

HBP-NH₂ grafted cotton fiber: Preparation and salt-free dyeing properties

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Abstract

In order to achieve salt-free dyeing on cotton fiber with reactive dyes, an amino-terminated hyperbranched polymer (HBP-NH₂) grafted cotton fiber (HGCF) was prepared by the oxidation of cotton fiber with sodium periodate in water and subsequent grafted with an aqueous solution of HBP-NH₂. Fourier transform infrared spectrophotometry (FTIR) of the HGCF indicated that all aldehyde groups of the oxidized cotton fiber have reacted with amino groups of the HBP-NH₂. As a result, the HGCF fabrics prepared under the optimum conditions displayed markedly enhanced colour strength when dyed with reactive dyes using salt-free dyeing. The washing fastness, rubbing fastness and levelling properties of the dyed HGCF fabrics were also good compared with those obtained by conventional dyeing. The zeta-potential of the HGCF in liquid phase was tested and found to be positive at pHs lower than 6.5. The dyeing behaviour of Reactive Brilliant Yellow A-4GLN on the HGCF was found to follow a Langmuir-type adsorption curve.

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1. Introduction

Periodate oxidation of cotton fiber results in a ring-opened product containing two aldehyde groups formed by oxidative scission at the C-2 and C-3 of the anhydro D-glucopyranose residues in the fiber molecule. The oxidized cotton fiber is an intermediate containing reactive aldehyde groups for various derivatives from cotton fiber. In particular, the oxidized cotton fiber can be converted to various nitrogen-containing derivatives in good yield by reaction at ambient temperature in aqueous medium (Maekawa & Koshijima, 1991). A new cotton fiber with a chitosan coating (CCCF) was prepared by the oxidation of a cotton thread with sodium periodate at 60 °C in water and subsequent treatment with a solution of chitosan in aqueous acetic acid (Liu, Nishi, Tokura, & Sakairi, 2001).

Traditionally, large quantities of salt (30–150 g/l) are needed to overcome the static repulsion between cotton fibers and reactive dyes in order to promote dyeability, but discharges of high electrolyte concentrations are undesirable since increased salinity of the rivers affects the delicate biochemistry of aquatic life. (Wu & Chen, 1993). Salt-free/low-salt dyeing technology using reactive dyes has become a popular topic. Most researchers focus on introducing cationic groups like amino or ammonium groups into cotton fabrics for interactions with anionic dyes. (Zhang et al., 2007; Lewis & Mellroy, 1997; Ma, Zhang, & Tang, 2005; Kin, Yoon, & Son, 2004; Burkinshaw, Mignanelli, Froehling, & Bide, 2000; Lim & Hudson, 2004).

Due to the unique chemical and physical properties, dendrimers and hyperbranched polymers have received much attention over the past two decades and have been applied to coatings, additives, drug and gene delivery, macromolecular building blocks, nanotechnology and supramolecular science. The preparation of dendrimer is

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generally tedious because of various protection, deprotection and purification steps. However, hyperbranched polymer is mostly synthesized by one-step polycondensation (Cao & Yan, 2004; Froehing, 2001). Amino-terminated hyperbranched polymer, characterized by a three-dimensional structure and a large number of imino groups and terminal primary amino groups, was synthesized from methyl acrylate and diethylene triamine by one-step polycondensation. The pretreatment of cotton with the amino-terminated hyperbranched polymers in the presence of citric acid as crosslinking agents can enhance the dyeability of the cotton fibre with reactive dyes (Zhang et al., 2007).

The purpose of the present work is to prepare an amino-terminated hyperbranched polymer (HBP-NH₂) grafted cotton fiber (HGCF) by the reaction of the aldehyde groups of the oxidized cotton fiber with the amino groups of the HBP-NH₂, and to dye the HGCF with reactive dyes in salt-free dyeing. *K/S* values, washing fastness, rubbing fastness and levelling properties of the dyed HGCF fabrics in salt-free dyeing were tested and compared with those properties in conventional dyeing. In addition, in order to study the mechanism of salt-free dyeing on HGCF with reactive dyes, the zeta-potential of HGCF in the liquid phase and the dyeing behaviour of Reactive Brilliant Yellow A-4GLN on the HGCF were examined.

