/S		
			Control	Ultrasound	% Difference
15	55	57	3.10	4.75	153
16	65	37	3.55	5.97	168
17	75	37	4.78	14.17	196
18	80	48	0.79	0.92	116

Disperse on Polyester

Dyeing of polyester was carried out on 100% Dacron fabric (supplied by Test Fabrics, Inc.) with Resolin Orange F3RN (C.I. Disperse Orange 25 supplied by Ciba-Geigy Corp) dye by the following method and conditions:

Dye Concentration: 4 g/L

Liquor Rage: 50:1

Temperature: 90°C

Dyeing Time: 1 min. and 5 min.

pH: 5.0 (adjusted with acetic acid)

The short dyeing times were selected to correspond with the capabilities of our pilot plant prototype.

The dyebath was set at 90° and polyester fabric was dyed for 1 and 5 minutes with and without ultrasound. In one experiment a booster horn (2.5 : 1) was used. The dyed fabric was given reduction clearing treatment with 4 g/L sodium hydroxide and 2 g/L sodium hydrosulphite at 65°C for 15 minutes. These samples were rinsed out and thoroughly washed with cold and hot water. Color measurements were taken for each sample and the mean values are graphed in Figure 2-12.

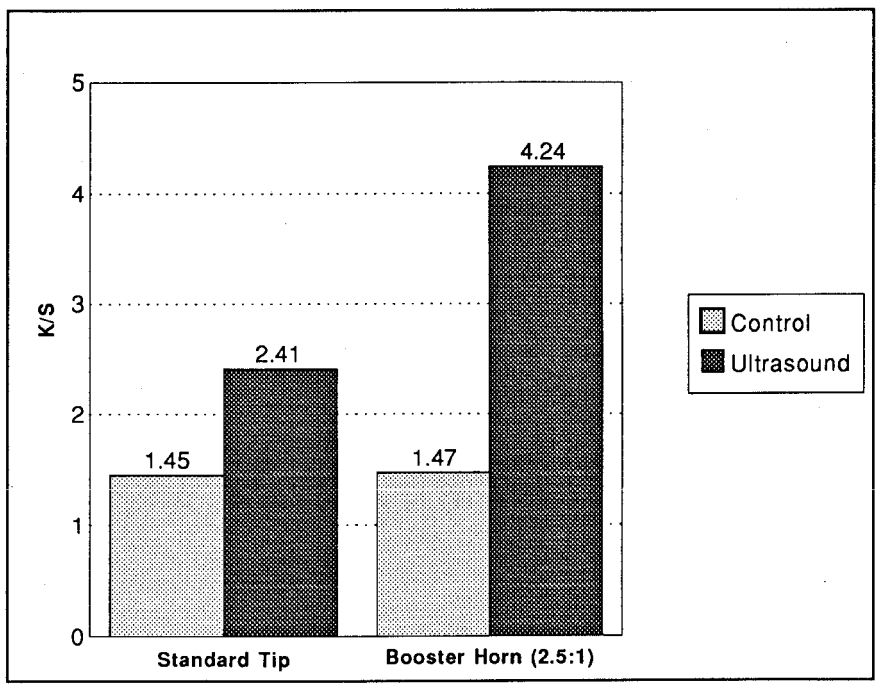


Figure 2-12
Color Strength Analysis for C.I. Disperse Orange 25 on Polyester With and Without the Use of Ultrasound

The color strength increased with increased ultrasound power levels. A 288% increase in color was obtained with a 2.5:1 ultrasound booster horn.

After each dyeing, the substrate was inspected for signs of fiber damage. Previous work, done before March 1990, showed no strength loss or other damage to the substrate. To further confirm this, differential scanning colorimetry was performed on several nylon samples exposed to ultrasound. No significant differences were found in control versus insonated fibers.

Summary

In general, ultrasound was found to: 1) greatly enhance dye diffusion without affecting affinity, 2) increase the reactivity of fiber reactive dyes, and 3) increases the reaction rate both for conversion of ß-sulfatoethyl to vinyl and for hydrolysis of vinyl to ß-hydroxyethyl form of C.I. Reactive Violet 5. The rate constant for vinyl formation is increased by about 50% whereas the rate constant for hydrolysis is increased by well over 500%. This increase was observed in a homogenous system.

A general trend in all dyeing studies was that when a process is very slow, (e.g., diffusion or dye through polymer film at low temperature) the application of ultrasound makes a greater difference. The effects of ultrasound are much less when the process is rapid.

The most significant increase in dye absorption with ultrasound was noticed on nylon yarn dyed with Acid dyes. Ultrasound increased the depth of the shade by as high as 300% at 75°C. For the equivalent amount of dye on the fiber, either the dyeing time can be reduced by 66% or the temperature could be lowered by 20°C using ultrasound at the 60 watts/in^2 power level. The light fastness ratings were the same for dyeings carried out with and without ultrasound. No fiber damage was ever observed when using ultrasound.

Section 3
PILOT PLANT EXPERIMENTAL DETAILS

Introduction

The pilot plant studies included the continuous dyeing of yarn and fabrics as well as washing off of fiber reactive dyed fabrics. Initial efforts were concentrated in optimizing the conditions for dyeing of various dye-fiber systems, optimum distance between the transducer surface and the substrate, synchronizing the speed of the main drive, the padder, and take-up devices, and setting up a prototype dyeing machine with control system.

