

# Dyeing Properties of Novel Regenerated Cellulosic Fibers

Joonseok Koh,<sup>1</sup> Ik Soo Kim,<sup>2</sup> Sung Soo Kim,<sup>3</sup> Woo Sub Shim,<sup>3</sup> Jae Pil Kim,<sup>3</sup> Seung Yeop Kwak,<sup>3</sup> Sang Wook Chun,<sup>3</sup> Yong Ku Kwon<sup>4</sup>

<sup>1</sup>T&F Research Team, Fiber and Textile, R&D Center, Hyosung Corporation, Anyang, Kyonggi-Do 431-080, South Korea

<sup>2</sup>enVix R&D Team, SK Chemicals, Suwon, Kyonggi-Do 440-745, South Korea

<sup>3</sup>School of Materials Science and Engineering, College of Engineering, Seoul National University, Seoul 151-742, South Korea

<sup>4</sup>Department of Polymer Science and Engineering, Division of Chemical Science and Biological Engineering, Inha University, Incheon 402-751, South Korea

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**ABSTRACT:** Two types of direct dyes (disazo and phthalocyanine) were applied to regular viscose rayon and a new regenerated cellulosic fiber (enVix), which was prepared from cellulose acetate fiber through the hydrolysis of acetyl groups, and their dyeing and fastness properties were compared. enVix exhibited better dyeability and fastness than

regular viscose rayon. This was explained by the differences in the supramolecular structures of the two fibers. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 91: 3481–3488, 2004

**Key words:** dyes/pigments; amorphous; supramolecular structures; WAXS

## INTRODUCTION

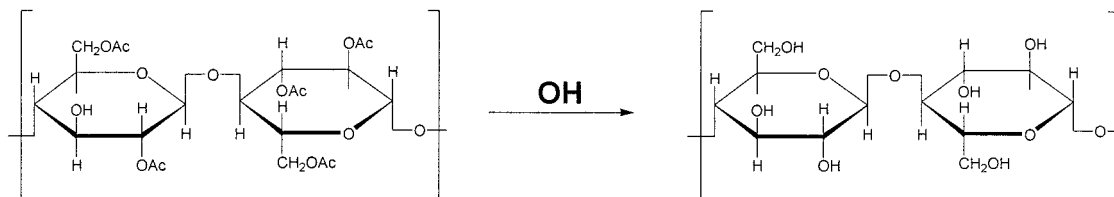
Rayon fiber is defined by the U.S. Federal Trade Commission as “a manufactured fiber composed of regenerated cellulose, as well as manufactured fibers composed of regenerated cellulose in which substituents have replaced not more than 15% of the hydrogens of the hydroxyl groups.”<sup>1</sup> Substituents consist of manufacturing impurities, pigments, fire retardants, or other additives. Rayon was the first manmade fiber. In 1924, rayon was adopted officially by the National Retail Dry Goods Association; before that time, it had been termed *artificial silk*, *fiber silk*, *wood silk*, or *viscose silk*. Rayon fibers are a diverse group, all consisting of regenerated cellulose derived from wood pulp. The basic fiber properties of rayons vary, particularly with respect to wet and dry tenacity, elongation, elastic recovery, water and alkali swelling, and, to some extent, abrasion resistance. They have certain characteristics in common that are attributable to their cellulose composition: they are hydrophilic, swell in water and alkali, are responsive to essentially the same dyes as cotton, and, when heated, decompose without melting.<sup>2</sup>

Rayon fibers are made from chemical cellulose (dissolved wood pulp), sodium hydroxide, carbon disulfide, and sometimes modifiers, which are usually based on ethoxylated natural fatty acid amines. Most rayon is made by the viscose process; however, some specialty rayon is made by the cuprammonium process in Italy, the former USSR, and Japan and by the solvent-spun process in the United States, the United Kingdom, and Austria. In the manufacture of the various viscose (wet-spun) rayon fibers, the same basic process is used. However, a number of important variables in the viscose process can be manipulated to obtain fibers of substantially different characteristics. In broad terms, the main fiber categories include regular (conventional) rayon, modified high-tenacity rayon, high-wet-modulus rayon, and polynosic rayon.<sup>2</sup>

Some of the raw materials used in the production of rayon are recoverable. The byproduct sodium sulfate is recovered and sold by rayon producers. Carbon disulfide is recovered to various extents by larger plants; on average, 30–35% is recovered, the balance being lost through volatilization or decomposition. Also, some zinc is collected as a precipitate (zinc sulfide) in the spinning process and is reworked by some producers.<sup>3</sup> However, although efforts by the major producers are expected to reduce carbon disulfide and zinc emissions, increasing environmental concern has centered on the conventional preparation of conventional regenerated cellulosic fibers because still more of the remaining zinc and carbon disulfide needs to be recovered in waste-treatment facilities at the plant site to meet established water pollution regulations.

Correspondence to: J. Koh (ccdjko@hanmail.net).