2. Experimental

2.1. Materials

The amino-terminated hyperbranched polymer (HBP-NH₂) was prepared from methyl acrylate and diethylene triamine by melt polycondensation as described in our recent paper (Zhang et al., 2007). All chemicals used for the following investigations were purchased from Sinopharm Chemical Reagent Co. Ltd. (China) and of analytical grade. Cotton fibers and cotton woven fabrics (120 g/m²), which were bleached and scoured were obtained from Huafang Group (Jiangsu, China).

Reactive dyes (Reactive Violet A-5RV, Reactive Brilliant Yellow A-4GLN, Reactive Brilliant Red A-EF, Reactive Deep Blue A-2GLN) were supplied by the Facozol Dye Corp (Shanghai, China). All the reactive dyes used are bifunctional reactive dye with a vinyl sulphone and a monochlorotriazine group. The majority of experiments were carried out using Reactive Brilliant Yellow A-4GLN.

2.2. Oxidation of cotton fiber with sodium periodate

Cotton fiber was immersed in an aqueous solution of sodium periodate (2.0 g/l) at a liquor ratio of 30:1. The solution was oscillated gently at 40 °C for 10–180 min. Then the oxidized cotton fiber was washed with deionized water several times to remove the oxidant, and was used for the next reaction without drying.

2.3. Preparation of HGCF

The oxidized cotton fiber was immersed in HBP-NH₂ aqueous solution (range from 4 to 40 g/l) for 5–60 min at five different temperatures (20, 40, 60, 80 and 100 °C), then washed with tap water several times. The resulting cotton fiber was air-dried at ambient temperature to produce the HBP-NH₂ grafted cotton fiber (HGCF).

2.4. Dyeing procedures

Dyeing with reactive dyes was carried out using a liquor ratio of 50:1. Salt-free dyeing of the HGCF fabric was commenced at 25 °C, and kept at this temperature for 40 min. Fixation was conducted subsequently for 40 min using sodium carbonate (20 g/l) at 60 °C. Conventional dyeing on the original cotton fabric was followed the procedures recommended by the dye manufacturer in the presence of sodium chloride (60 g/l), and sodium carbonate (20 g/l) was also added for dye fixation. After dyeing, the cotton fabric was removed from the bath and rinsed thoroughly in hot water and soaped in a soap solution (5 g/l soap power, 5 min) at 90 °C, and then rinsed thoroughly in tap water and air dried.

2.5. Measurement

2.5.1. FTIR of the oxidized fiber and the HGCF

Infrared spectra of the oxidized cotton fiber and the HGCF were recorded on a Nicolet 5700 FT-IR spectrophotometer using KBr disk. The measurements were performed at 20 °C and a relative humidity of 65%.

2.5.2. Determination of aldehyde group in the oxidized fiber

The aldehyde content in periodate-oxidized cotton samples was determined by Schiff base reaction with hydroxylamine hydrochloride (Marte & Owens, 1956). Hydrochloric acid released from hydroxylamine hydrochloride was titrated by 0.03 M NaOH methanol solution (Qian & Li, 2001). The formula is as follows:

$$\text{Aldehyde content (mmol/g)} = 30 \, v/w$$

Where, *V* is the volume of sodium hydroxide methanol solution used in titrimetry (l); *W* is the mass of oxidized cotton fiber sample (g).

2.5.3. Breaking strength measurements

Breaking strength measurements of fabric were realized at YG026B Electronic fabric strength tester (Ningbo Textile Instrument Factory, China) according to ISO 13934-1. Test length was 30 cm, and speed was 200 mm/min.

2.5.4. Colour measurements

The colour measurements of the dyed samples were performed under D₆₅ illuminant at 10° observer using an Ultrascan XE (HunterLab Co. Ltd., USA) spectrophotometer. The *K/S* values were calculated at the wavelength of maximum absorption (*k*_{max}) for each sample.