Ultrasound Tank Initial Design and Construction

One of the main activities of this project was the construction of a pilot plant scale prototype ultrasound dyeing machine and control system. The physical components include the tank, the transport system, and the components used to control and monitor this system.

The heart of this dyeing system is the 150 liter ultrasonic tank. This tank is made by Blackstone Corporation and is designed to clean small metal parts. Thermostatic control is used to vary the temperature up to 250 degrees Fahrenheit. Four ultrasonic generators are located underneath the bottom of the tank. These generators produce an ultrasonic frequency of 43 KHz. The ultrasonic power is 3200 watts when all generators are operating at maximum. The power is adjustable from 0% (no power) to 100% (full power).

The transport system is fabricated from stainless steel and ultra-high molecular weight (UHMW) plastics. Large rollers are turned in unison to move the yarn or fabric through the dye bath (Figure 3-1 and Figure 3-2). A reed guides the yarn in the correct path around those rollers. The reed is removable to allow fabric to pass through the tank. A frame holds the rollers and reed together and allows the transport system to be easily inserted and removed from the tank. All of these parts are made of stainless steel, so they will not rust or corrode, which could contaminate the dye bath. The bearing surfaces for the rollers and the chain used to turn the rollers are made of the UHMW plastic, which creates a low friction surface that does not require lubricants.

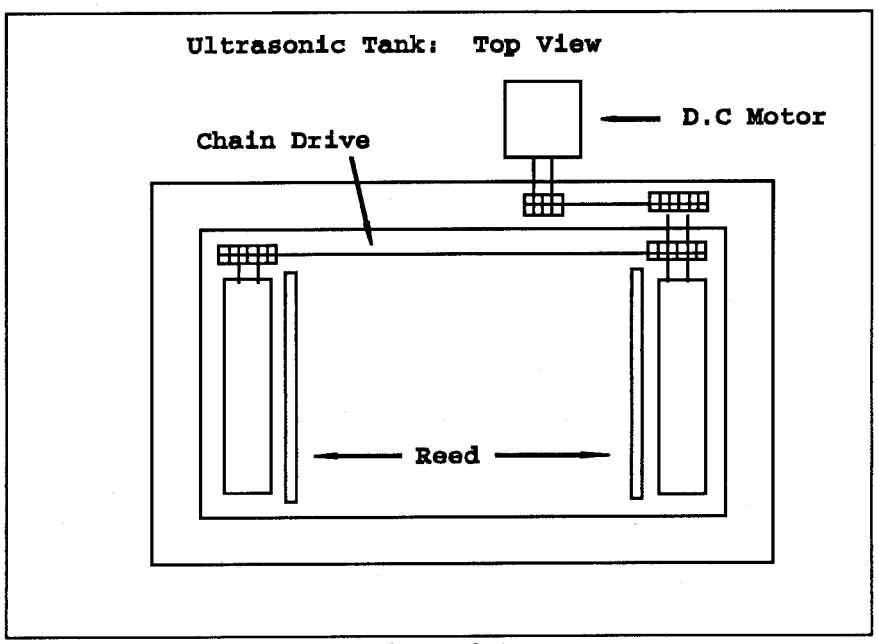


Figure 3-1
Roller and Drive System in the Ultrasound Tank

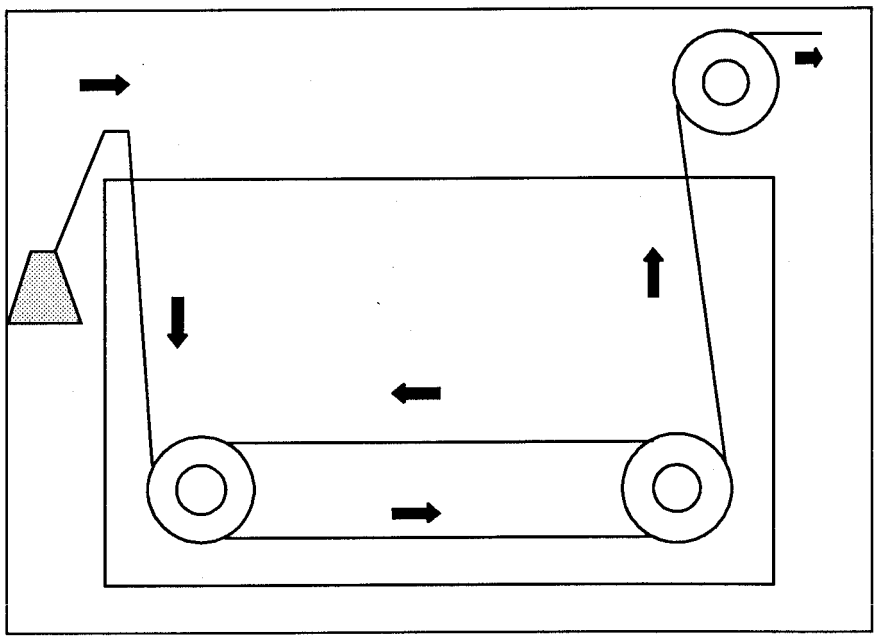


Figure 3-2
Yarn Flow through the Ultrasound Tank

A washing station and windup station were installed to collect the yarn after it is dyed as illustrated in Figure 3-3. The washing station is driven by a motor synchronized with the drive motor of the yarn delivery system in the tank. After the yarn is rinsed and squeezed, the