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**Scheme 1** Alkali hydrolysis of cellulose acetate fibers.

Relatively recently, Acordis Cellulosic Fibers (Netherlands) and Lenzing (Austria) introduced a lyocell fiber, the newest cellulosic fiber, with a more efficient and economical process than the universally used viscose process. Lyocell fiber is manufactured directly from high-purity cellulosic wood pulp, whereas rayon is manufactured from a cellulose derivative that is chemically regenerated back to cellulose during the spinning process. The cellulose is dissolved into a solvent, an amine oxide, and then is wet-spun. The solvent is recycled, and this eliminates the chemistry of the viscose process, which uses caustic soda, carbon disulfide, and sulfuric acid to dissolve the pulp. Although environmentally safer, solvent-spun cellulose fibers are unlikely to replace viscose rayon to a significant extent in the near future because of the high cost of converting or building facilities.<sup>2</sup>

More recently, SK Chemicals introduced a novel regenerated cellulosic fiber, enVix, which is prepared from a cellulose acetate fiber with a degree of substitution of 2.0 or higher through the saponification of 75% or more of the total acetyl groups of the cellulose acetate fiber into hydroxyl groups and has a composite crystalline structure of cellulose II and cellulose IV (Scheme 1).<sup>4,5</sup> This regenerated fiber is claimed to offer environmental advantages over other conventional regenerated fibers because it does not emit toxic materials such as carbon disulfide and heavy-metal ions.

The purpose of this work was to compare the dyeability of this new regenerated cellulose fiber (enVix) to that of viscose rayon with two types of direct dyes; it was not to determine the superiority of one type of dye. The generally accepted model for the diffusion of direct dyes into cellulosic fibers is based on a heterogeneous system in which dye anions diffuse through the liquid in the pores of the substrate and are simultaneously absorbed on pore walls.<sup>6</sup>

Two types of direct dyes were applied to regular viscose rayon and the new regenerated cellulosic fiber, which was prepared from cellulose acetate fiber; their dyeing properties and fastness properties were compared, and the results were also explained through the differences in the supramolecular structures of the two fibers.

## EXPERIMENTAL

### Materials

Viscose rayon fabrics (plain weave, warp = 82 threads/in., weft = 62 threads/in., filament fineness = 2.5 den/filament) and new regenerated cellulosic fabrics (plain weave, warp = 96 threads/in., weft = 56 threads/in., filament fineness = 2.5 den/filament) obtained from cellulose acetate fibers by alkali hydrolysis<sup>2,3</sup> were generously supplied by SK Chemicals (Suwon, Korea).

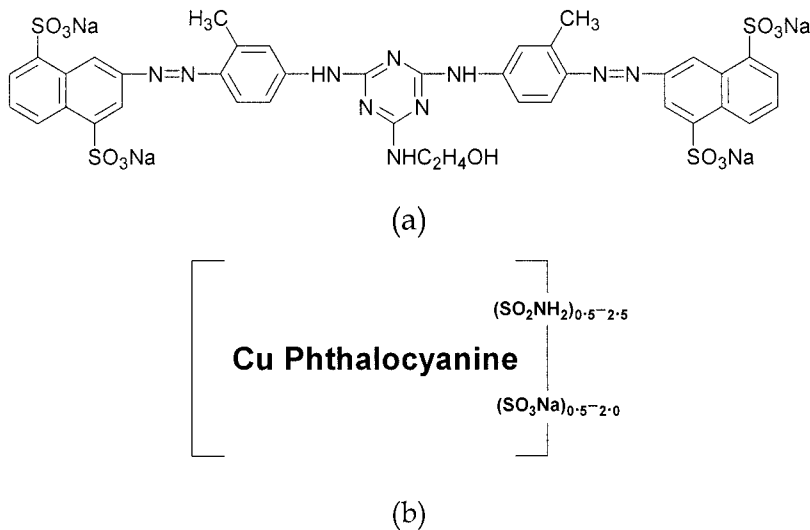
The two direct dyes used were commercial samples that were not purified before use. Kayarus Supra dye [C.I. direct yellow 86, disazo-type; Scheme 2(a)] was kindly supplied by Nippon Kayaku (Seoul, Korea), and Solophenyl dye [C.I. direct blue 199, phthalocyanine-type; Scheme 2(b)] was provided by Ciba Specialty Chemicals (Seoul, Korea).

A commercial sample of a fixing agent (Neofix RP-70C, a polyethylene-polyamine-based cationic resin) was supplied by Korea Fine Chemical Co. (Dneagu, Korea). All other reagents were general-purpose-grade.

