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Reactive dyeing properties of novel regenerated cellulosic fibres

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Abstract

Two types of reactive dye (monochlorotriazine and vinylsulfone types) were applied to regular viscose rayon and new regenerated cellulosic fibre (*enVix*®), which was prepared from cellulose acetate fibre by the hydrolysis of acetyl groups, and their dyeing and fastness properties were compared. From the results, it was found that *enVix*® exhibits better dyeability and fastness than the regular viscose rayon. These results were also explained by the differences in the supramolecular structure of these two fibres.

Keywords: Reactive dye; Viscose rayon; Regenerated cellulosic fibre; Cellulose acetate; Dyeing properties; Fastness; Supramolecular structure

1. Introduction

Rayon was the first man-made fibre. In 1924, the name "rayon" was adopted officially by the National retail Dry Goods Association; before that, it had been termed artificial silk, fibre silk, wood silk or viscose silk. Rayon fibres are a diverse group, consisting of regenerated cellulose derived from wood pulp. Rayon fibre is defined by the U.S. Federal Trade Commission as "a manufactured fibre composed of regenerated cellulose, as well as manufactured fibres composed of regenerated cellulose in which substituents have replaced not more than 15% of the hydrogens of the hydroxyl groups" (Rules and Regulations Under the Textile Fibre Products Identification Act, U.S. Federal Trade Commission). Substituents consist of manufacturing impurities, pigments, fire retardants or other additives [1].

Most rayon is made by the viscose process; rayon fibres 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. Some of the raw materials used in production of rayon are recoverable. By-product sodium sulfate is recovered and sold by rayon producers. Carbon disulfide is recovered in varying degrees by larger plants; on the average, 30–35% is recovered, the balance being lost through volatilization or decomposition. Also, some zinc is collected as precipitates (zinc sulfide) in the spinning process and reworked by some producers [2]. Although efforts by the major producers are expected to reduce carbon disulfide and zinc emissions, increasing environmental concern has centred on the conventional preparation of conventionally regenerated cellulosic fibres since still some more amount 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.

Recently, Acordis Cellulosic Fibres (Netherlands) and Lenzing (Austria) introduced a lyocell fibre, the newest member of the cellulosic fibres, using a more efficient and economical process than the universally used viscose process. Lyocell fibre is manufactured directly from high-purity cellulosic wood pulp, whereas

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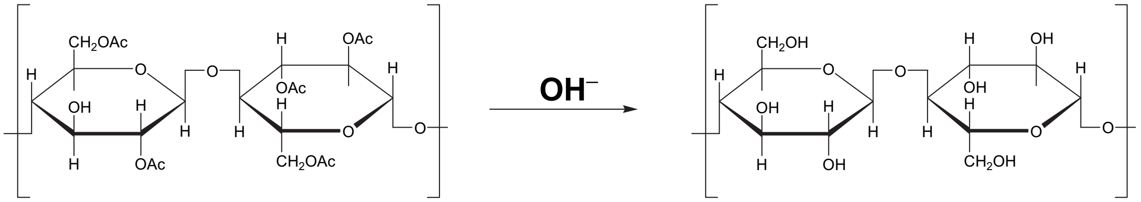


Scheme 1. Alkali-hydrolysis of cellulose acetate fibres.

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 wet-spun; the solvent is recycled, eliminating the chemistry of the viscose process, which uses caustic soda, carbon disulfide and sulfuric acid to dissolve the pulp. Although environmentally safer, solvent-spun cellulosics are unlikely to replace viscose rayon to a significant extent in the near future because of the high cost of converting or building facilities.

Recently, SK Chemicals introduced a novel regenerated cellulosic fibre, *enVix*®, which was prepared from a cellulose acetate fibre with a degree of substitution of 2.0 or higher by saponifying 75% or greater of the total acetyl groups of the cellulose acetate fibre into hydroxyl groups and has a composite crystalline structure of cellulose II and IV (Scheme 1) [3–5]. This regenerated fibre is claimed to offer environmental advantages over other conventional regenerated fibres because it does not emit toxic materials such as carbon disulfide and heavy metal ions.