K/S values of 20 random points on each sample were measured and then $\delta_{(\lambda)}$ was calculated to evaluate the levelling properties of the dyed samples according to the formulae as described in our recent paper (Zhang et al., 2007). The levelling properties can then be evaluated using $\delta_{(\lambda)}$, the lower $\delta_{(\lambda)}$ meaning better levelness.

2.5.5. Fastness testing

Washing fastness tests were performed according to AATCC61-2003 (3A) using the SW-12A washing fastness tester (Wuxi Textile Instrument Factory, China). Rubbing fastness tests were performed according to AATCC8-2004 using the LFY-304 rubbing fastness tester (Shandong Textile Research Institute, China).

2.5.6. Zeta potential measurements

The HGCF and the original cotton fibre were cut to the length of approximately 1 mm. The 0.10% solids were prepared in 0.0010 M KCl at various pHs and stirred with magnetic stirrer for 10 min. The zeta potentials were then measured using a JS94-H electrophoresis instrument (Zhongchen Instrument Co. Ltd., China).

2.6. Adsorption of reactive dyes onto the HGCF

The HGCF fabrics were dyed with Reactive Brilliant Yellow A-4GLN at a concentration range of 40–640 mg/l for 3 h at 25 °C and 50 °C to determine the adsorption isotherm. In order to quantify the amount of reactive dye adsorbed on the treated cotton fabrics, the concentration of the dye solution was measured using an ultraviolet-visible (UV-vis) spectrophotometer (751MC; Shanghai Analytical Instruments, China) at 426 nm.

3. Results and discussion

3.1. Preparation of HGCF

The preparation process of HGCF is summarized in Scheme 1. The cotton fiber was first oxidized by sodium periodate to cleave the 2,3-vicinal diol of the cellulose

glucose units (1) according to the reported procedure (Maekawa & Koshijima, 1991), giving the so-called oxidized cotton fiber (2). Subsequently, the oxidized cotton fiber was immersed in a HBP-NH₂ aqueous solution, and the resulting aldehyde groups of the oxidized cotton fiber would be coupled with the amino groups of HBP-NH₂ to produce HGCF (3).

The reaction process was monitored by infrared spectroscopy. Fig. 1 shows the spectra of the oxidized cotton fiber and the HGCF. The characteristic absorption band of the oxidized cotton fiber, which clearly appeared at 1730.2 cm⁻¹ due to the stretching vibration of the C=O double bond of the aldehyde group, disappeared after the graft reaction with HBP-NH₂. Meanwhile, an absorption at near 887.5 cm⁻¹, which is observed in the IR spectrum (a) of the oxidized cotton fiber and indicate the formation of a cyclic hemiacetal linkage (Maekawa et al., 1991), is not apparent as shown in the IR spectrum (b) of the HGCF. In addition, the absorption peak at 1569.6 cm⁻¹ in the IR spectrum of the HGCF corresponds to the N–H bending of the primary amine in HBP-NH₂. This finding leads to the conclusion that HBP-NH₂ has been grafted to the oxidized cotton fiber by the reaction of the aldehyde groups of the oxidized cotton fiber with the amino groups of HBP-NH₂.

