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Regenerated cellulose fibers spun-dyed with carbon black/latex composite dispersion

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ABSTRACT

A carbon black (CB)/latex composite was prepared by the method of miniemulsion polymerization for use as a colorant for spun dyeing of regenerated cellulose fibers. Analysis of experimental results revealed that the CB/latex composite had a small particle size and a narrow particle size distribution which were important to ensure a stable dispersion being later added to spinning solution. A good stability of the prepared CB/latex composite dispersion in the spinning solution indicated that it was highly possible to use the CB/latex composite as a colorant for spun dyeing of regenerated cellulose fibers. When a 3.5% mass ratio of CB/latex composite to cellulose was used for spun dyeing, the spun-dyed fibers had the highest tensile strength, breaking elongation and color strength. The rubbing and washing color fastnesses of spun-dyed regenerated cellulose fibers could satisfy requirements of most textiles. This study provided a new insight into producing spun-dyed regenerated cellulose with a novel colorant.

1. Introduction

Spun-dyed regenerated cellulose fibers have attracted more and more people's attention due to its many advantages over the conventional post-dyeing, especially in reducing the environmental impacts and improving the dyeing levelness (Hao, Wang, Liu, & Liu, 2012; Kulpinski, Namyslak, Grzyb, & Lis, 2012; Manian, Ruef, & Bechtold, 2007; Schoenbach, Weissert, & Teige, 1967; Sui et al., 2012). Pigment is an important kind of colorants in spun-dyeing of regenerated cellulose fibers. However, commercial pigments can greatly affect the spinning process and the microstructure, surface morphology, color performance and mechanical properties of regenerated cellulose fibers due to their large particles and uneven distribution (Broda, Gawlowski, Slusarczyk, Wlochowicz, & Fabia, 2007; Fu, Luo, Yao, Tian, & Wang, 2013b; Hao et al., 2012; Mamaza & Folarsnmi, 1996; Marcincin, 2002; Nypelo, Pynnonen, Osterberg, Paltakari, & Laine, 2012). Therefore, modification is required for pigments to be applied for spun-dyeing.

Milling pigments with the aid of polymeric dispersants is a common method to produce pigment dispersions by which the dispersants build voluminous shells to intensify the charges on pigment surface, thus avoiding the flocculation and coagulation of pigment in aqueous media (Zhou, Yu, Xi, & Chen, 2003). Nowadays, a variety of structured polymeric dispersants have been developed for this purpose (Ellen, Stefan, & Christian, 1999; Nsib, Ayed, & Chevalier, 2007; Xu, Liu, Du, Fu, & Liu, 2012). However, pigment dispersions prepared by this method show poor stability in spinning solutions of regenerated cellulose fibers due to the high concentrations of electrolyte and sodium hydroxide in spinning solutions (Schoenbach et al., 1967; Stanley, Budworth, & Leonard, 1964), and as such pigments form flocculation or coagulation in spinning solutions to greatly impair the quality of spun-dyed regenerated cellulose fibers (Dong & Bhattacharyya, 2012; Fu, Du, Wang, Tian, & Xu, 2013a; Marcincin, Hricova, & Lucivjansky, 2005; Roy & Bhowmick, 2012). Therefore, it is desirable to find an effective method to prepare pigment dispersions that can be applied for spun-dyeing of regenerated cellulose fibers.

Encapsulating pigments with polymeric materials may be an alternative method for this purpose because the polymers are hard to be peeled off so as to maintain a high stability of pigment particles in regenerated cellulose fiber spinning solution (Fu et al., 2013a), and also they can take part in film-forming around pigment particles to help improve the color fastnesses of spun-dyed regenerated cellulose fibers. At present, lots of techniques have been devised for pigment encapsulation such as emulsion or miniemulsion polymerization (Lelu, Novat, Graillat, Guyot, & Bourgeat-Lami, 2003; Lu, Ramos, & Forcada, 2007; Ni, Sheibat-Othman, Shan, Fevotte, & Bourgeat-Lami, 2005; Steiert & Landfester, 2007; Tiarks,

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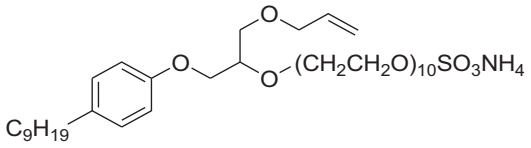

Chart 1. Chemical structure of DNS86.