In the previous work, supramolecular structures of *enVix*® and viscose rayon were investigated in a comparative manner [5]. The crystallinity of *enVix*® was found to be lower than that of viscose rayon and the orientation of crystallites along the fibre axis in the former was somewhat lower according to measurements of birefringence. Also, careful comparison of the direct dyeing and vat dyeing properties and reactive printing properties of two rayons confirms that there is a correlation between supramolecular structures and the dyeing properties [5–7].

enVix® is a cellulosic fibre and therefore can be dyed with any class of dyestuff suitable for other cellulosics. Information in this article is confined to reactive dyes as they are seen as the major dye class used on cellulosic fibres in the world. Three types of reactive dyes were applied to regular viscose rayon and a new regenerated cellulosic fibre which was prepared from cellulose acetate fibre; their dyeing and fastness properties were compared and the results were also explained by the differences in the supramolecular structures of these two fibres.

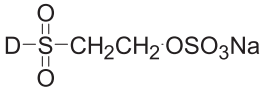
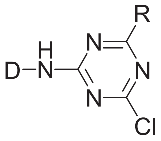
2. Experimental

2.1. Materials

Viscose rayon fabrics (plain weave, warp 82 threads/inch, weft 62 threads/inch, filament fineness 2.5 denier/filament) and new regenerated cellulosic fabrics (plain weave, warp 96 threads/inch, weft 56 threads/inch, filament fineness 2.5 denier/filament) obtained from cellulose acetate fibres by alkali-hydrolysis [3–5] were generously supplied by SK Chemicals (South Korea).

The two reactive dyes used were commercial samples that were not purified prior to use; Remazol dye (C.I. Reactive Red 23, vinylsulfone type) and Procion dyes (C.I. Reactive Blue 71, monochlorotriazine type) were kindly supplied by BASF (Table 1). A commercial sample of soaping agent (SNOGEN CS-940N, non-ionic)

Table 1
Structures of the reactive dyes used in this study

Dye	Type	Reactive group	Fixation mechanism	Dyeing temp. (°C)
C.I. Reactive Red 23	Vinylsulfone (VS)	 $\mathrm{D{-}SO_2{-}CH_2CH_2{\cdot}OSO_3Na}$	Addition	60
C.I. Reactive Blue 71	Monochlorotriazine (MCT)		SN2	80

was supplied by Daeyoung Chemicals. All other reagents were of general purpose grade.

2.2. Fourier transform infrared spectroscopy

FT-IR spectra were recorded on a Prospect-IR spectrometer (MIDAC, USA) using potassium bromide disks. A total of 32 scans for each sample were taken with a resolution of 4 cm^{-1}. The sample for FT-IR analysis was prepared as follows. Fibres were powdered in a mortar cooled with liquid nitrogen, and 1.0 mg of the obtained powder was dispersed in KBr to obtain the required discs. Both fibres and KBr were carefully dried before disk preparation and were subjected to FT-IR analysis immediately afterwards.

2.3. Dyeing

A 50 ml dyebath, suitable for a 2.0 g sample of rayon (liquor ratio 1:25), containing a reactive dye and Glauber's salt was prepared. Dyeing was performed under various appropriate conditions (Fig. 1) in a laboratory dyeing machine (Ahiba, Datacolour International, Switzerland). The fabric that had been dyed with reactive dyes was after-treated with soaping agent (0.5 g/l) for 30 min at 98 °C.

The rayon was dyed at various dyebath conditions (salt concentration, alkali concentration, liquor ratio, and dye concentration) in order to investigate their effects on the dyeing properties of the regenerated cellulosic fibres. Exhaustion behaviour of reactive dyes on the fabric was also investigated by monitoring the exhaustion (%) values of dyed fabrics as dyeing proceeded.

The extent of dye exhaustion (%) achieved for an appropriate dye concentration on each of the two types of fibre was determined using Eq. (1) by the absorbance spectroscopic analysis of the dyebath before and after dyeing:

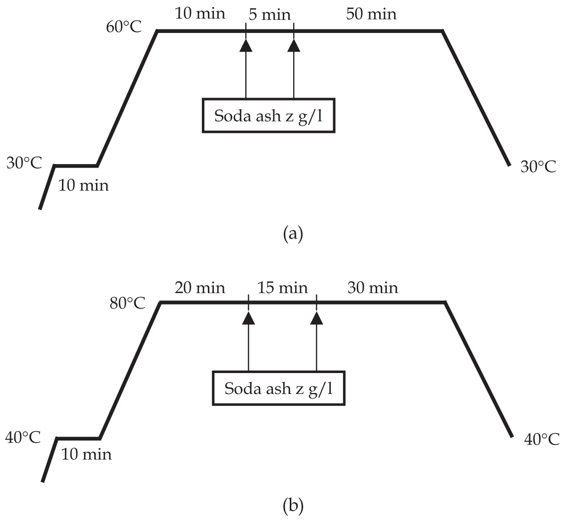


Fig. 1. Reactive dyeing profile: (a) vinylsulfone type, (b) monochlorotriazine type.

$$\text{Exhaustion}\,(\%) = \frac{A_b - A_a}{A_b} \qquad (1)$$

where

A_a: absorbance of the dyebath after dyeing,
A_b: absorbance of the dyebath before dyeing.