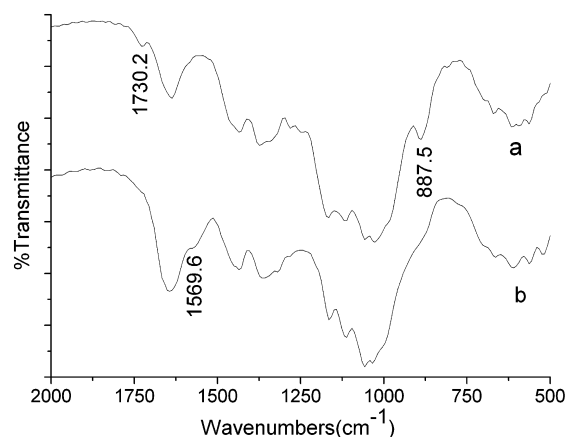
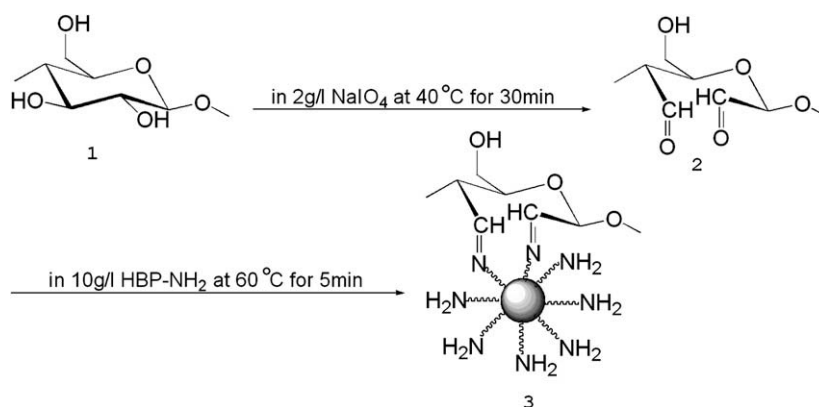


Fig. 1. IR spectra of: (a) oxidized cotton fiber; (b) HGCF.



Scheme 1.

3.2. The optimum condition to prepare HGCF

In order to choose an optimal condition in preparing HGCF, K/S values of the HGCF fabrics prepared under different conditions and dyed with 2% Reactive Brilliant Yellow A-4GLN in salt-free dyeing were tested as a measure of the amount of HBP-NH₂ grafted to the oxidized cotton fiber. Several factors, which may affect the amount of HBP-NH₂ grafted to the HGCF, such as the aldehyde content of the oxidized cotton fiber, concentration of HBP-NH₂ aqueous solution, reaction temperature and reaction time were studied.

Theoretically, increasing temperature, time and oxidant concentration may increase the aldehyde content of the oxidized cotton fiber during the oxidation process, and high aldehyde content may subsequently increase the amount of the HBP-NH₂ grafted to cotton fiber. Consequently, the HGCF can get high colour strength when dyed with 2% Reactive Brilliant Yellow A-4GLN in salt-free dyeing. However, the oxidation by sodium periodate breaks to some extent the crystalline structure of cellulose in the original cotton fiber, therefore, high aldehyde content may weaken the mechanical properties of the oxidized cotton fiber (Zhao & Xia, 2003). Figs. 2 and 3 show the effects of the time in preceded oxidation on the breaking strength and aldehyde content of the oxidized cotton fabrics and the K/S values of the dyed HGCF fabrics. As an exciting result, it is discovered that short time yields adequate aldehyde content for the oxidized cotton fiber to ensure sufficient amount of the HBP-NH₂ grafted to the oxidized cotton fiber for achieving high K/S value when dyed with reactive dyes in salt-free dyeing. To obtain such aldehyde content, cotton fiber is oxidized in 2.0 g/l sodium periodate aqueous solution at 40 °C for 30 min. Certainly, we can further reduce the oxidation time by increasing temperature and oxidant concentration.

Fig. 4 shows the correlation between the K/S values of the dyed HGCF fabrics and the condition used in prepar-

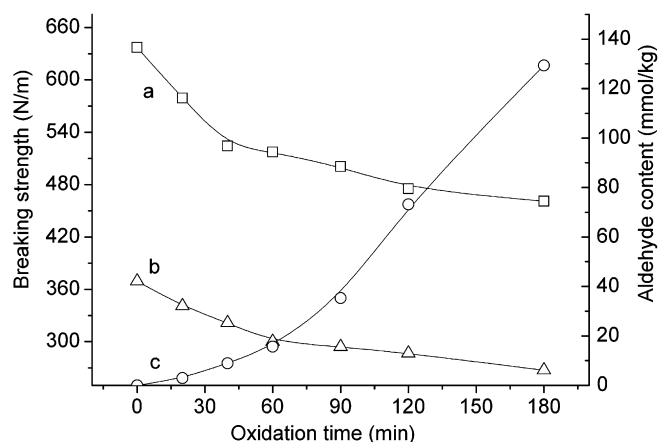


Fig. 2. Change in the breaking strength and aldehyde content of the oxidized cotton fabric against the time of oxidation: (a) warp-wise breaking strength; (b) weft-wise breaking strength; (c) aldehyde content.