### Wide-angle X-ray analysis

Wide-angle X-ray diffraction (WAXD) data were collected for bundles of commercial viscose rayon and enVix fibers at room temperature. The fibers were aligned perpendicularly or parallel to the direction of the X-ray beam to obtain equatorial and meridional X-ray diffraction data, respectively. The intensity measurements were gathered with a Bruker 2D-GADDS diffractometer (Massachusetts) with Cu K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). The intensities were measured within the range of scattering angles ( $2\theta$ ) of 5–40° in the equatorial and meridional directions. The total scattering data were obtained by the summation of both equatorial and meridional scattering data.

To estimate the approximate crystallinities of these fibers, we separated the empirical amorphous background and Bragg diffraction peaks in the total scattering data by a conventional curve-fitting method with commercial software. The individual peaks and amorphous background were assumed to have Gaussian-type peak profiles.



Scheme 2 Direct dyes used in this study.

**Birefringence measurements**

The birefringence values were obtained with a polarized microscope (Pol-12, Leica, Bensheim, Germany) with a 30-order Berek’s compensator; the retardation change was recorded, and the degree of orientation ( $\Delta n$ ) was calculated as follows:

$$\Delta n = \frac{\tau}{d} \tag{1}$$

where  $\tau$  is the retardation and  $d$  is the diameter of the fiber.

**Dyeing**

A 50-mL dye bath, suitable for a 2.0-g sample of rayon (liquor ratio = 1 : 25), containing a direct dye and Glauber’s salt was prepared. Dyeing was performed for 30 min at 100°C in an Ahiba Nuiance laboratory dyeing machine (Ahiba, Datacolor International, Switzerland). The dyeing method used is shown in Figure 1. The fabrics, dyed with direct dyes, were rinsed and then treated with a fixing agent (2.5% on the weight of fabric) for 15 min at 60°C.

The rayon was dyed under various dye bath conditions (salt concentration, liquor ratio, and dye concentration) to investigate their effects on the dyeing properties of the regenerated cellulosic fibers. The exhaustion behavior of the direct dyes on the fabric was also investigated by the monitoring of the exhaustion (%) values of the dyed fabrics as dyeing proceeded.

The extent of dye exhaustion (%) achieved for an appropriate dye concentration on each of the two types of fibers was determined with eq. (2) by the absorbance spectroscopy analysis of the dye bath before and after dyeing:

$$\text{Exhaustion (\%)} = \frac{A_b - A_a}{A_b} \tag{2}$$

where  $A_a$  is the absorbance of the dye bath after dyeing and  $A_b$  is the absorbance of the dye bath before dyeing.

The color strength ( $K/S$ ) of the dyed samples was measured with a Macbeth Color-Eye 3000 spectrophotometer (New York) (standard light D65, 10° standard observer, and specular component included) interfaced with a personal computer:

$$K/S = \frac{(1 - R)^2}{2R} \tag{3}$$

where  $R$  is the reflectance of an infinitely thick layer of the material illuminated with light of a known wave-

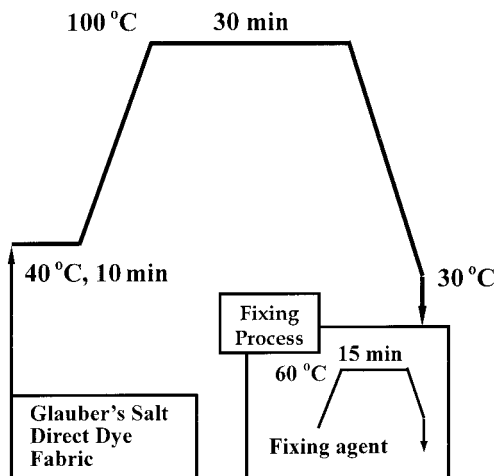
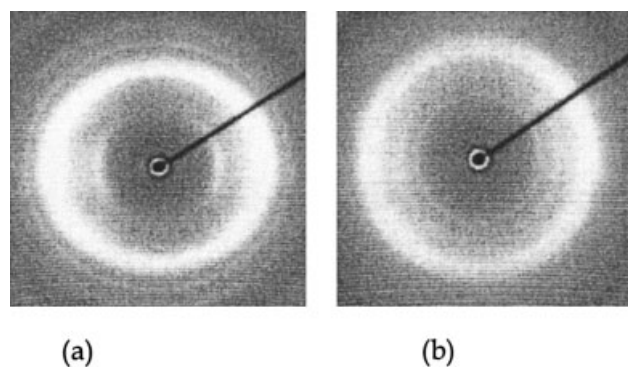


Figure 1 Direct dyeing profile.



**Figure 2** Fiber diagrams of (a) viscose rayon and (b) enVix fibers.

length,  $K$  is the absorption coefficient, and  $S$  is the scattering coefficient. The function  $K/S$  is directly proportional to the concentration of the colorant in the substrate.<sup>7</sup>

#### Color fastness

The regenerated cellulosic fibers were dyed (1/1 standard depth), treated with a fixing agent, and heat-set (170°C, 60 s) to test the color fastness. The color fastness was determined according to International Standards; the specific tests were ISO 105 C06/C2S (color fastness to washing), ISO 105 E04 (color fastness to perspiration), ISO 105 X12 (color fastness to rubbing), and ISO105 B02 (color fastness to light). Staining and changes in color were assessed with gray scales.