The fixation (%), percentage of dye exhausted which is covalently bonded to the fibre, was determined as follows. At the end of dyeing, the dyed fabric was cut into two pieces (1.25 g each); one piece was allowed to dry in the open air and the other piece was treated in an aqueous solution containing Sandozine NIE (5.0 g/l) and Na_2CO_3 (2.5 g/l), using a 50:1 liquor (62.5 ml of liquor) ratio at 98 °C for 30 min and finally rinsed thoroughly in tap water before being allowed to dry in the open air [8]. The colour strength (K/S) of the each of the two dry, dyed samples is measured using spectrophotometer and the extent of apparent fixation (%) achieved is calculated using following equation:

$$F\% = 100 \times \frac{(K/S)_2}{(K/S)_1} \qquad (2)$$

where

$(K/S)_1$ = K/S values of dyed sample before washing,
$(K/S)_2$ = K/S value of dyed sample after washing,
$F\%$ = degree of fixation of absorbed dye.

2.4. Colour fastness

The regenerated cellulosic fibres were dyed (1/1 standard depth), after-treated with soaping agent and heat-set (170 °C, 60 s) in order to test the colour fastness. The colour fastness was determined according to International Standards; the specific tests used were ISO 105 C06/C2S (colour fastness to washing), ISO 105 E04 (colour fastness to perspiration), ISO 105 X12 (colour fastness to rubbing) and ISO 105 B02 (colour fastness to light). Staining and change in colour were assessed using grey scales.

3. Results and discussion

3.1. FT-IR spectrum of regenerated cellulosic fibres

Fig. 2 reports the FT-IR spectrum of viscose rayon (c) and *enVix*® fibre (b) together with the spectrum of its precursor, cellulose acetate fibre (a). The spectrum of cellulose acetate is satisfactorily identical with that of cellulose acetate reported in the literature [9]; it shows

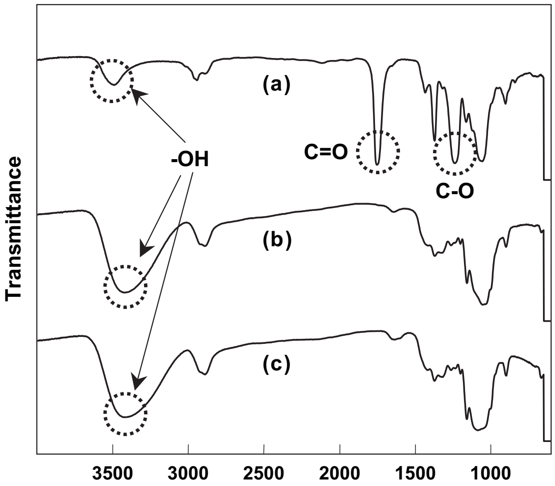


Fig. 2. FT-IR spectra of cellulose acetate (a), *enVix*® (b), and viscose rayon (c).

carbonyl absorptions ($\nu_{C=O}$) at 1745 cm^{-1}, as well as the typical absorption of the cellulose residue and the additional functional groups of the ester substituents (Scheme 1).