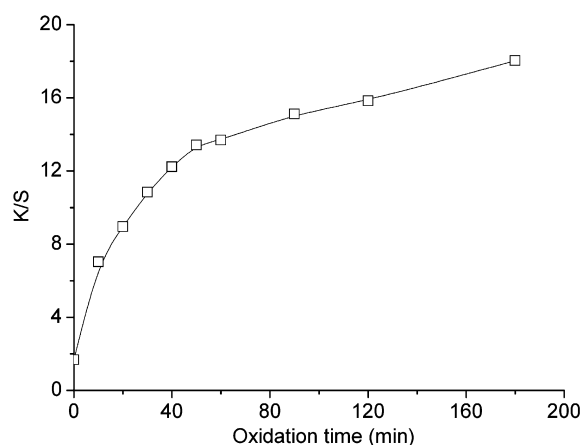


Fig. 3. Relationship between the colour strength of the dyed HGCF fabrics and the time of preceded oxidation.

ing the HGCF fabrics. The general trend was that the amount of the HBP-NH₂ grafted to the HGCF, the reaction temperature, the reaction time and the concentration of HBP-NH₂ aqueous solution increased till the reaction between the aldehyde groups of the oxidized cotton fiber and the amino groups of HBP-NH₂ was completed. From the Fig. 4(a), it seems that 10 g/l HBP-NH₂ aqueous solution can provide enough HBP-NH₂ to react with the oxidized cotton fiber. Fig. 4(b) and Fig. 4(c) indicated that the heat is an important factor, high temperature may speed the reaction between the aldehyde groups of the oxidized cotton fiber and the amino groups of HBP-NH₂ and cut down the reaction time. According to the experimental results, the optimal concentration of the HBP-NH₂ aqueous solution to prepare HGCF is 10 g/l, the optimal reaction temperature is 60 °C and the optimal reaction time is 5 min.

3.3. Dyeing properties of HGCF

Fig. 5 is the colour strength of the HGCF fabrics and original cotton fabrics dyed with 2% owf Reactive Brilliant Yellow A-4GLN in the absence or the presence of electrolyte and alkali, using a competitive dyeing method in which the HGCF fabrics and the original cotton fabrics were dyed in the same dye bath. It clearly shows that the colour strength (K/S) achieved for the HGCF fabrics were very higher than those obtained for the corresponding dyeing of the original cotton fabrics in the absence or presence of electrolyte and alkali. In addition, the colour strength gained for the HGCF fabrics with either electrolyte or alkali absent was much higher than that obtained for the original cotton fabrics in the presence of both electrolyte and alkali. This could be explained with the positively charged imino and amino groups in the HGCF fabrics (Gupta & Haile, 2007).

K/S values, washing fastness, rubbing fastness and leveling properties of the dyed HGCF fabrics in salt-free dyeing and those properties of the original cotton fabrics in

