Cotton Preparation with Alkaline Pectinase: An Environmental Advance

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Cotton remains the international fiber of choice among the world's expanding population, with as much as 42 billion pounds of this fiber consumed annually in textile use.[1] There are, however, severe environmental costs associated with the widespread use of cotton. Unlike manmade cellulosic fibers such as rayon or lyocell, cotton must be thoroughly prepared for subsequent wet-processing treatments such as mercerizing, bleaching, dyeing, printing, or finishing. The purpose of the preparation step is to make the textile substrate uniformly absorbent, and the traditional method to achieve such absorbency is to scour the substrate with strong solutions of sodium hydroxide at elevated temperatures. Such scouring results in significant contributions to the effluent on a worldwide basis. Rößner notes that about "75% of the organic pollutant level arising from textile finishing is derived from the preparation of cotton goods."[2] It is useful, therefore, to discuss an alternative method that is commercially viable and cost-effective for preparing cotton fiber substrates. Such a method is based on the use of alkaline pectinase, a newly discovered enzyme.[3]

ABSTRACT

The replacement of environmentally harsh sodium hydroxide by alkaline pectinase in cotton preparation has the potential to revolutionize wet processing internationally. Using this enzyme decreases both effluent load and preparation costs. The mechanism of enzymatic preparation and dyeing characteristics of the resulting cotton are discussed.

Key Terms

Cotton
Dyeing
Enzymes
Pectinase
Preparation

Primary Wall

The cotton fiber primary wall is responsible for the lack of water absorbency of the fiber when no wetting agents are included in the various aqueous processing baths. This primary wall is illustrated schematically by the cut-out diagram given in Fig. 1.[4]

According to a summary given by BASF, the primary wall is about 0.1 microns thick and comprises only about 1% of the total thickness of cotton fiber.[5] This thin wall consists of about 52% cellulose in which 12% pectins, 7% wax, 12% proteins, 3% ash, and 14% other organic compounds are dispersed. The exact percentage of each component of the primary wall is determined by the type of the cotton plant, its origin, the growth conditions, and the degree of maturity.[5] The wax in the primary wall is a source of the hydrophobic nature of unscoured cotton. This wax consists of fatty alcohols, fatty acids, their esters, cholesterin, and other hydrocarbons. In addition to waxes, insoluble calcium, magnesium, iron, and other salts of polygalacturonic acids (pectins) contribute to the hydrophobic nature of unscoured cotton fiber. These pectate salts also act as biological glue, binding the noncellulosic components of the primary wall within the cellulosic matrix. Fig. 1 shows that the noncellulosic components of the primary wall need not be uniformly distributed in the cross-section of the wall. In fact, it is more likely that the concentration of these components increases as the outer zone of the wall is approached. This outer zone is often referred to as a cuticle, although it is clear that the cuticle of a cotton fiber does not resemble the thin continuous film of wax that is found on the surface of many leaves, stems, and fruits of other plants.

Caustic Scouring

Traditional scouring of cotton involves the use of strong solutions of sodium hydroxide that also contain wetting, dispersing, and chelating agents. These strong alkaline solutions are applied at high temperatures, sometimes above the boiling point of water. Such solutions attack the primary wall matrix and result in a very high removal of the noncellulosic components. However, unless the process is carefully controlled, the cotton fiber may be damaged during the scouring process by the formation of oxycellulose. Most cotton that is subjected to caustic scouring develops a harsh hand due to removal of most of the lubricating wax from the fiber.