### RESULTS AND DISCUSSION

Figure 2(a,b) shows the fiber diagrams of regular viscose rayon fibers and the new regenerated cellulosic fiber, enVix. The two sets of data contain almost the same features: an amorphous halo with several crystalline Bragg peaks. The intense amorphous halo in data sets indicates the existence of a significant amount of an amorphous, disordered phase. The numbers and positions of the Bragg peaks in both patterns are similar, indicating that the crystalline structures of both fibers are similar. However, the peak width in the azimuthal direction for viscose rayon is smaller than that measured from the enVix fiber, and this reveals that the orientation of the crystalline phase of viscose rayon is better.<sup>8</sup> The higher amorphous content and poor crystalline orientation of the enVix fiber may arise either from the completely amorphous nature of the cellulose acetate fiber, used as the raw fiber, or from the severe thermal and mechanical treatments used during the manufacturing process.

The dyeing properties of fibrous polymeric materials depend on the relative amount of the amorphous

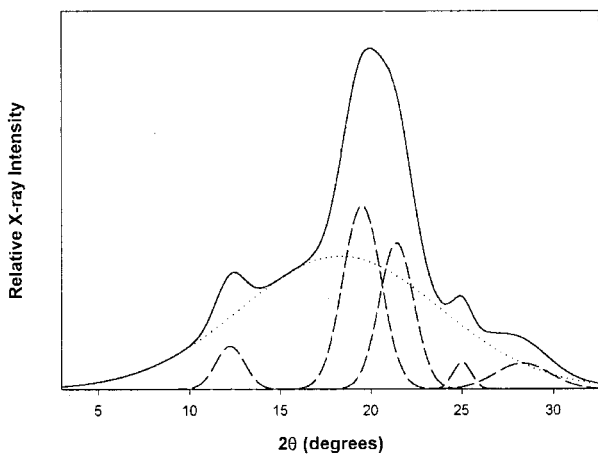
phase and the chain packing, especially in the intermediate phase between crystalline and amorphous phases. Both structural parameters strongly affect the sorption characteristics of fibrous polymers on solvent and dye molecules. Because the molecular orientation provides information on the degree of chain packing in both the crystalline and intermediate phases, birefringence measurements were conducted to measure the degree of orientation of these viscose rayon and enVix fibers. Birefringence data, determined by polarized microscopy, supported the WAXD results: the birefringence of viscose rayon (3.63) was found to be higher than that of enVix fibers (1.93). These results show that the chain packing of viscose rayon fiber is better than that of enVix fiber, and this indicates the poor dyeability of viscose rayon.

Figure 3 depicts the total scattering data of viscose rayon fiber and enVix fiber measured at room temperature. The noncrystalline part was taken as that contributing to the intensity of the diffuse background, and the crystalline peaks was taken as that contributing to the intensity of the selectively diffracted radiation (the peaks occurring on top of the background). In Figure 3, the amorphous backgrounds and Bragg peaks of both fibers were resolved by a curve-fitting method. In both sets of data, we found three Bragg peaks at  $2\theta \approx 12$ ,  $2\theta \approx 19$ , and  $2\theta \approx 22^\circ$ , which are indexed as (101),  $(10\bar{1})$ , and (002) peaks, respectively, of cellulose II. Conventional rayon fibers, such as viscose rayon, cupraammonium rayon, Bemberg rayon, high-tenacity rayon, and Fortisan, are known to have the crystalline structure of cellulose II.<sup>9</sup> In Figure 3(b), we found another peak ( $2\theta \approx 15.4^\circ$ ), which is the  $(10\bar{1})$  peak of the cellulose IV phase. The peak at  $2\theta \approx 22^\circ$  was attributed to the (002) peak of cellulose IV, which overlapped (002) peaks of cellulose II. The coexistence of the Bragg peaks from cellulose II and cellulose IV for enVix fibers indicates the bimorphic behavior of the crystalline structure of the enVix fibers.

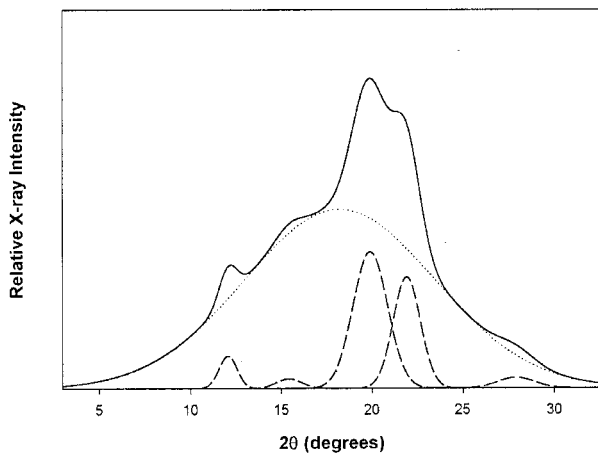
Because most crystalline Bragg peaks of viscose rayon and enVix fibers were located in the region of  $2\theta \leq 40^\circ$ , their approximate apparent crystallinities were estimated on the basis of the curve-fitted data shown in Figure 3. The estimated values of the crystallinity are  $39 \pm 2\%$  and  $27 \pm 2\%$  for viscose rayon and enVix fibers, respectively. The lower value of the crystallinity of the enVix fiber indicates better solvent-processing characteristics and dyeability.