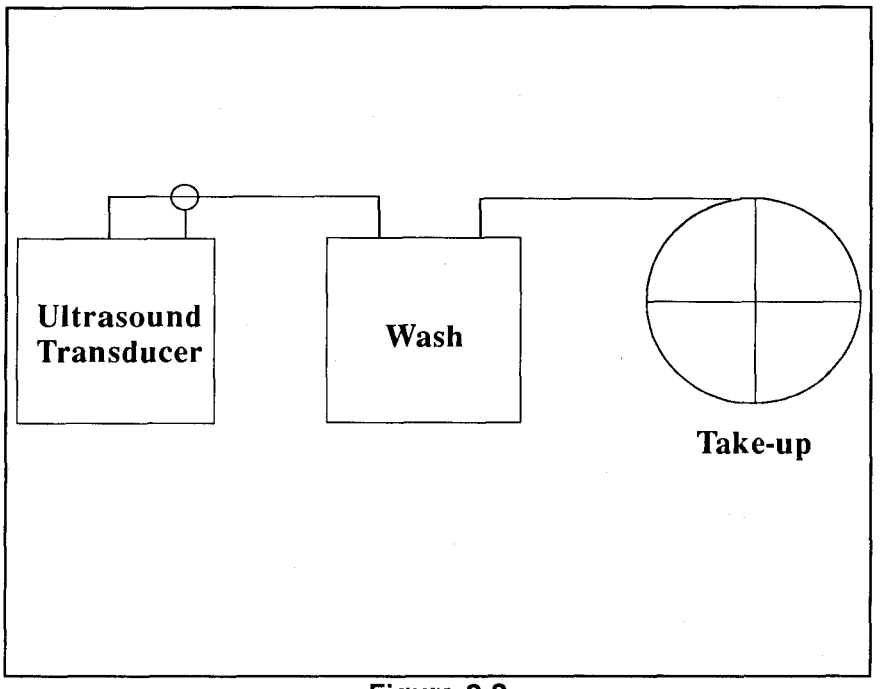


Figure 3-3
Washing and Windup after Processing in an Ultrasound Tank

yarn is passed under an infrared heater and wound onto a constant torque winder.

The ultrasound dyeing tank control system hardware, which controls operating conditions, is composed of four basic components: an IBM industrial personal computer, a digital Optomux unit, an analog Optomux unit, and a power supply. These Optomux units were developed by Opto 22. The first three of these components form a network with the Optomux units operating as slave devices to the PC. The PC serves as the host computer. The power supply is needed to provide DC power to each Optomux device. The digital and analog Optomux units both consist of a removable brain board and an I/O mounting rack. The digital units have the capability to monitor and control on/off devices while the analog units have the capability to monitor and control analog devices. Analog devices differ from digital devices in that the process is continuous, and it varies over a range. Digital devices are either on or off.

The brain board component of the Optomux unit has a microprocessor which handles communications between the host computer and the mounting racks. The mounting racks accept plug-in I/O modules that form the interface between external devices and the control

system. The plug-in modules are one of two types. Input modules monitor a device, and output modules control a device. Any combination of input or output modules may be mounted on an Optomux unit; however, digital and analog modules cannot be combined on the same mounting rack. Only digital modules can be installed in a digital mounting rack, and only analog modules can be installed in an analog mounting rack.

Communications between the Optomux units and the host computer occur over an RS422/485 serial communications link. The RS422/485 link allows for long distances between the host computer and the Optomux units. Although great distances are possible between the host and each Optomux unit, all components in this system are mounted in one cabinet that is designed specifically for holding computer equipment.

Evaluation of the Original Prototype

Several factors in the design and operation of the prototype are critical to the performance in terms of dyeing. These include:

- power level,
- presentation of substrate to transducers,
- temperature,
- dye, salt, alkali, and other chemical concentration, and
- dwell time.

The effects of each of these factors were studied to determine optimum values for various dye/fiber systems.

Distance Between Transducer and Substrate

The distance between the transducer surface and the substrate is an important criterion which influences the efficiency of sound treatment. This parameter is crucial, depending on the physical form of substrate (*e.g.*, yarn or fabric). In dyeing fabric, a distance of $\lambda/2$, λ, $3/2\lambda$, etc. (where λ = the wavelength, approximately 3.7cm to 5.4cm) resulted in patchy or no dyeing because of concentrated energy or node points. If fabric is close to the transducer surface the waves are damped because fabric acts as a barrier to wave propagation. In the case of yarn dyeing, however, such effects are not observed because of spacing between the yarns and also because the design of the transport system provides a distance greater than λ. In the qualitative analysis of dyed fabric, a distance of $5/4\lambda$ (4.7cm to 6.7cm at 40kHz and 26kHz, respectively)

gives more uniform dyeing than other distances. Therefore, 5/4λ may be considered optimum for fabric dyeing.

Parameters and Materials Studied

Details of dyes and fabric/yarn used in the experiments are given in Table 3-1.

Table 3-2 gives an account of initial dyeing trials conducted to optimize temperature, dwell time, power level and distance between transducer and substrate.

Table 3-1
Details of Material Studied Using the Pilot Plant Ultrasound Tank

Dyes: Trade Name	Generic Name	Supplier
1. Erio Floxine GN	C.I. Acid Red 1	Ciba-Geigy
2. Neolan Flavine E-3G	C.I. Acid Yellow 17	Ciba-Geigy
3. Resolin Orange F3RN	C.I. Disperse Orange 25	Mobay Corp.
4. Pergasol Red 3BAN	C.I. Direct Red 80	Ciba-Geigy
5. Remazol Blue BB	C.I. Reactive Blue 27	Hoechst
Yarn/Fabric: Type	**Construction Details**	**Supplier**
1. Nylon (flat) filament	200/32 Nylon 6,6	BASF
2. Polyester	Homopolymer	Test Fabric, Inc.
3. Nylon Fabric		Test Fabric, Inc.
4. Cotton (bleached)	28/1, 28/2 and 28/4	West Point Pepperell
5. Cotton Fabric Woven, bleached		Test Fabric, Inc.