In addition to the negative environmental impact that caustic scouring causes, much time and water are needed to rinse sodium hydroxide from the cotton substrate. In fact, the usual practice is to neutralize the alkali in the substrate

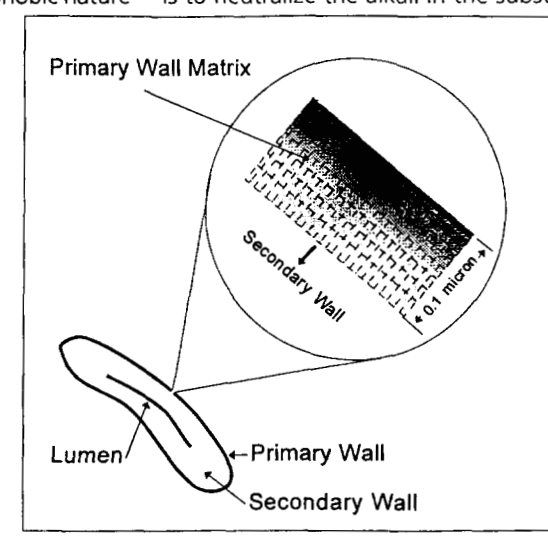

Fig. 1. Cotton fiber primary wall matrix, illustrating the concentration gradient of noncellulosic material within the cellulose matrix.[4]

by adding acetic acid to the rinse water. Sodium acetate is formed by the neutralization reaction, and if this compound is not completely rinsed out, fabric yellowing can result during high-temperature drying or curing. The presence of sodium acetate can also have a negative influence on print thickening agents, leading to less crisp detail in print designs.

Textile Enzyme Use

Several pertinent reviews of the use of enzymes in textile wet processing are available in the literature.[6-11] In fact, the publication of papers dealing with the application of enzymes in textiles appears to be accelerating each year. Bach and Schollmeyer, however, were perhaps the first to report the effect of pectinase enzymes in biopreparation.[12,13] Rößner, another researcher, conducted a series of experiments on the influence of various hydrolases, including pectinase, on the absorbency and other properties of cotton.[2,14]

In the United States, much of the early published research on biopreparation was in conjunction with the Ph.D. dissertation of Yonghua Li, under the direction of Ian Hardin.[15-17] The work indicated that a synergism could occur if mixtures of cellulase and pectinase were used in biopreparation. It must be noted, however, that since the enzymes used in the research were not pure, it is possible that it was the pectinase impurity in the cellulase enzyme that was responsible for the improved absorbency when the enzyme was applied at low concentrations. When pure cellulase is applied at high concentrations, increased absorbency will occur due to the destruction of cellulose in not only the primary wall, but also the secondary wall, leading to significant weight and strength loss.

It should be remembered that enzymes are nothing more than biological catalysts. These catalysts are large globular molecules that do not easily diffuse into cotton fiber, but are capable of accelerating reactions at the fiber surface. In fact, enzymes cannot cause a reaction to occur that normally would not occur. It is useful, therefore, to examine the possible enzymatic mechanisms involved in hydrolysis of cotton fiber catalyzed by both cellulase and pectinase.

Enzymatic Hydrolysis

Cellulase

Although cellulase is beneficial for biopolishing fabric to achieve a smooth appearance or for treating denim fabric to simulate the stonewashed look, cellulase is not the best choice for biopreparation. As shown in Fig. 2, when cotton fiber is treated with an aqueous solution of cellulase, the enzyme is adsorbed onto the fiber surface to form a primary wall/cellulase complex. At the interface, hydrolysis of the cellulose occurs very rapidly, releasing decomposition cellulose products and also noncellulosic components which are dispersed within the primary wall matrix. The process is repeated over and over at an extremely rapid rate, producing pathways in the primary wall that permit the enzyme to reach and accelerate hydrolysis of the cellulose that constitutes the very substance of the cotton fiber—the secondary wall. It is clear that enzymatic destruction of cotton by cellulase will result in an absorbent fiber, but at the expense of much weight and strength loss.