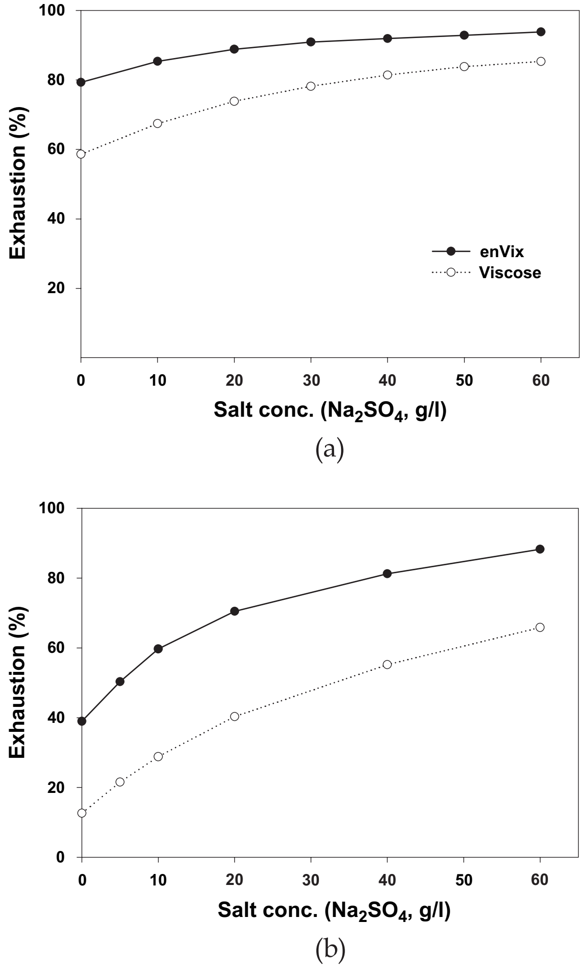
The spectrum of *enVix*® prepared from cellulose acetate via the alkaline treatment is almost identical in both location and relative intensities of the peak with that of regular viscose rayon. The spectra of two regenerated cellulosic fibres show two changes compared with that of cellulose acetate. The main changes (indicated by arrows in curves) are the increase of the −OH band at 3490 cm^{-1}, and the decrease of the C=O and C−O ester group band at 1745 cm^{-1} and 1235 cm^{-1}, respectively. The FT-IR information obtained clearly demonstrates that the applied saponification reaction by alkaline treatment (alkaline hydrolysis) has been successfully carried out.

3.2. Dyeing properties

In Figs. 3–8, the dyeing properties of two rayons were compared. The authors investigated the variation in dye yield with the variation in salt concentration, alkali concentration and liquor ratio, comparing the effect on the shade of *enVix*® with that on viscose rayon.

The molecules of monofunctional reactive dyes are smaller than those of direct dyes, and their smaller size is accompanied by a correspondingly lower substantivity. The molecules of direct dyes are made deliberately large so as to build up the physical attraction between fibre and dye, thus making them more substantive. Much smaller molecules may be suitable for use as reactive dyes because one covalent bond is about 30 times as strong as one van der Waals bond [10].

The generally accepted model for dyeing of reactive dyes into cellulosic fibres consists of two phases i.e. adsorption and diffusion phase and fixation phase [11].

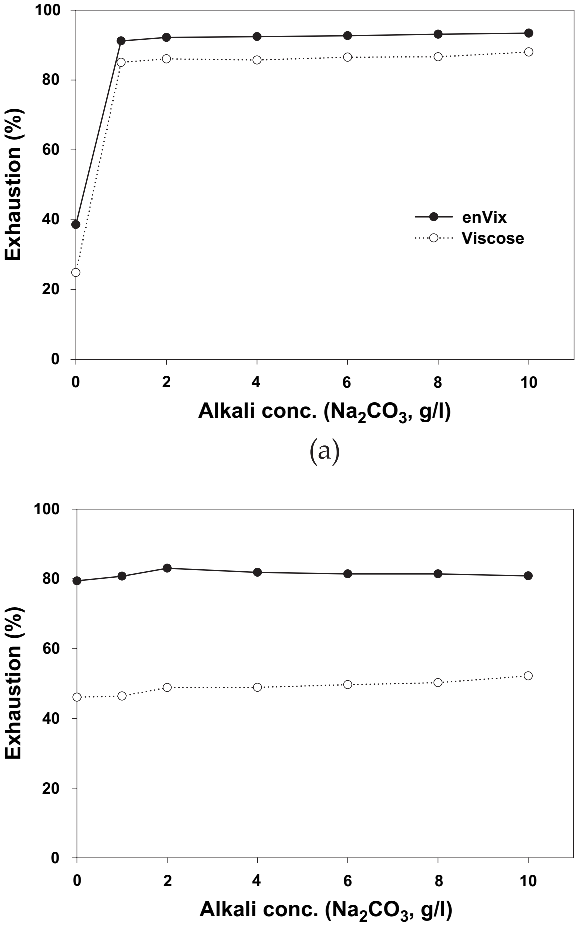


(a)

(b)

Fig. 3. Salt effects on exhaustion (%) of dyeings (dye 2.0% o.w.f., liquor ratio 1:20): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MT).