Table 3-2
Details of Dyeing Studies
Prototype Pilot Plant Unit - Original Configuration

Dye/Substrate	Conditions					ATCC Fastness	Remarks
	Temp. (°C)	Dwell Time	pH	Dye Concn (L)	Liquor Volume (L)		
Dir. Red 81/ Cotton Yarn 4 ply	15	15 min.	9.0	5.8	142	Wash Crock Light	Dyed yarn washed with saturated salt solutions
"	15	30 min.	9.0	5.8	142		
"	55	7.5 min.	9.0	5.8	142		
"	55	15 min.	9.0	5.8	142		
"	40	3.5 min.	9.0	6	150		Slight apparent color strength after knitting and washing
"		5 min.	9.0	6	150		
"	70	3.5 min.	9.0	6	150		
"	80	1 min.	9.1	5	150		
Disperse Orange 25/ polyester yarn	72	3 min.	5.1	4	150		Increase in color depth using ultrasound, but washed out after clearing and washing. Slight tinting with ultrasound.
2"	85	1.5 min.	5.0	3.5	120		
"	85	2.5 min.	5.0	3.5	120		
"	80	4.5 min.	5.0	3.5	120		
	85	3.2 min.	5.0	3.5	120		
Acid Red 1/Nylon Yarn	40	57 sec.	4.5	4.0	120		Slight tinting after washing.
	55	57 sec.	4.5	4.0	120	Light	
	65	37 sec.	4.5	4.0	120	Light	
	75	37 sec.	4.5	4.0	120	Light	
Acid Red 1/Nylon Fabric (unset, spun).	80	48 sec.	4.5	3.5	120	Light	

The dwell time was controlled by varying the drive speed and/or number of loops (passes) of yarn. To maintain a uniform level of dye liquor, the tank was covered with polyethylene film to minimize heat and vapor loss due to evaporation at high temperatures. Dyeing was carried out with and without ultrasound in each case. Depending upon the experimental conditions, the dyed yarn was washed either as it came out of the tank on a bench scale winch dyeing machine with continuous water spray or in skein form after winding on a skein winder. The yarn was then knitted and washed. The dyed samples were subjected to color measurement on Macbeth Spectrophotometer and tested for fastness properties.

Cotton/Direct Dyes

Bleached cotton yarn was dyed with C.I. Direct Red 80 and Red 81. Experimental details are as follows for the Red 80 dyeing:

Dye:	3.5 g/L C.I. Direct Red 80	
Substrate:	Bleached cotton yarn	28^5 count single 28^5 count/2 ply 28^5 count/4 ply
Sodium Chloride:	10 g/L	
Temperatures:	70°C and 90°C	

After the yarn was dyed, it was washed on a padder with a continuous water spray, squeezed and wound on a take-up device. The fabric knitted from the dyed yarn was washed, dried and tested for color value and fastness properties.

Ultrasound increased the color depth on cotton yarn with Direct Red 81 by 13% (Table 3-3). The fastness properties of knitted fabric dyed with and without ultrasound were comparable (Table 3-4).

Table 3-3
Color Strength Analysis for
C.I. Direct Red 81 on Cotton (λmax 520nm)

K/S		Percent Difference
Control	Ultrasound	
1.52	1.72	113

Table 3-4: Fastness Data of Cotton Dyed with C.I. Direct Red 80

A. CROCK FASTNESS (AATCC TEST METHOD #8-1981)

Dyeing	Gray Scale Rating	
	Wet	Dry
Without Ultrasound	3	3.5
With Ultrasound	3.5 - 4	> 4

B. COLORFASTNESS TO LAUNDERING
(AATCC TEST METHOD 61-1986)

Dyeing	Gray Scale Ratings for Staining
Without Ultrasound	3.5 - 4
With Ultrasound	> 4

Polyester

In polyester dyeing for instance, the difference in the depth of the shade after dyeing and before washing was significant, which indicates increased dye transfer from the ultrasonic dyebath to the product. The dye on the surface could be fixed by increasing the ultrasonic energy in the bath or by other methods, e.g., thermosol or radiation treatment.

In one study, 100% polyester fabric samples were dyed with and without ultrasound and passed through an IR dryer at 200°C, with a dwell time of 30 seconds. The fabric dyed with ultrasound had a 200% increase in color. Similar results were obtained when dyed fabrics were fixed on a metal frame and subjected to 200°C temperature for about 40 seconds in a Despatch oven.

A combination of these two electrotechnologies, ultrasound dyeing and IR drying, opens a new era of dyeing polyester at atmospheric pressure and at comparatively lower temperatures than those used in conventional methods.

Modified Ultrasound Tank

The amount of power used in the lab studies was about 16 watts per square inch of exposed area, at which power polyester could be dyed at 90°C without the use of carrier or chemical assistance. The power density was only 4 watts/in^2 in the pilot plant ultrasound tank. The tank was upgraded to about 6 watts/in^2 by adding three sets of transducers, Figure 3-4, each rated at 600 watts, to the top of the tank. This setup provided an total additional 1800 watts of power to the bath. The drive motors, controllers, and take-up system were also improved.