In Figures 4–7, the dyeing properties of the two rayons are compared. Direct dyes are usually applied with the addition of an electrolyte at or near the boil. The extent to which direct dyes are affected by the addition of electrolytes to the dye bath is known as *salt sensitivity*. Direct dyes vary appreciably with respect to the effect of electrolytes. The addition of an electrolyte increases the rate of strike of the dye. The commonly used electrolytes are Glauber's salt (sodium

sulfate) and common salt (sodium chloride); when cellulose is immersed in a solution of a direct dye, it absorbs the dye from the solution until equilibrium is attained, and at this stage, most of the dye is taken up by the fiber. Cellulose carries a negative charge in pure water. As the dye is also an anion and, therefore, is negatively charged, there is an electrostatic anion-anion repulsion between the dye and cellulose. Hence, the probability of the dye adsorbing onto the fiber, that is, its level of substantivity, is reduced. The substantivity of the dye for cellulose is the proportion of the dye absorbed by the fiber compared with that remaining in the dye bath. Through the addition of an inert electrolyte such as common salt or Glauber's salt to the dye bath, this electrostatic barrier, known as the *Donnan potential*, can be largely suppressed, facilitating dye-fiber contact, allowing better interactions of

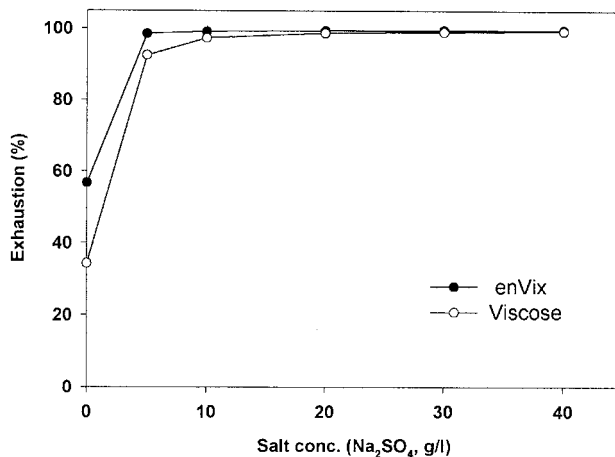


(a)

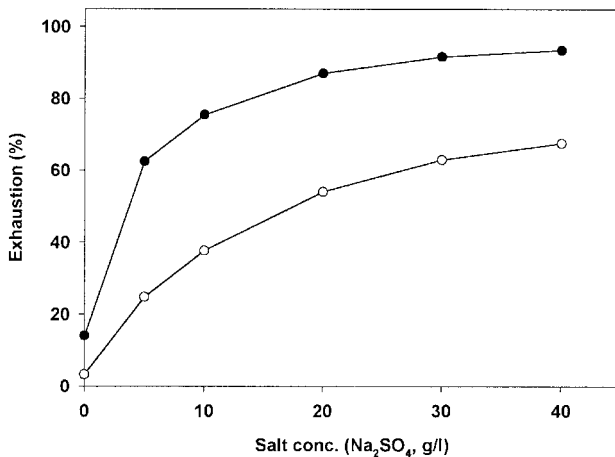


(b)

**Figure 3** WAXD pattern resolved into individual integral Bragg intensities and the amorphous background: (a) viscose rayon and (b) enVix fibers. The dotted and broken lines represent the estimated background and Bragg peaks, respectively.



(a)

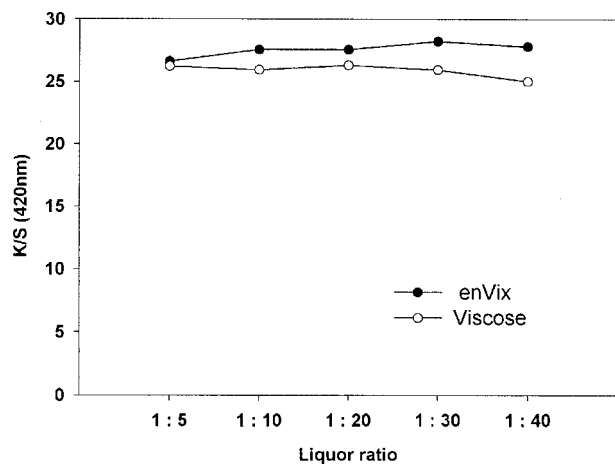


(b)