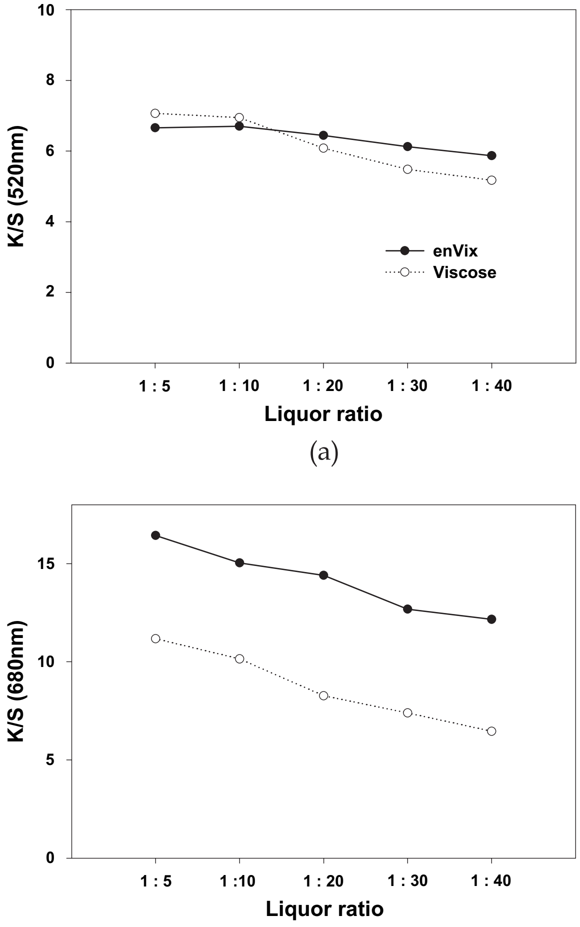
Reactive dyes are usually applied with the addition of electrolyte and the extent to which reactive dyes are affected by the addition of electrolytes to the dyebath is known as salt sensitivity. The addition of electrolyte increases the rate of strike of the dye; when cellulose is immersed in a solution of a reactive dye it absorbs dye from the solution until equilibrium is attained, and at this stage most of the dye is taken up by the fibre. Cellulose carries a negative charge in pure water. As the dye is also anion, hence negatively charged, there is an electrostatic anion–anion repulsion between dye and cellulose. Therefore, by adding inert electrolyte such as common salt or Glauber's salt to the dyebath, this electrostatic barrier, known as the Donnan potential, can be largely suppressed, facilitating dye/fibre contact and allowing better interaction of the Yoshida and van der Waals forces and hence improving substantivity. The diffusion coefficient of the dye is therefore a function of both dye and electrolyte concentration [12]. The result in Fig. 3 supports this statement, in that the exhaustion (%) of the dyeings increased with increasing



(a)

(b)

Fig. 4. Alkali effects on exhaustion (%) of dyeings (dye 2.0% o.w.f., liquor ratio 1:20): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MCT).



(a)

(b)

Fig. 5. Liquor ratio effects on K/S of the dyed fabrics (dye 2.0% o.w.f., salt 20 g/l): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MCT).

concentration of salt applied. The dye exhaustion (%) value of reactive dye is a linear function of salt concentration particularly at lower dye concentrations within the cellulose in the presence of a fixed amount of added dyes, although the slope decreases with increasing salt concentration. It is considered that the initial rapid rise is due to the response of dye to the lowering of electrical potential barrier to diffusion as the concentration of electrolyte increases.

It is also evident from Fig. 3 that the exhaustion (%) of the dyeings on *enVix*® is higher than that on viscose rayon for each of the two dyes and these results are consistent with the previous work investigating their supramolecular structures in a comparative manner [5–7]. The excellent dyeing yields on *enVix*® can be ascribed to the lower crystallinity (%) values (*enVix*® 27%, viscose rayon 39%) and degree of orientation (*enVix*® 1.93, viscose rayon 3.63); as a useful generalisation, fibres may be regarded as structures in which there is a spread of molecular order, ranging from highly ordered crystalline domains to disordered amorphous regions [12]. The strength originates in the crystalline material whilst the amorphous material provides the flexibility, porosity and the regions generally accessible to liquids, dyes and other reagents. Therefore, the fibre properties, including dyeing properties, vary depending upon the relative degrees of order and disorder in the structure (often described loosely as the crystalline/amorphous ratio) and also molecular alignment (degree of orientation), i.e. lower orientation and crystallinity mean a higher rate of dye diffusion with these fibres.