The total power was increased from 3200 watts to 5000 watts by installing three immersible transducers (IM-12) manufactured by Blackstone Ultrasonics. These transducers were configured in the tank as shown in Figure 3-4. The yarn or fabric passes between the bottom of the tank and the three transducers. This configuration provides a much more intense energy

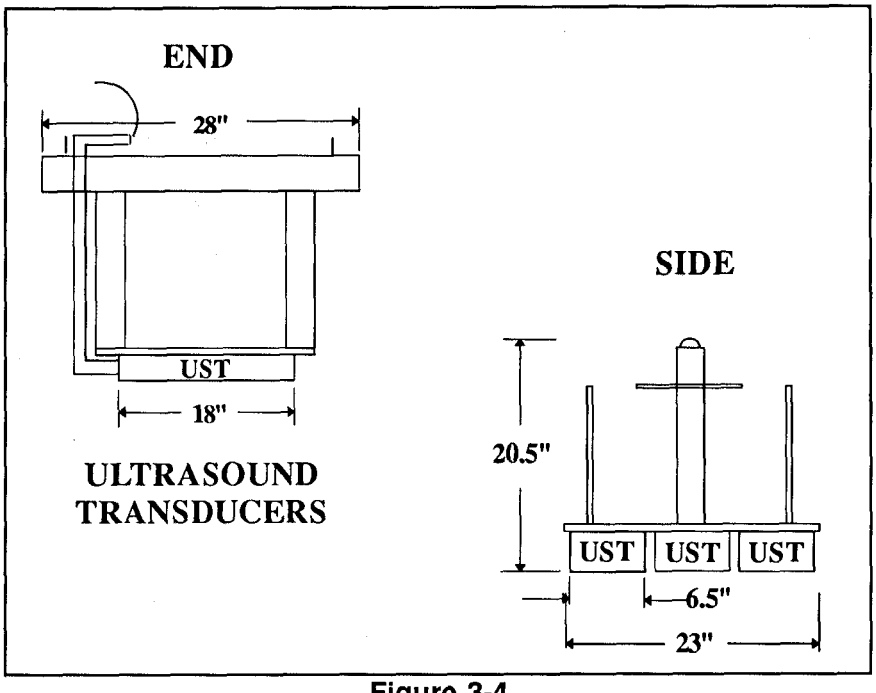


Figure 3-4
Schematic Diagram of Additional Ultrasound Transducers Added to the Ultrasound Tank

field.

Figure 3-5 shows a schematic diagram of the improved machine in which two transducer sets face each other. Yarn or fabric can be passed between the transducers at a specific distance from each transducer.

Studies on the Modified Ultrasound Tank

The previously described pilot plant work was all done on the pilot plant ultrasound tank in its original (3200 watts) configuration. After upgrading to a full 5 kW power, the following work was done.

Dyeing of Nylon with C.I. Acid Yellow 17 and Red 1

Dyeing studies were carried out on nylon yarn with two dyes: 1) C.I. Acids Red 1, and 2) Yellow 17. Three dyeings were done varying the dyebath temperature, dwell time and with and without ultrasound. In an attempt to simulate the conditions of a continuous dyeing process, the dyed yarn was squeezed, washed on a padder and dried on a winding device. The dyed yarn was knitted, and the knitted fabric was thoroughly washed and evaluated for color yield and fastness properties. K/S strength difference data for Yellow 17 and Red 1 are given in Tables 3-5 and 3-6 respectively. A few ultrasound dyeings with Red 1 showed higher reflectance in the

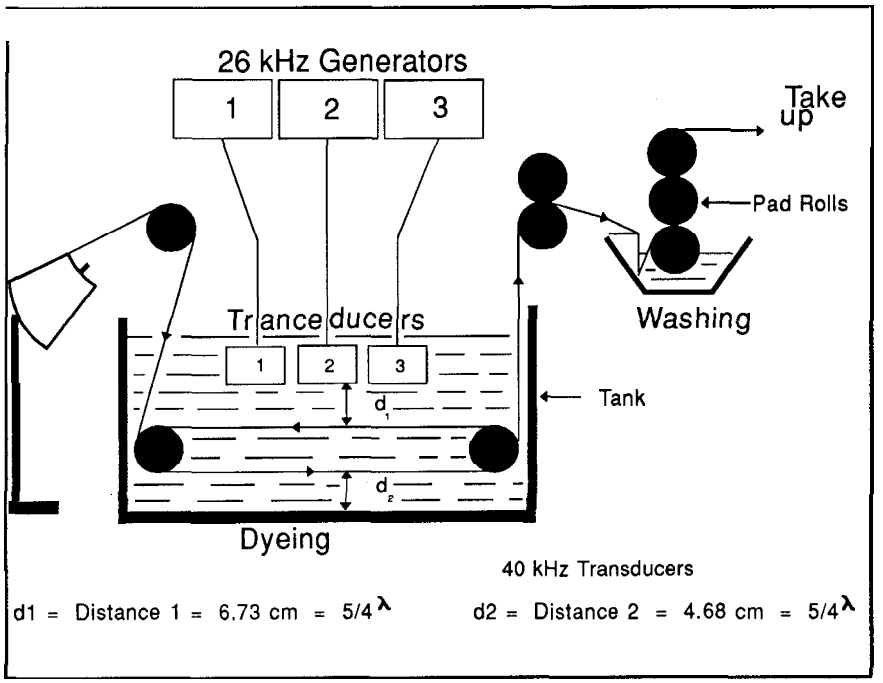


Figure 3-5
Schematic Diagram of Modified Ultrasonic Tank

red region. However, the wavelength of maximum absorption was equal in both cases. Integrated k/s values gave almost the same percent difference as calculated at λ_{max}.