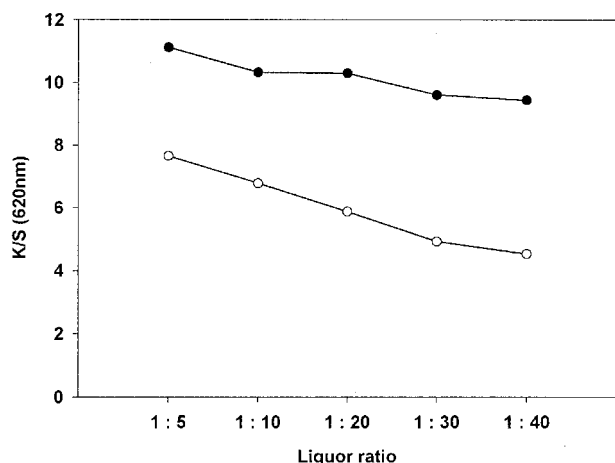
**Figure 4** Salt effects on the exhaustion of dyeing (dye concentration = 2% o.w.f., liquor ratio = 1 : 25): (a) Color Index (C.I.) direct yellow 86 and (b) C.I. direct blue 199.

the Yoshida and van der Waals forces, and thereby improving substantivity. The diffusion coefficient of the dye is, therefore, a function of both the dye and electrolyte concentrations.<sup>10</sup> The results shown in Figure 4 support this statement, in that the exhaustion (%) of the dyeing increases as the concentration of the salt increases. The dye exhaustion (%) value of a direct dye is a linear function of the salt concentration, particularly at lower dye concentrations, within cellulose in the presence of a fixed amount of added dyes, although the slope decreases with increasing salt concentration. It is thought that the initial rapid rise is due to the response of the dye to the lowering of the electrical potential barrier to diffusion as the concentration of the electrolyte increases.

It is also evident from Figure 4 that the exhaustion (%) of the dyeing on enVix is stronger than that on viscose rayon for each of the two dyes, presumably because of the lower crystallinity values (%), degree of



(a)



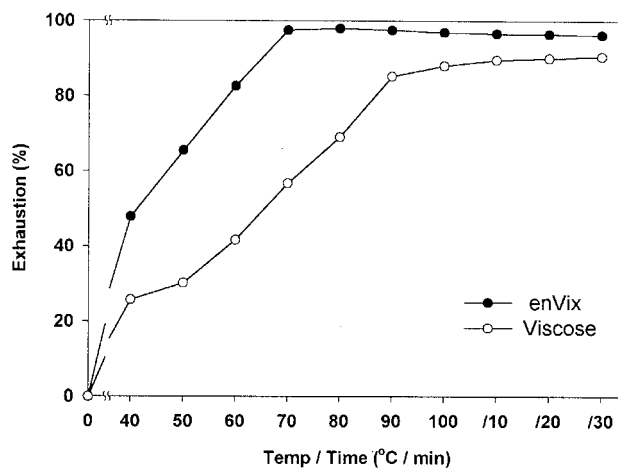
(b)

**Figure 5** Liquor ratio effects on  $K/S$  of the dyed fabrics (dye concentration = 2% o.w.f., salt concentration = 20 g/L): (a) C.I. direct yellow 86 and (b) C.I. direct blue 199.

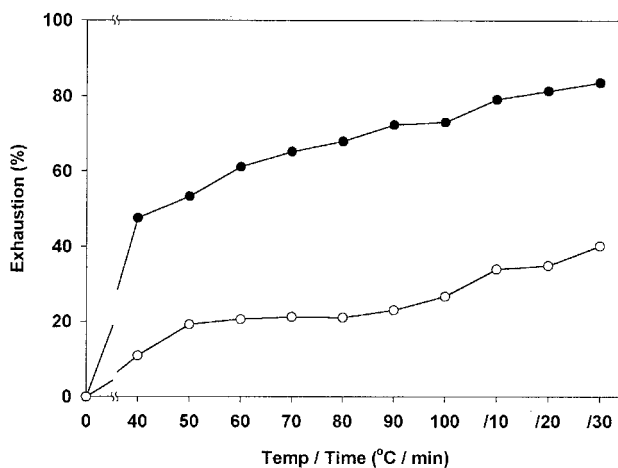
orientation, which is shown in WAXD patterns (Figs. 2 and 3), and birefringence; as a useful generalization, fibers may be regarded as structures in which there is a spread of molecular order, ranging from highly ordered crystalline domains to disordered amorphous regions.<sup>11</sup> The strength originates in the crystalline material, whereas the amorphous material provides the flexibility, porosity, and regions generally accessible to liquids, dyes, and other reagents. Therefore, the fiber properties, including the dyeing properties, vary, depending on the relative degrees of order and disorder in the structure (often described loosely as the crystalline/amorphous ratio) and the molecular alignment (degree of orientation); that is, lower orientation and crystallinity mean a higher rate of dye diffusion with these fibers.