Once again, Fig. 4 shows that the exhaustion (%) of the dyeings on *enVix*® is higher than that on viscose rayon for each of the two dyes and this result is also consistent with the previous work investigating direct dyeing properties [5]. The exhaustion values of two dyes applied reached the saturation levels at low alkaline concentration values of less than 2 g/l presumably because of the excellent dyeing properties of regenerated cellulosic fibres; salt and alkali requirements are generally lower for reactive dyes on regenerated cellulosic fibres than for the corresponding dyeings on cotton.

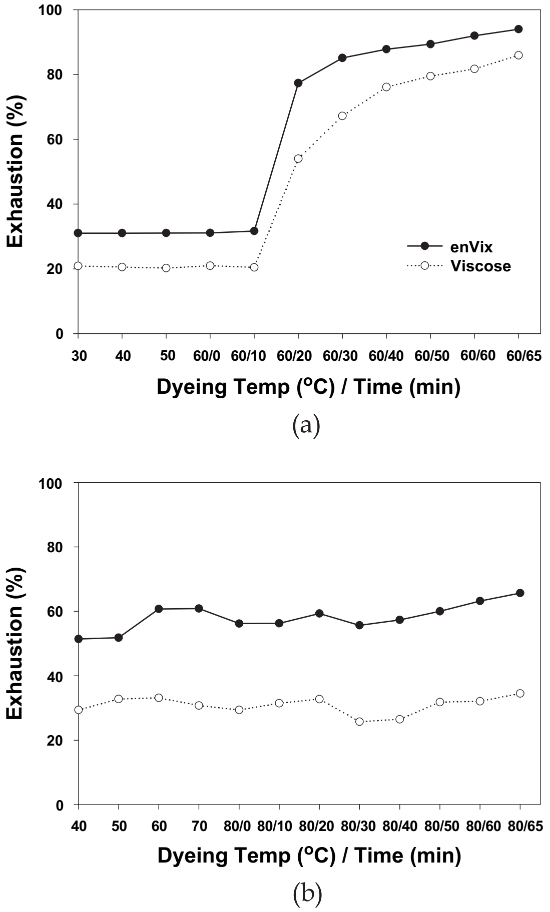


Fig. 6. Exhaustion behaviours of reactive dyes on regenerated cellulosic fibres (dye 2.0% o.w.f., salt 20 g/l, liquor ratio 1:20): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MCT).

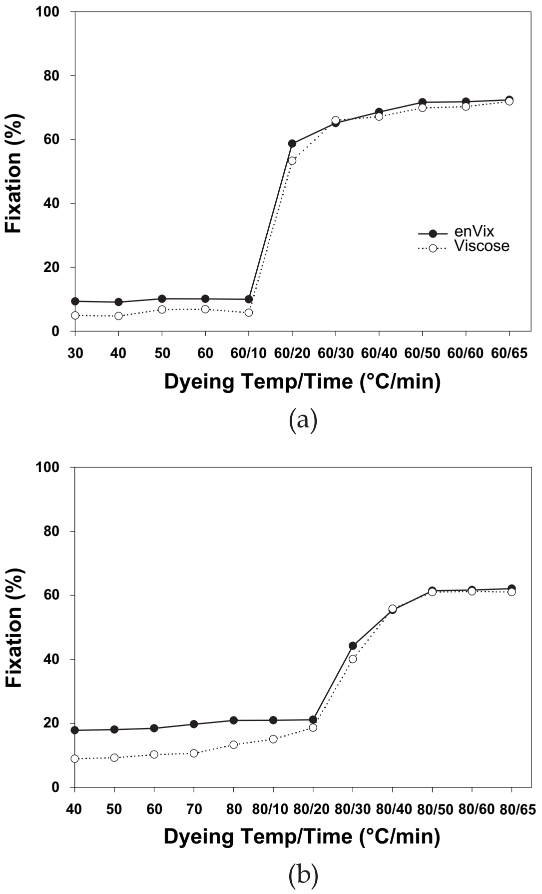
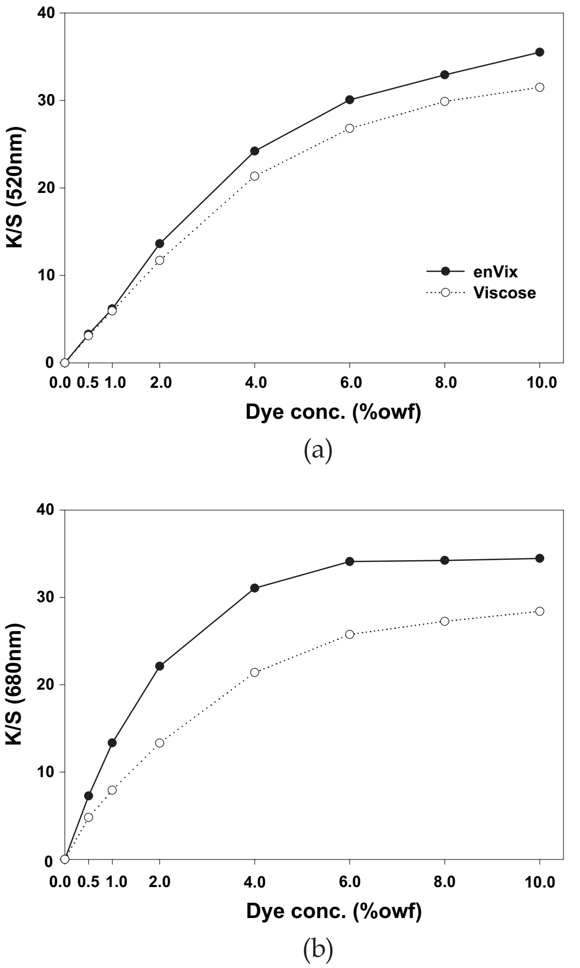