Table 3-5
Color Strength Analysis for
C.I. Acid Yellow 17 on Nylon (λ max 540 nm)
Dye Concentration: 3 g/L
Dyebath Volume: 150 L

Temperature °C	Dwell Time (seconds)	Yarn Speed (yds/min)	K/S		
			Control	Ultrasound	% Diff.
90	165	8.2	24.3	27.3	112
90	65	19.0	19.2	23.9	125
90	32	41.0	13.8	20.6	149
70	165	8.2	18.7	22.9	122
70	65	19.0	15.9	20.7	130
70	32	41.0	11.2	16.2	145

Table 3-6
Color Strength Analysis for
C.I. Acid Red 1 on Nylon (λmax 420 nm)
Dye Concentration: 3 g/L
Dyebath Volume: 150 L

Temperature (°C)	Dwell Time (seconds)	Yarn Speed (yds/min)	K/S		
			Control	Ultrasound	% Diff.
90	165	8.2	22.3	25.9	116
90	65	19.0	17.7	22.8	129
90	32	41	10.2	13.9	136
70	165	8.2	16.8	21.8	130
70	65	19.0	11.0	18.4	167
70	32	41.0	3.5	7.6	218
50	165	8.2	2.9	3.45	119
50	65	19.0	1.4	2.21	157
50	32	41.0	0.69	1.21	174

These results show that ultrasound increased the color yield in all dyeing and in all conditions. The maximum increase in color yield, 118%, occurred using C.I. Acid Red 1 on nylon (λmax 420nm) at a dwell time of 32 seconds and a temperature of 70°C. The minimum increase in color yield, 12%, occurred using C.I. Acid Yellow on nylon (λmax 540nm) at a dwell time of 2.75 minutes and a temperature of 90°C.

The dyed samples were tested for light and wash fastness properties. Tests results are given in Tables 3-7 and 3-8:

Table 3-7
Lightfastness Data (AATCC Test Method 16E - Xenon Arc)
Exposure: 20 Hours

Substrate	Rating	
	Control	Ultrasound
Nylon Fabric (Woven-Unset)	3-4	4
Nylon - Knitted	4	4

Table 3-8
Fastness Evaluation - Acid Dyes on Nylon

Dye Conditions (Acid Red 1)	Product Evaluation Features	
	Light	Wash
Red 1 - Control, 90°C, 165 sec	pass 20 hrs.	4
Red 1 - Ultrasound, 90°C, 165 sec	pass 20 hrs.	2
Red 1 - Control, 90°C, 65 sec	pass 20 hrs.	4
Red 1 - Ultrasound, 90°C, 65 sec	pass 20 hrs.	2-3
Red 1 - Control, 90°C, 32 sec	pass 20 hrs.	4
Red 1 - Ultrasound, 90°C, 32 sec	pass 20 hrs	2-3
Red 1 - Control, 70°C, 165 sec	pass 20 hrs	2-3
Red 1 - Ultrasound, 70°C, 165 sec	pass 20 hrs	2
Red 1 - Control, 70°C, 32 sec	pass 20 hrs	2
Red 1 - Ultrasound, 70°C, 32 sec	pass 20 hrs	1-2
Yellow 17 - Control, 90°C, 165 sec	pass 20 hrs	2-3
Yellow 17 - Ultrasound, 90°C, 165 sec	pass 20 hrs	2-3
Yellow 17 - Control, 90°C, 165 sec	pass 20 hrs	2-3
Yellow 17 - Ultrasound, 90°C, 165 sec	pass 20 hrs	3
Yellow 17 - Control, 90°C, 32 sec	pass 20 hrs	4-5
Yellow 17 - Ultrasound, 90°C, 32 sec	pass 20 hrs	4
Yellow 17 - Control, 70°C, 165 sec	pass 20 hrs	4
Yellow 17 - Ultrasound, 70°C, 165 Sec	pass 20 hrs	3-4
Yellow 17 - Control, 70°C, 32 sec	pass 20 hrs	3
Yellow 17 - Ultrasound, 70°C, 32 sec	pass 20 hrs	2-3

All of the material dyed with and without ultrasound passed 20 hour light fastness. In general, the control dyeings gave a better wash fastness than the material dyed using ultrasound. This result was expected because the product dyed without ultrasound had very little dye on them, so it took only a few washes to wash off the excess dye. In fact, the ultrasound dyed product was 30 to 120% darker in color than the control.

As in the lab scale experiments, the magnitude of increase is depended on the temperature, dwell time (or yarn speed) and dye. Maximum difference in color yield between yarn dyed with and without ultrasound was observed at high yarn speed (short dwell time). As the dwell time increased, the depth difference decreased.

One interesting observation in dyeing with Red 1 was the ability of ultrasound to cover minor irregularities in the yarn quality. In all dyeing, the yarn which was dyed without ultrasound had white spot appearances at intervals which were prominently seen on the knitted fabric. The sample dyed with ultrasound was uniform in appearance. The cause of the irregularities was unknown but thought to be knitting marks.