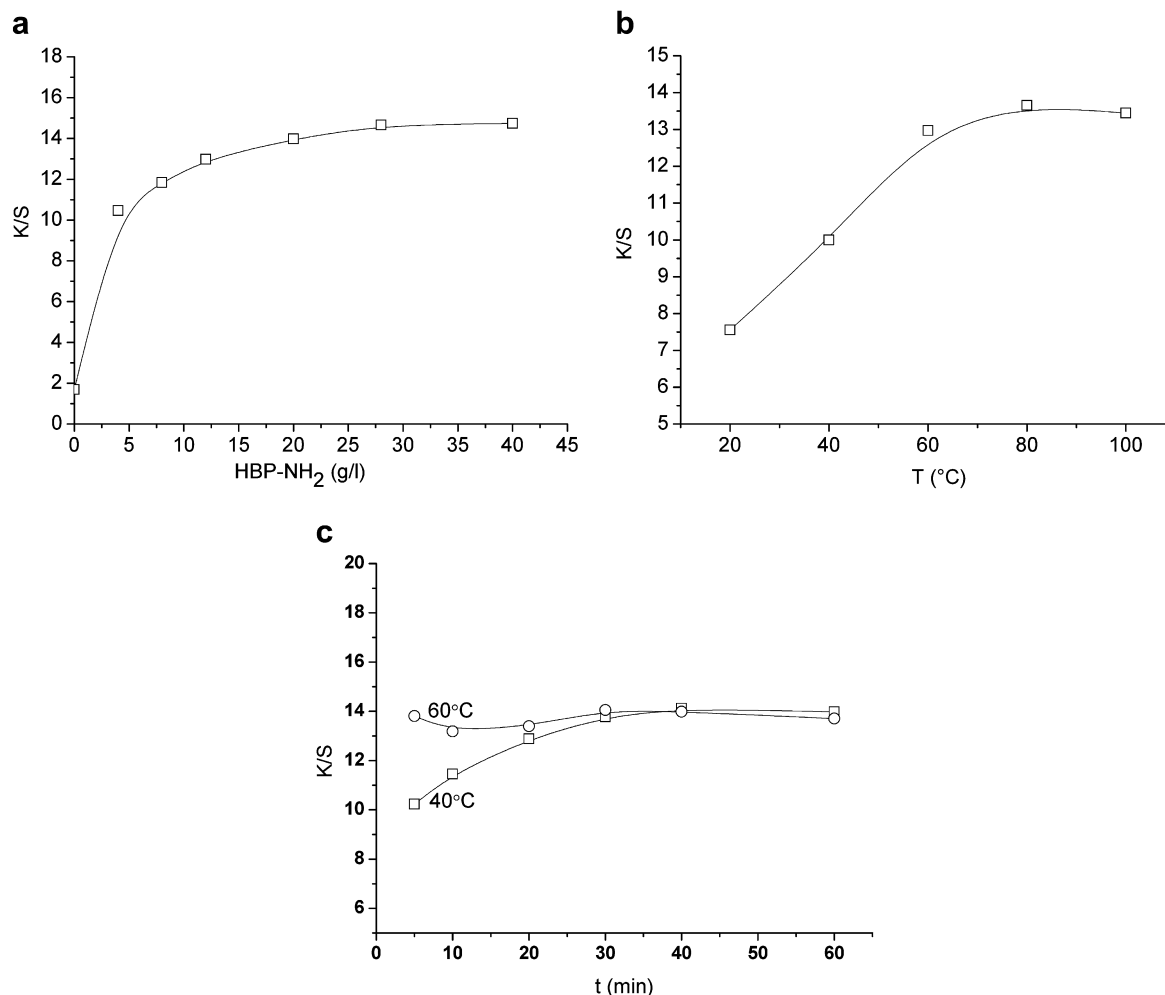


Fig. 4. Correlation between the colour strength of the dyed HGCF fabrics and the concentration of HBP-NH₂ aqueous solution, reaction temperature and reaction time used in preparing HGCF fabrics.

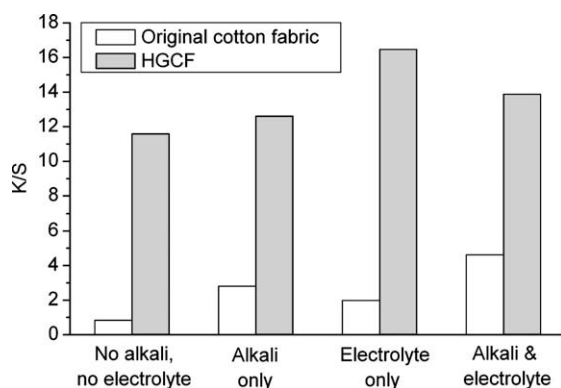


Fig. 5. Colour strength of 2% owf Reactive Brilliant Yellow A-4GLN dyeings produced in the absence or the presence of electrolyte and alkali.

conventional dyeing were tested. The results are shown in Table 1. Compared with the original cotton fabrics in conventional dyeing, the HGCF fabrics displayed markedly enhanced colour strength when dyed with four reactive dyes using salt-free dyeing and had good levelness, washing fastness and rubbing fastness. Therefore, if the cotton fab-

rics were grafted with the amino-terminated hyperbranched polymer, salt-free dyeing would be possible.