Fig. 7. Fixation behaviours of reactive dyes on regenerated cellulosic fibres (dye 2.0% o.w.f., salt 20 g/l, liquor ratio 1:20): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MCT).

In the case of Reactive Blue 71, it shows high exhaustion even without the presence of alkali although its exhaustion (%) value was not enhanced very much with the addition of alkali probably due to its poor migration properties. It is the size of phthalocyanines that makes them substantive to cellulose when the appropriate amounts of electrolytes are present [10]. Their attachment is through both hydrogen bonds and van der Waals forces since the intensity of the latter increases with increasing molecule size. Their hydrogen bonding capability is also aided by their square and flat molecular structure which enables them to lie along a cellulose chain in register with hydroxyl groups. Therefore, the higher exhaustion (%) compared with that of azo dye (Reactive Red 23) in the absence of alkali is associated with high substantivity due to the larger molecular size.

Fig. 5 shows the liquor ratio effects on the dyeability of the regenerated cellulosic fibres. As expected, the reactive 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 fibre surface decreases, i.e. fewer dye molecules (per unit of time) are adsorbed onto the fibre surface. Equilibrium between adsorption and desorption is thus displaced, desorption becoming stronger, i.e. substantivity is lower. This decrease in substantivity as the liquor ratio was relatively less marked with *enVix*® than viscose rayon especially in the case of C.I. Reactive Red 23. Usually the higher the liquor ratio, the higher is the substantivity required to produce a good colour yield in a reasonable time. It is interesting to note that reproducibility on *enVix*® compared with that of viscose rayon is excellent, being particularly resilient to changes in liquor ratio. The previous work investigating direct dye also drew the similar conclusions [5]; extremely high dye yields on *enVix*® were seen. Once again, it can be seen that *enVix*® is relatively insensitive to process variables.

Fig. 6 shows the exhaustion behaviours of reactive dyes on regenerated cellulose fibres during dyeing. As is to be expected, the dyeing rate of two rayons is not the same because of their supramolecular structures [5]. Different dyeing rates brought about by the difference in physical structures are clearly of practical importance. Predictably, *enVix*® showed faster exhaustion behaviour



(a)

(b)

Fig. 8. Build-up properties of direct dyes on regenerated cellulosic fibres (salt 20 g/l, liquor ratio 1:20): (a) C.I. Reactive Red 23 (VS), (b) C.I. Reactive Blue 71 (MCT).

than viscose rayon, presumably due to the lower crystallinity (%) values and degree of orientation. In all types of cellulose fibres there are 'crystalline' regions (with high parallelism of the cellulose macromolecules) and 'non-crystalline' regions (where the macromolecules are disordered to some extent). The crystallites lie preferentially parallel to the fibre axis and are separated by regions of lower order and intermicellar spaces. The average size of the crystallites and the quantitative ratio of crystallites to regions of lower order are strongly fibre-specific. Only water-swollen intermicellar spaces and regions of lower order of the fibre are accessible to large reactive dyes molecules. It is completely impossible for a dye to diffuse into the highly oriented crystallites. Dyeing therefore only proceeds at the outer walls of the crystallites and in the non-oriented cellulose.