Washing of Reactive Dyed Fabrics

Washing off studies were carried out on 100% cotton fabrics dyed with C.I. Reactive Blue 17 by a pad batch method. The dyed fabric was washed with and without ultrasound in the tank.

Pad liquor recipe:

Dye:	3 g/L Remaxol Blue BB (C.I. Reactive Blue 17)
Urea:	50 g/L
Wetting agent:	3.75 g/L
Sodium Silicate:	180 g/L (42°Be)
Caustic Soda:	3.75 g/L (50% Solution)
% Pick Up:	65
Batch Time:	21 hours

Dye and alkali solutions were prepared separately and mixed in a padding trough. Fabric was padded at 65% wet pick up and batched for 21 hours.

Washing was carried out with fabric stationary in the tank. A 15 inch by 15 inch piece of fabric was fixed on a frame and washed for a period of time (0.5 to 16 minutes). While washing with ultrasound, two configurations were used: 1) only one side of the fabric received ultrasound waves, and 2) the fabric was sandwiched between two transducer sets (one above and the other below the fabric).

Each experiment was carried out with the fabric being in the tank, maintaining a distance of 1 1/4" (corresponding to 5/4 λ) from the bottom surface of the transducer. Both experiments were also carried out at room temperature (25°C) without the use of wetting agents or detergent. The wash liquor was continually drained with simultaneous addition of fresh water to maintain the level in the tank. Washed samples were squeezed through a padder and wrapped with a polyethylene sheet to avoid fabric drying.

Fabric swatches approximately 4 inches by 4 inches were taken for soxhlet extraction from each washed sample for the determination of unfixed dye not removed in washing. Extracts were collected, diluted to a constant volume and their absorbencies were measured on a Perkin-Elmer double beam spectrophotometer. Figures 3-6 and 3-7 depict the washing efficiency with and without the use of ultrasound.

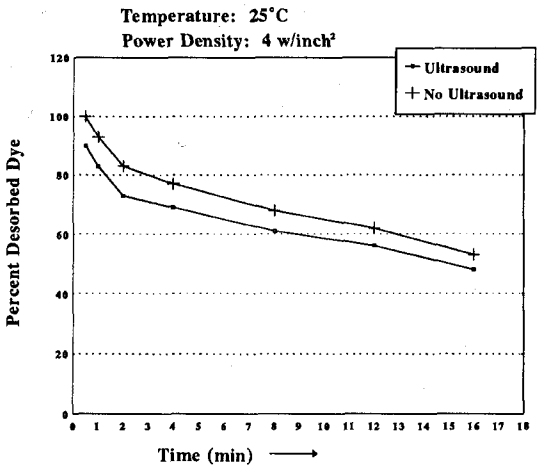


Figure 3-6
Washing Off of Reactive Dyed Fabric
Single Set Transducers (Bottom)

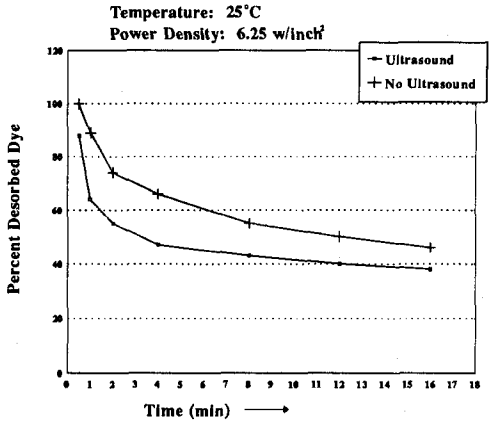


Figure 3-7
Washing Off of Reactive Dyed Fabric
Sandwich Transducers
(Top and Bottom)

In Figure 3-6, where only one set of transducers was used, the power density was only 4 watts/in^2; the washing off was accelerated only marginally. However, when ultrasound was applied to both sides of the fabric (power density of 6.25 watts/in^2), the process was accelerated considerably. In fact, washing time was reduced by approximately 60%.

Note that unlike industrial washing machines, the fabric was stationary in this test. In an industrial washing machine, the mobile fabric causes turbulence in the water, and a water spray can be used to increase the washing effectiveness.

While this data indicates beneficial effects of ultrasound in washing off of reactive dyed fabrics in terms of increasing productivity, the need to study ultrasound washing on a full scale industrial washer is vital. This aspect of investigation has been considered and is a candidate for future work.

Summary

In the qualitative analysis of dyed fabric, a distance of 5/4λ (4.7cm to 6.7cm at 40kHz and 26kHz, respectively) gave uniform dyeing and increased color depth more than placement at other distances. Therefore, 5/4λ may be considered optimum for fabric dyeing.

Ultrasonic energy increases the mass transfer onto the surface of the substrate. This inference was drawn from the polyester dyeing carried out using the tank with and without ultrasound, and half the fabric samples were washed immediately, whereas the other half were thermofixed. There was a substantial difference in color yield in the samples which were thermofixed after dyeing but without washing. This suggests that while ultrasound increases the transfer of dye from the bath onto the surface of the fiber or yarn, the energy in the original tank is not adequate to diffuse the dye inside the fiber. This may be achieved either by increasing the power level in the tank or by fixing the dye.

One substantial limitation of the prototype was its power limit of 3200 watts in an area of 24 X 36 inches or about 3.7 watts/in^2. This power level was inadequate to accomplish desired results. Thus, an additional 1800 watts was added which provided a power level of about 6 watts/in^2, which is still low too a power level. Future work needs to be done with power capabilities in the 25 to 50 watt/in^2 range, or about 20 to 40 kW in the tank (compared to the present 5 kW).