3.4. Zeta potential of HGCF

In order to understand the mechanism of salt-free dyeing on HGCF with reactive dyes, the characteristic surface charges of the HGCF and the original cotton fiber were investigated through zeta potential measurement. The results are shown in Fig. 6. Compared with the zeta potential of the original cotton fiber in liquid phase, the zeta potential values of the HGCF increased over the entire pH range, rendering the surfaces positively charged at pHs lower than 6.5. This is probably attributed to the presence of imino groups and amino groups on HGCF (Ma et al., 2005).

3.5. Investigation of dyeing behaviour on HGCF

To investigate the adsorption pattern of Reactive Brilliant Yellow A-4GLN on the HGCF in the absence of

Table 1

Comparison of dyeing properties of the HGCF fabrics in salt-free dyeing and original cotton fabrics in conventional dyeing (all dye concentrations were 2% owf)

Dyestuff	Cotton fabrics	K/S	Levelness $\delta_{(A)}$	Rubbing fastness		Washing fastness	
				Dry	Damp	Fading	Staining
Reactive Violet A-5RV	HGCF	4.50	0.0538	4–5	3–4	3–4	4–5
	Original cotton	2.95	0.0623	4–5	4	4	4–5
Reactive Deep Blue A-2GLN	HGCF	16.64	0.3015	4	3	4–5	4–5
	Original cotton	13.46	0.6503	4	3–4	4–5	4
Reactive Brilliant Red A-EF	HGCF	16.30	0.3390	3–4	3–4	4–5	3–4
	Original cotton	12.06	1.4539	4	4	4–5	3
Reactive Brilliant Yellow A-4GLN	HGCF	16.72	0.3375	4	3–4	4–5	4–5
	Original cotton	4.73	0.2740	4	3–4	4–5	4–5

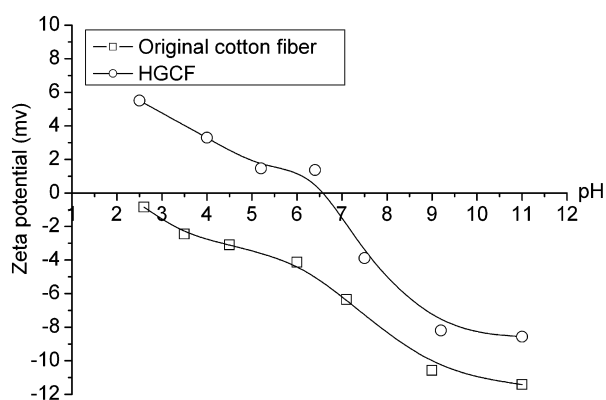


Fig. 6. Zeta potential values of the HGCF and original cotton fiber.

the electrolyte, dyeings were carried out at a concentration range of 40–640 mg/l for 3 h at 25 and 50 °C. The concentration of the dye adsorbed on the fibre ($[D_f]$) and the dye remaining in the dyebath ($[D_s]$) were calculated (Janhoma, Griffithsb, Wataneska, & Wataneska, 2004) to draw the adsorption isotherms for reactive Brilliant Yellow A-4GLN on HGCF. The curves at 25 and 50 °C in Fig. 7(a) show that the adsorption capacity of the dye decreased with increasing temperature, thereby indicating that the process is an exothermic one. Fig. 7(b) is the curves plotted $1/[D_f]$ against $1/[D_s]$, where the Langmuir model fit-

ted the experimental data very well with high correlation coefficients ($R_{25^\circ\text{C}} = 0.9935$, $R_{50^\circ\text{C}} = 0.9960$). It indicates that the adsorption pattern of Reactive Brilliant Yellow A-4GLN on the HGCF in the absence of the electrolyte is Langmuir-type adsorption. Similar observations were reported for the adsorption of lac dye on cotton pretreated with chitosan (Rattanaphani, Chairat, Bremner, & Rattanaphani, 2007). The typical Langmuir-type adsorption of Reactive Brilliant Yellow A-4GLN on HGCF demonstrates the mechanism of salt-free dyeing on the HGCF. In the dyeing of the HGCF fabrics in the absence of electrolyte, the adsorption of the reactive dyes is assisted by ionic attraction between the cationic $-\text{NH}_3^+$ sites on the HGCF and the disulphonated anions on the reactive dyes.