Landfester, & Antonietti, 2001), phase separation (Fu & Fang, 2007), *in situ* polymerization (Liu, Ye, & Tang, 2007), layer-by-layer assembly (Yuan, Xing, Gun, & Wu, 2008), sol–gel (Li, Pi, Wen, Cheng, & Yang, 2008; Yuan, Zhou, Wu, & You, 2006), and free-radical precipitation polymerization (Fu & Xu, 2010), of which miniemulsion polymerization has been preferably employed for its attractive advantages (El-sayed, Kamel, Morsy, & Taher, 2012).

However, a common emulsifier used in miniemulsion polymerization is problematic for producing a stable pigment/latex dispersion because it may be desorbed from the latex, especially in highly alkaline solutions at high temperatures, which cannot satisfy the requirement of spun-dyeing of regenerated cellulose fibers. It is thus necessary to find an appropriate emulsifier for use in miniemulsion polymerization for preparation of the pigment/latex dispersion. A polymerizable dispersant can be used as an emulsifier in miniemulsion polymerization, which also plays a role of monomer in copolymerization to form a covalent bond on surface of the formed latex, and thus overcome the drawbacks of the common emulsifier (Taniguchi, Takeuchi, Kobaru, & Nakahira, 2008; Zhao et al., 2006). To the best of our knowledge, the polymerizable dispersant is rarely reported to be used in miniemulsion polymerization for preparing a pigment/latex dispersion as colorants for spun-dyeing of regenerated cellulose fibers.

In this study, we proposed to use a polymerizable dispersant as an emulsifier in miniemulsion polymerization for encapsulation of carbon black (CB) with latex. The CB/latex composite dispersion was used as a colorant for spun-dyeing of regenerated cellulose fibers. The performance of spun-dyed regenerated cellulose fibers was further investigated. This study will provide new insight into spun dyeing of regenerated cellulose fibers with the carbon black/latex composite dispersion.

2. Experimental

2.1. Materials

CB (150 m^2/g specific surface area) was purchased from Mitsubishi Chemical Co. Ltd., Japan. Allyloxy nonyl phenoxy propanol polyoxyethylene ether ammonium sulfonate (DNS86) (Chart 1) was purchased from Foshan Kodi Industries Co. Ltd., China. Methyl methacrylate (MMA) was purchased from Shanghai Chemical Reagent Co. Ltd., China, and purified by passing through an inhibitor-removal column. Ammonium persulfate (APS), sodium hydroxide (NaOH) and hydrochloric acid (HCl) were of AR grade and purchased from Shanghai Chemical Reagent Co. Ltd., China. Regenerated cellulose fiber spinning solution (cellulose concentration 8.0%) was supplied as a gift by Shandong Hailong Industries Co. Ltd., China. Distilled water was used in all the experiments.

2.2. Preparation of CB/latex composite dispersion

7.5 g DNS86 was dissolved into 202.5 g distilled water, followed by the addition of 30 g CB. The mixture was stirred at 800 r/min for 30 min, and then homogenized with a bead mill (Minizeta 03E, Netzsu, Germany) at 2000 r/min for 2 h to give DNS86-dispersed CB dispersion.

7.5 g MMA was slowly dropped into the phase of DNS86-dispersed CB dispersion, mixing on a bead mill for 30 min at room temperature. The dispersion was transferred to a 4-neck flask equipped with stirrer, thermometer and condenser. 52.5 g water solution of APS (0.35%) was dropwise added to the flask at 78 °C. Polymerization was carried out for 5 h. The CB/latex composite dispersion was obtained by filtering to remove a minor fraction of agglomerates through a paper filter (1 μm) under vacuum conditions.