By using ultrasound to wash reactive dye off of a stationary fabric, washing time can be reduced by over 60 percent. Ultrasound must be placed into an industrial washing machine to adequately compare this result to conventional washing. Savings may result in increased productivity, decreased water consumption, and decreased steam consumption if the washing is accomplished with hot water.

Section 4

CONCLUSION
&
SCOPE FOR FURTHER WORK

The main objective of this work was to develop a beneficial modification of textile wet processes by the use of ultrasonic waves. A literature review showed that the work in this area over the past fifty years has enough promise for application to commercial scale processes. Benefits may include saving in energy, reduction in consumption of chemicals and/or dyes, reduction in process time, improvement in quality and easier process control. Among all wet processes, application to the dyeing process seems to be most advantageous because it currently is the most expensive process. Washing being the least expensive process, the use of ultrasound may or may not be advantageous in terms of saving money.

On a laboratory scale, the fundamental effects and technological aspects of applying ultrasonic energy to the dyeing process were studied. The main effects studied were dye diffusion rates and reactivity of fiber reactive dyes. The effects of ultrasound on the diffusion of direct dyes in cellulose film was studied using commercial samples of Pyrazol Fas Red 8 BL (Direct Red 81) and Pargasol Orange 2R (C.I. Direct Orange 15). The experiment used an ultrasonic microtip operating at a frequency of 20 kHz and at a distance of one inch. The direction of propagation of waves was parallel to the position of the cellophane film and the microtip operated at 50% power (600 watts). Using this configuration and Direct Red 81, an approximate increase of 30% in diffusion and 310% in permeability was obtained with the use of ultrasound. C.I. Direct Orange 15 was studied at different temperatures and the cellophane film was positioned perpendicular to the direction of waves. The increase in the diffusion coefficient was found to be temperature dependent and showed an increase from 30% to 200%. The greater increases occurred at lower temperatures.

Investigation were carried out to determine the effects of ultrasound on the kinetics of vinyl sulphone fiber reactive dye hydrolysis of C.I. Reactive Violet 5. The results show that ultrasound increases the reaction rate both for conversion of β-sulfatoethyl to vinyl and for hydrolysis of vinyl to β-hydroxyethyl form of C.I. Reactive Violet 5. The rate constant for vinyl formation is

increased by approximately 50% whereas the rate constant for hydrolysis is increased by well over 500%.

Results of dyeing trials done in a beaker using a boaster horn operating at a frequency of 20 kHz showed an increase in dye absorption on nylon yarn dyed with acid dyes. Ultrasound increased the depth of shade by as high as 300% at 75°C. For the equivalent amount of dye on the fiber, either the dyeing time can be reduced by 66% or the temperature could be lowered by 20°C using ultrasound at the 60 watts/in^2 power level. The light fastness ratings were the same for dyeings carried out with and without ultrasound. No fiber damage was ever observed when using ultrasound.

The pilot plant studies included the continuous dyeing of yarn and fabrics as well as washing off of fiber reactive dyed fabrics. Pilot plant studies were performed in a modified ultrasound cleaning tank. One substantial limitation of the prototype was its power limit of 3200 watts in an area of 24 by 36 inches, or about 3.7 watts/in^2. This power level is inadequate to accomplish as good of results that were obtained in laboratory tests using beakers and an ultrasound probe. Even with an additional 1800 watts of ultrasound energy added to the tank, the power level was only raised to 6 watts/in^2.

In the qualitative analysis of dyed fabric, a distance of 5/4λ (4.7cm to 6.7cm at 40kHz and 26kHz, respectively) gave uniform dyeing and increase color depth more than placement at other distances. Therefore, 5/4λ may be considered optimum for fabric dyeing.

Dyeing tests done in the ultrasound tank showed that ultrasound increased the color yield in all dyeing and in all conditions. The maximum increase in color yield, 118%, occurred using C.I. Acid Red 1 on nylon (λmax of 420nm) at a dwell time of 32 seconds and a temperature of 70°C. The minimum increase in color yield, 12%, occurred using C.I. Acid Yellow on nylon (λmax of 540nm) at a dwell time of 2.75 minutes and a temperature of 90°C.

Ultrasound can be used to aid in washing reactive dye off of a stationary fabric. With the aid of ultrasound, washing time can be reduced by over 60 percent. To adequately compare this result to conventional washing, ultrasound must be placed into an industrial washing machine. Savings may result in increased productivity, decreased water consumption and decreased steam consumption.

Research in the use of ultrasound to enhance textile processes continues at North Carolina State University with funding from the National Textile Center, North Carolina Alternative Energy Corporation, and the Electric Power Research Institute. Three major tasks have been outlined.

1) Enhance Ultrasound Application Technology - Applicator technical requirements shall be specified using data collected from past work. A prototype ultrasound unit shall be fabricated and the applicators shall be installed at the University.

2) Laboratory Process Testing - Tests will be performed to determine the following parameters for wetting, dyeing, and washing: a) power level sensitivity, b) minimum and maximum power requirements, c) bath residence time, d)water temperature requirements, and e) investigate parameters affecting product quality.

3) Cost/Benefit Investigation - Economic savings potential shall be quantified based on laboratory testing utilizing actual plant process data and computer models, where applicable, including: a) energy, b) pollution, c) throughput, d) linespeeds, e) reject or rework levels, and f) raw material usage.

Section 5

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