Alkaline Pectinase

An extremely powerful alkaline pectinase (patent pending) has been isolated by scientists at Novo Nordisk.[3] This enzyme is now being produced in volume and reduced to commercial use in biopreparation on a worldwide basis.[4,18,19] As shown in Fig. 3, the major benefit of this enzyme in biopreparation is that it does not destroy the cellulose of the cotton fiber. The enzyme is a pectate lyase that very rapidly catalyzes hydrolysis of salts of polygalacturonic acids (pectins) in the primary wall matrix. The term alkaline pectinase is used to describe the enzyme because it is used under mildly alkaline conditions that are very beneficial in preparation processes. It is known that alkaline conditions inactivate other pectinases. As the biological glue is hydrolyzed, the other noncellulosic components of the primary wall are released and dissolved, dispersed, or emulsified by the surfactants and mild chelating agents in the biopreparation bath. The result of biopreparation with alkaline pectinase is that the cellulose is not degraded, so less weight or strength loss occurs than with either caustic scouring or cellulase treat-

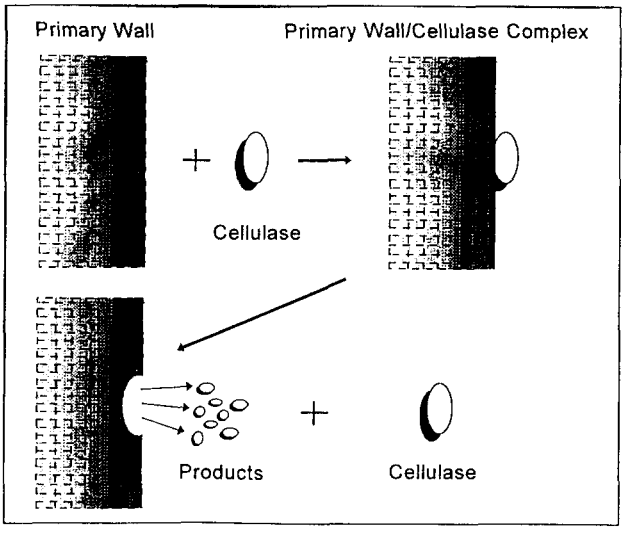

Fig. 2. Schematic of enzymatic hydrolysis of cotton fiber cellulose that is catalyzed by cellulase.

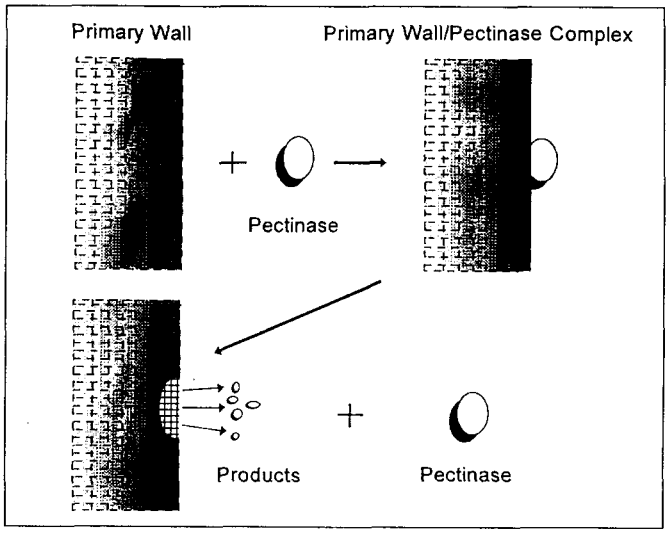

Fig. 3. Schematic of enzymatic hydrolysis of pectin in the cotton fiber primary wall that is catalyzed by alkaline pectinase.

ment. In addition, the amount of wax removed is less than that removed by the other processes, which results in an improved hand.

Experimental

It is helpful to compare the wicking and dyeing characteristics of cotton fabric that has been prepared by caustic scouring and by enzymatic preparation with alkaline pectinase. To obtain data in these two areas, the following experiment was conducted.

Desizing

Carded 100% cotton army sateen greige fabric, with a weight of 242 g/m^2 (Test Fabrics Inc.), was desized with 1.0% owb Thermozyme 280L, a high-temperature amylase enzyme, supplied by Novo Nordisk. The desizing bath contained the surfactant auxiliaries Tergitol 15-S-12, 1.0%, supplied by Union Carbide, and Geropon SS-0/75, 0.1%, supplied by Rhône-Poulenc. The pH of the desizing bath was adjusted to 6.0 with the use of 0.10M monosodium phosphate. Desizing was conducted at 90C in a Werner Mathis pad steam range, model PSA, using a desize incubation time of 30 minutes.