Also, C.I. direct blue 199 with a phthalocyanine structure shows greater magnitudes of the difference

in the exhaustion values between enVix and viscose rayon than C.I. direct yellow 86 with a disazo structure. The relatively greater difference in the exhaustion (%) of C.I. direct blue 199 between dyeing on enVix and viscose implies that the shape of a dye molecule also affects its equilibrium uptake; the phthalocyanine dyes (turquoise products), which tend to be square and broad in structure, have substantially poorer diffusion properties than elongated yellow and red chromophores (even when their high molecular weights are considered) because the dye molecule is prevented by its larger size from penetrating the more orderly arrangement of the noncrystalline regions of the fibers. Therefore, the amount of salt affects dyes with low affinity (C.I. direct blue 199) considerably more than those with high affinity (C.I. direct yellow 86).

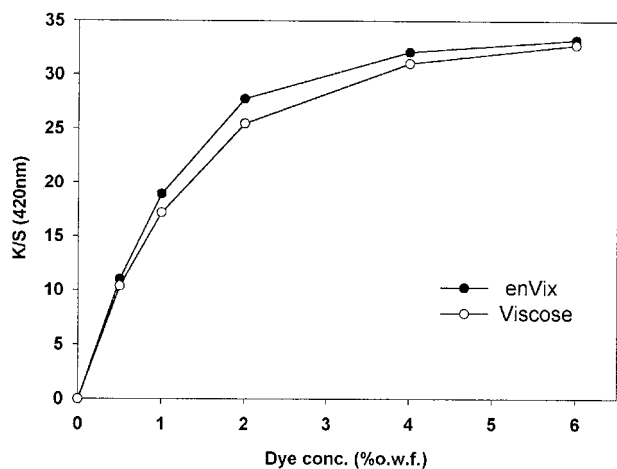


(a)

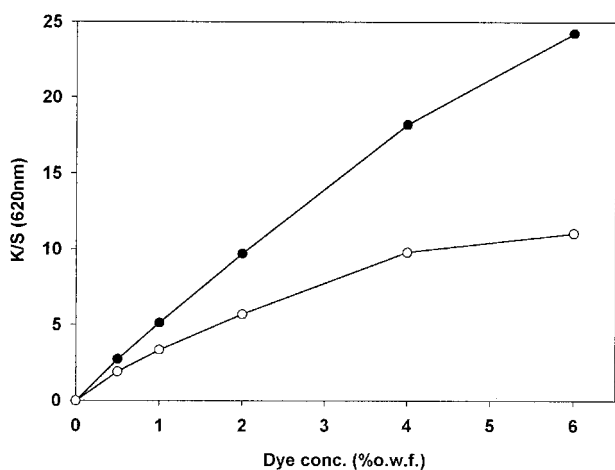


(b)

**Figure 6** Exhaustion behavior of direct dyes on regenerated cellulose fibers (dye concentration = 2% o.w.f., salt concentration = 20 g/L, liquor ratio = 1 : 25): (a) C.I. direct yellow 86 and (b) C.I. direct blue 199.



(a)



(b)

**Figure 7** Buildup properties of direct dyes in regenerated fibers (salt concentration = 20 g/L, liquor ratio = 1 : 25): (a) C.I. direct yellow 86 and (b) C.I. direct blue 199.

Figure 5 shows the liquor ratio effects on the dyeability of the regenerated cellulosic fibers. As expected, the direct dyes decrease in substantivity to a greater or lesser extent when the liquor ratio is increased. It follows logically that as the liquor ratio increases, the probability of contact between the dye molecules and fiber surface decreases; that is, fewer dye molecules (per unit of time) are adsorbed onto the fiber surface. Equilibrium between adsorption and desorption is thus displaced, desorption becoming stronger; that is, substantivity is lower. This decrease in substantivity with the liquor ratio is relatively less marked with enVix than viscose rayon, especially in the case of C.I. direct blue 199. Also, it is well known that the liquor ratio affects dyes with low affinity considerably more than those with high affinity, and the results in Figure 5 support this statement, in that the reproducibility of the *K/S* value and the shade of

low-affinity direct dyes (C.I. direct blue 199) is generally worse than that of those with high affinity (C.I. direct yellow 86) in the exhaust method.

Figure 6 shows the exhaustion behaviors of direct dyes on regenerated cellulose fibers during dyeing. As expected, the dyeing rates of the two rayons are not the same because of their supramolecular structures. Different dyeing rates, brought about by differences in the physical structures, are clearly of practical importance. Predictably, enVix shows faster exhaustion behavior than viscose rayon, presumably because of the lower crystallinity (%) values and degree of orientation.<sup>12</sup> There is a general rule in textile dyeing: faster exhaustion means uneven dyeing. However, in the dyeing results, no differences in levelness between the two fibers have been observed.