Also, as seen in Fig. 6, it is evident that C.I. Reactive Blue 71 shows much slower dyeing rate than C.I. Reactive Red 23, presumably because the shape of a dye molecule affects its diffusion rate as was mentioned earlier; the phthalocyanine dyes with high substantivity have poor migration properties, since their big size and the strong interaction work against release of dye from the initial point of attachment. Such dyes are more difficult to diffuse and the rate of diffusion, i.e. the mobility of the dye within the cellulose matrix, is thus a decisive factor in the final appearance of the dyeing, particularly as regards exhaustion (%) of the dyeing. Therefore, even after the addition of alkali, the exhaustion (%) is not enhanced very much due to the poor migration property of phthalocyanine dyes [10,11].

Fig. 7 shows the fixation behaviours of reactive dyes on regenerated cellulosic fibres. The differences between two fibres in fixation (%) values were negligibly small compared with those of their exhaustion behaviours, which implies that once the dyes are exhausted, the fixation ratios of the exhausted dyes on two different fibres are similar. However, in terms of the colour yield, *enVix*® exhibited higher K/S values than viscose rayon due to its higher exhaustion value as is seen in Fig. 8.

Further evidence that the dyeability of *enVix*® is better than that of viscose rayon was provided by the results of build-up properties obtained for the two types of dye; as Fig. 8 shows *enVix*® dyed to a much deeper shade than viscose rayon; in the case of C.I. Reactive Blue 71, the colour strength of the dyed rayons reached saturation around the dye concentration of 10.0% o.w.f. which gave a K/S value of around 34.5 for *enVix*® and 28.4 for viscose rayon.

3.3. Fastness properties

Table 2 shows that the fastness to washing (especially the change of the dyed fabric) of 1/1 standard depth dyeings of the reactive dyes on *enVix*® was slightly higher than that of comparable depth dyeings on viscose rayon. The results could be attributed to *enVix*®'s excellent affinity to dyes due to its supramolecular characteristics. Table 3 also reveals that whilst the perspiration fastness of the 1/1 standard depth dyeing of C.I. Reactive Blue 71 on *enVix*® was identical to that of the dye on viscose rayon, the acid perspiration of C.I. Reactive Red 23 was slightly higher than that on viscose rayon. In the case of rubbing fastness, the wet rubbing fastness of the reactive dyes on *enVix*® was slightly better than that on viscose

Table 2
Wash fastness of the reactive dyes on regenerated cellulose fibres

	enVix®		Viscose rayon	
	Change	Staining	Change	Staining
C.I. Reactive Red 23 (VS)	4–5	5[a]	4–5	4–5[a]
C.I. Reactive Blue 71 (MCT)	5	4[b]	4	4–5[b]

[a] Staining on nylon.

[b] Staining on cotton.

Table 3
Perspiration fastness of C.I. Reactive Red 23 on regenerated cellulose fibres

Adjacent fabric	*enVix*®		Viscose rayon	
	Alkali	Acid	Alkali	Acid
Cotton	4	4	4	3–4
Nylon	5	4–5	5	4

Table 4
Rubbing fastness of the reactive dyes on regenerated cellulose fibres

	enVix®		Viscose rayon	
	Dry	Wet	Dry	Wet
C.I. Reactive Red 23 (VS)	5	5	5	4–5
C.I. Reactive Blue 71 (MCT)	5	4–5	5	4–5

rayon although the dry rubbing fastness figures were virtually identical (Table 4). The fastness to light of the two dyed rayons was excellent (a rating of over 5), irrespective of dyes applied.

4. Conclusions

A new regenerated cellulosic fibre was prepared by saponifying at least 75% of the total acetyl groups of a cellulose acetate fibre with a degree of substitution of 2.0 or higher into hydroxyl groups.

The dyeing properties on new regenerated cellulosic fibre, *enVix*®, were compared with that on regular viscose rayon, and shown to be excellent. Careful comparison of the dye yield on two rayons with reactive dyes confirms that there is a correlation between supramolecular structures and the dyeing properties; *enVix*® exhibited higher exhaustion values and better build-up properties than viscose rayon, presumably due to the lower crystallinity (%) and degree of orientation. In addition, it showed stable final colour yields, irrespective of liquor ratio changes in dyeing process, hence reproducibility of dyeing of *enVix*® is expected to be excellent. Fastness properties of reactive dyes on *enVix*® were also slightly better than that on viscose rayon. The results obtained suggest that this novel regenerated cellulosic fibre could be used as an important alternative to conventional viscose rayon although more detailed studies on the new regenerated cellulosic fibre are necessary before any definite conclusions can be drawn.

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