Conventional Preparation

A solution of 2.0% NaOH (50% solution) and 0.2% owb Tergitol 15-S-12 was padded onto the desized fabric to a wet pickup of 85%. The fabric was then incubated for 60 minutes at 100C in a Werner Mathis pad steam range, model PSA, followed by rinsing in the rinsing chambers of the range.

Biopreparation

A padbath, consisting of a 0.055% owb BioPrep 3000L, an alkaline pectinase supplied by Novo Nordisk, and 0.2% owb Tergitol 15-S-12, was buffered to pH 9.5 with 0.05M disodium phosphate. The biopreparation solution was padded on the fabric to a pickup level of 85%, and the fabric was incubated for 60 minutes at 55C in a Werner Mathis pad steam range, model PSA, followed by rinsing in the rinsing chambers of the range.

Wicking Test

Strips of fabric (45 cm x 2.5 cm) that had been prepared by the two methods were suspended vertically in the warp direction with a 35-gram clamp attached to one end of the strips. The fabric strips were then immersed in a bath, containing 1 g/L of a direct dye and 0.5 g/L of sodium dioctyl sulfosuccinate wetting agent at room temperature. Wicking height up the fabric from the surface of the bath at 0.5 cm intervals and corresponding times were recorded. A diagram of the wicking apparatus is shown in Fig. 4.

Dyeing Procedure

The caustic scoured and the bioprepared fabrics were dyed at a 40/1 liquor ratio in an Ahiba Texomat dyeing machine with four different commercial direct dyes: Red 9, Yellow 106, Blue 25, and Green 26. The concentration of each dye was 4.0% on the weight of the fabric, the dyeing temperature was 90C, the dyeing time was 60 minutes, and the salt concentration was 20 g/L. After the fabrics had been dyed and dried, reflectance readings were taken for each fabric, using a Macbeth spectrophotometer. The reflectance value obtained for each dyeing at the wavelength of minimum reflectance was converted to *K/S* to obtain a measure of color depth.

Results

Wicking

No difference in water sorption was observed between fabric strips prepared by caustic scouring or by biopreparation. The rate of movement of the water front was the same in the case of both fabrics. However, in the case of the direct dye solutions, an interesting phenomenon was observed for all dyes. The wicking rate of the dyes on bioprepared fabric was greater than that for caustic scoured fabric (Fig. 5).

The difference in the wicking rates of the dyes on the two substrates can be explained as substantivity differences and is analogous to the differences found in traditional thin layer chromatography. These data suggest that, in the absence of salt in the dye solutions, the substantivity of the dyes studied is lower on the bioprepared fabric. The reason for this can be found in the higher concentration of beneficial wax that remains on bioprepared fabric. The higher amount of wax results in a more hydrophobic substrate for which direct dye has less thermodynamic affinity, at least in the absence of salt. A question to answer

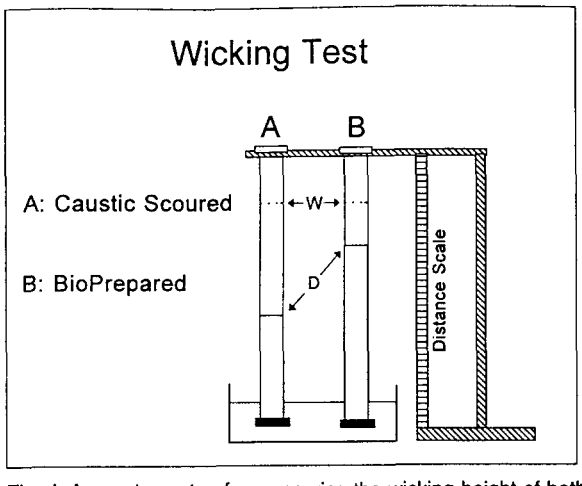

Fig. 4. Apparatus setup for measuring the wicking height of both water and direct dye on caustic scoured and bioprepared fabric. *W* refers to the water wicking distance and *D* is the dye wicking distance at a given time.