In all types of cellulose fibers, there are crystalline regions (with high parallelism of the cellulose macromolecules) and noncrystalline regions (in which the macromolecules are disordered to some extent). The parallel macromolecules in crystalline regions are linked together by hydrogen bonds between OH groups of the cellulose. The crystallites lie preferentially parallel to the fiber axis and are separated by regions of lower order and intermicellar spaces. The average size of the crystallites and the quantitative ratio of the crystallites to regions of lower order are strongly fiber-specific. Only water-swollen intermicellar spaces and regions of lower order of the fiber are accessible to large direct dye molecules. It is completely impossible for a dye to diffuse into highly oriented crystallites. Dyeing, therefore, only proceeds at the outer walls of the crystallites and in the nonoriented cellulose.

Also, as shown in Figure 6, C.I. direct blue 199 has a much slower dyeing rate than C.I. direct yellow 86, presumably because the shape of a dye molecule affects its diffusion rate, as mentioned earlier. The phthalocyanine chromophore, which is square and broad in structure, tends to have poorer diffusion properties and, therefore, poorer migration and washing-off properties, for example, than azo chromophores, and this is consistent with the results of Figure 6.

Further evidence that the dyeability of enVix is better than that of viscose rayon is provided by the results for the buildup properties obtained for the two types of dyes; as Figure 7 shows, the buildup properties of two direct dyes on enVix are relatively better than on viscose rayon. For a given dye concentration on the fiber, enVix dyes to a much deeper shade than viscose rayon, especially with C.I. direct blue 199; the *K/S* value of dyed enVix increases up to the dye concentration of 6.0% o.w.f. (*K/S* = 24.3) without reaching saturation, whereas that of dyed viscose rayon reaches saturation around a dye concentration of 6.0% o.w.f., which gives a *K/S* value of about 11.1. In the case of

**TABLE I**  
Wash Fastness of the Direct Dyes on Regenerated Cellulose Fibers

Dye fiber	Staining				Change			
	Direct yellow 86		Direct blue 199		Direct yellow 86		Direct blue 199	
	en Vix	Viscose	en Vix	Viscose	en Vix	Viscose	en Vix	Viscose
Acetate	5	5	5	5				
Cotton	2	1	2	2-3				
Nylon	5	5	3-4	3-4				
PET	5	5	5	5	4	4	3-4	2
Acrylic	5	5	5	5				
Wool	5	5	5	5				

C.I. direct yellow 86, the  $K/S$  value of the dyed rayons reaches saturation around a dye concentration of 4.0% o.w.f., which gives a  $K/S$  value of around 33, and the differences in the buildup properties between enVix and rayon are relatively small, probably because of the relatively higher substantivity of the dye applied.

Table I shows that the fastness to washing of 1/1 standard depth dyeing of both C.I. direct yellow 86 and C.I. direct blue 199 on enVix is slightly higher than that of comparable depth dyeing on viscose rayon. The results can be attributed to the excellent affinity of enVix to dyes because of its supramolecular characteristics. Table II reveals that, although the rubbing fastness of the 1/1 standard depth dyeing of C.I. direct yellow 86 on enVix is identical to that of the dye on viscose rayon, the rubbing fastness of C.I. direct blue 199 is higher than that on viscose rayon. The fastness to light of the two dyed rayons is excellent (a rating of over 5), regardless of the dye applied.

## CONCLUSIONS

A new regenerated cellulosic fiber, which possesses a composite crystalline structure of cellulose II and cellulose IV, was prepared by the saponification at least 75% of the total acetyl groups of a cellulose acetate fiber with a degree of substitution of 2.0 or higher into hydroxyl groups.

The structural characteristics and dyeing properties of the novel regenerated cellulose fiber, enVix, have been compared with those of conventional viscose rayon; the crystallinity of enVix is lower than that of

viscose rayon, and the orientation of crystallites along the fiber axis in the former is somewhat lower according to measurements of birefringence. Also, a careful comparison of the dyeing properties of the two rayons with direct dyes has confirmed that there is a correlation between the supramolecular structures and the dyeing properties; enVix exhibits faster exhaustion behavior and better buildup properties than viscose rayon, presumably because of the lower crystallinity (%) and degree of orientation. In addition, it shows stable final color yields, regardless of liquor ratio changes in the dyeing process, and so the reproducibility of enVix dyeing is expected to be excellent. The fastness properties of direct dyes on enVix are also better than those of viscose rayon. The results suggest that this novel regenerated cellulosic fiber can be used as an important alternative to conventional viscose rayon, although more detailed studies on this new fiber are necessary before any definite conclusions can be drawn.

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**TABLE II**  
Rubbing Fastness of the Direct Dyes on Regenerated Cellulose Fibers

Dye fiber	Direct yellow 86		Direct blue 199	
	en Vix	Viscose	en Vix	Viscose
Dry	5	5	5	4-5
Wet	5	5	4	2-3