2.3. Spun-dyeing of regenerated cellulose fibers

A certain amount of CB/latex composite dispersion was mixed with regenerated cellulose spinning solution under adequate stirring to give a homogenous mixture. The regenerated cellulose fibers (133.3dtex/30f with a linear winding speed of 81 m/min and elongation rate 18%) were spun by extruding the mixture into an acid bath containing 132.0 g/L H_2SO_4, 320 g/L Na_2SO_4, and 9.0 g/L $ZnSO_4$ at 52 °C.

2.4. Instrumentation

2.4.1. Transmission electron microscope (TEM)

A sample was diluted with distilled water to 2000 times, and then placed on a 400-mesh carbon-coated copper grid for air drying. Morphology observation was performed on a JEM-100SX transmission electron microscope.

2.4.2. Scanning electron microscope (SEM)

All samples of the spun-dyed regenerated cellulose fiber were sputter-coated with gold. SEM was performed a Zeiss EVO scanning electron microscope.

2.4.3. Digital scanning microscope (DSM)

The distribution of particles in regenerated cellulose spinning solutions and the surface of spun-dyed regenerated cellulose fiber were observed under a Zeiss DSM 950 digital scanning microscope. DSM photos were taken at a magnification of 2000×.

2.4.4. Dynamic light scattering method (DLS)

A sample was prepared by diluting the original dispersion with distilled water to 2000 times. The particle size (D) and its distribution were determined by dynamic light scattering method (DLS) using a Malvern Zetasizer Nano ZS90 instrument at 25 °C.

2.4.5. Viscometry

The viscosity of a dispersion sample was measured using DV-III Rheometer (Brookfield Instruments, America).

2.4.6. Tensile tests

Tensile tests were carried out by a single-fiber strength tester at 25 °C and 50% humidity according to GB/T 3921-2008. Seven specimens were tested with an Instron universal mechanical testing machine (Norwood, MA) with a load of 1 mN at a grip separation speed of 50 mm/min.

2.4.7. X-ray diffraction (XRD) analysis

The X-ray diffraction (XRD) spectra of the samples were determined by XRD (ARL-X' TRA) using CuKa as radiation at a wavelength of λ = 1.54183 Å. The generator settings were 40 kV and 35mÅ. The diffraction data were collected over a 120 min range of 3°–90°, with a step width of 0.02° and a counting time of 1 s per step. Samples were prepared by spreading the powders into the holder (about 3 mm thick) and pressing slightly with a glass slide to ensure a flat surface.

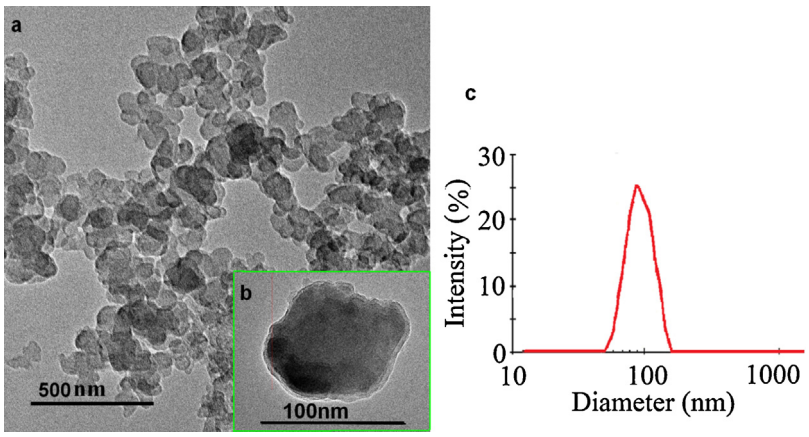

Fig. 1. Morphology and particle size distribution of CB/latex composite dispersion (a, b) TEM photo, (c) particle size distribution measured by DLS.

2.4.8. Color measurement

The reflectance of fabric made from the spun-dyed regenerated cellulose fiber was measured using an X-rite 8400 spectrophotometer (X-rite Incorporated, America) under illuminant D65 and 10° standard observers. The color strength (K/S) was calculated from the reflectance at 540 nm using the well-known Kubelka–Munk equation as given in Eq. (1),

$$\frac{K}{S} = \frac{(1-R)^2}{2R} - \frac{(1-R_0)^2}{2R_0} \quad (1)$$

where R and R_0 are the reflectance of the colored and uncolored cellulose membrane.