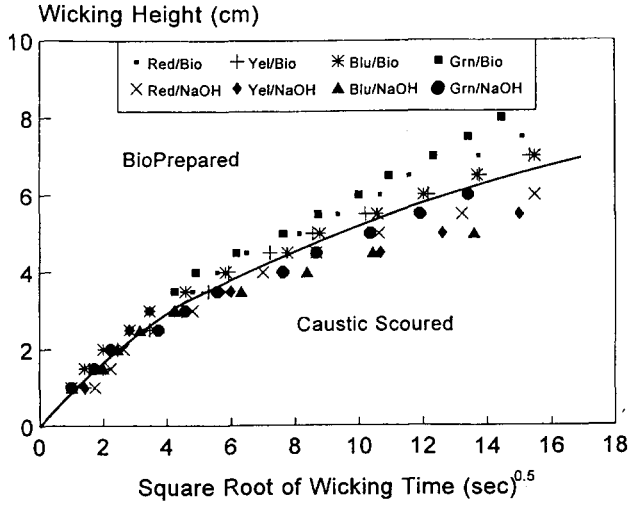

Fig. 5. Rate of wicking of direct dyes on caustic scoured and on bioprepared fabric. The solid line represents the division of wicking rates between dyes on the two substrates.

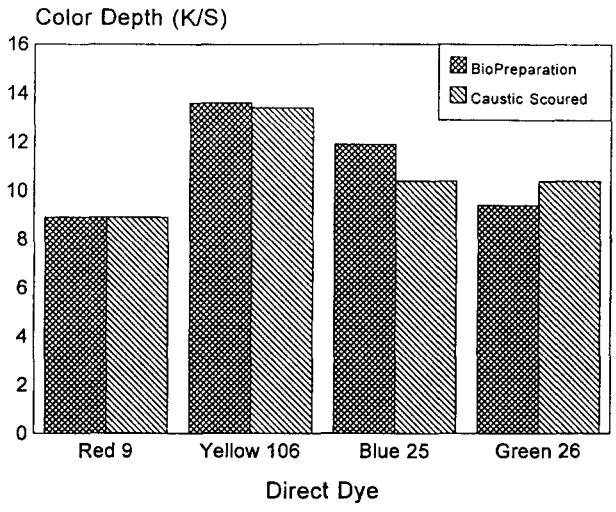

Fig. 6. Color depth expressed in terms of *K/S* for four direct dyes on caustic scoured and bioprepared cotton fabric.

is—does the initial substantivity result in less color yield in an actual dyeing in which sodium chloride is included in the dyebath? The answer to this question is given in Fig. 6.

Color Depth

As shown in Fig. 6, no significant differences in color depth for caustic scoured and bioprepared fabric are apparent. Although small differences are observed in the case of Direct Blue 25 and Direct Green 26, it is believed that the differences may be within the range of experimental variation.

Uniformity

The results of the wicking and color depth data suggest that commercial dyeing of bioprepared fabric may be more consistently uniform than that obtained with caustic scoured fabric. The reason for this speculation is the probability that the dye will become more easily and quickly distributed in the capillary network of bioprepared fabric. After salt is added to the dyebath, the dye that is uniformly distributed throughout the textile mass will tend to exhaust uniformly. The improved dyeing uniformity that is possible with bioprepared fabric is an area that deserves further experimental substantiation.

Concluding Remarks

When a new preparation process can be shown to result in greatly reduced effluent load, reduced preparation costs, improved fabric softness, and possibly improved uniform dyeing consistency, the process deserves close attention from the international textile wet-processing community. It is likely that preparation of cotton with alkaline pectinase will soon become the preparation method of choice worldwide.

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