4. Conclusions

In conclusion, an amino-terminated hyperbranched polymer (HBP- NH_2) grafted cotton fiber (HGCF) was obtained by the reaction between the aldehyde groups on the oxidized cotton fiber and the amino groups of the HBP- NH_2 . In order to ensure that the amount of the HBP- NH_2 grafted onto the oxidized cotton fiber is sufficient for achieving salt-free dyeing with reactive dyes on the HGCF and to preserve the breaking strength of the oxidized cotton fabrics, it requires the following optimal con-

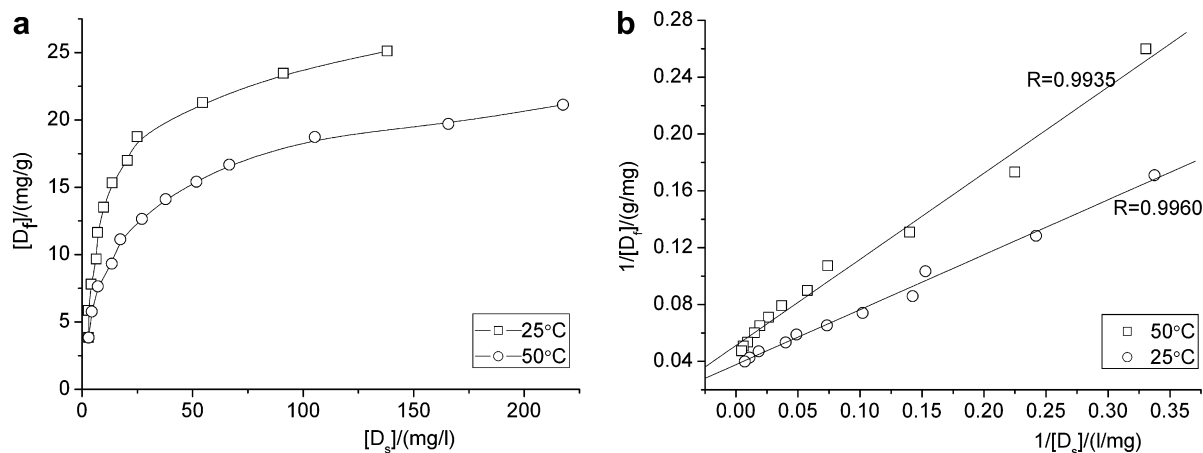


Fig. 7. Adsorption isotherms for Reactive Brilliant Yellow A-4GLN on HGCF in salt-free dyeing.

ditions: the aldehyde content of the oxidized cotton fiber: 8 mmol/kg; the concentration of the HBP-NH₂ aqueous solution: 10 g/l, the reaction temperature: 60 °C; and the reaction time: 5 min.

The HGCF prepared under optimal conditions and dyed with reactive dyes in salt-free dyeings showed markedly enhanced colour strength (*K/S*). Satisfactory levelness, washing fastness and rubbing fastness were also obtained. Grafting the HBP-NH₂ to the oxidized cotton fiber may be a possible means to achieve salt-free dyeing with reactive dyes.

The zeta potential of the HGCF in liquid phase and the typical Langmuir-type adsorption of Reactive Brilliant Yellow A-4GLN on the HGCF demonstrate the mechanism of salt-free dyeing on the HGCF. In the dyeing of the HGCF fabrics in the absence of electrolyte, the adsorption of the reactive dyes is assisted by ionic attraction between the cationic -NH₃⁺ sites on the HGCF and the disulphonated anions on the reactive dyes.

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