2.4.9. Evaluation of color fastness

The color fastnesses to rubbing and washing were evaluated according to the standard methods of GB/T 3921-2008 and GB/T 3922-1995, respectively.

3. Results and discussion

3.1. Morphology and stability to pH of CB/latex composite dispersion

Fig. 1 shows the morphology and the particle size distribution of CB/latex in the dispersion. It can be seen that CB/latex particles are small and uniformly distributed in aqueous media. The thickness of encapsulation layer is observed under TEM photo to be about 5 nm, which indicates that the miniemulsion polymerization is an effective method to prepare CB/latex composite dispersion with core–shell structure.

Since spun dyeing process of regenerated cellulose fiber involves in strong alkaline and acidic conditions, a prerequisite for spun dyeing of regenerated cellulose fiber is that the dispersion needs to have a good stability to pH. In this paper, the stability of CB/latex composite dispersion to pH (S_{pH}) was evaluated in terms of the change in particle size with respect to its initial particle size, as given by Eq. (2),

$$S_{pH} = \frac{D_{pH} - D_0}{D_0} \times 100\% \quad (2)$$

where D_0 is the particle size of the original CB/latex composite dispersion and D_{pH} is the particle size of the CB/latex composite dispersion of which pH was adjusted to a target value within the range from 2 to 14. A smaller value of S_{pH} indicates a higher stability to pH.

Fig. 2 shows that the value of S_{pH} is overall less than 4% in the range from pH 2 to 14, which indicates that the CB/latex composite dispersion has high stability to pH value. In addition, it seems that the stability of the CB/latex composite dispersion was slightly decreased as pH increased from 8 to 14. This could be most likely ascribed to the fact that the increasing concentration of

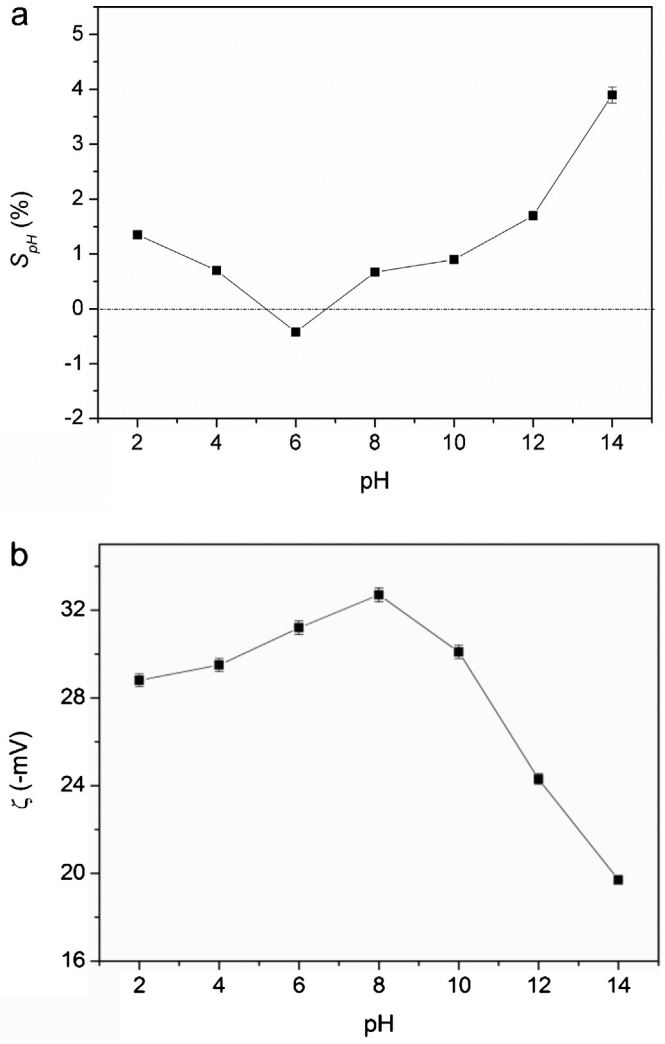

Fig. 2. Effect of pH value on (a) change rate of particle size and (b) zeta potentials of CB/latex composite dispersion.

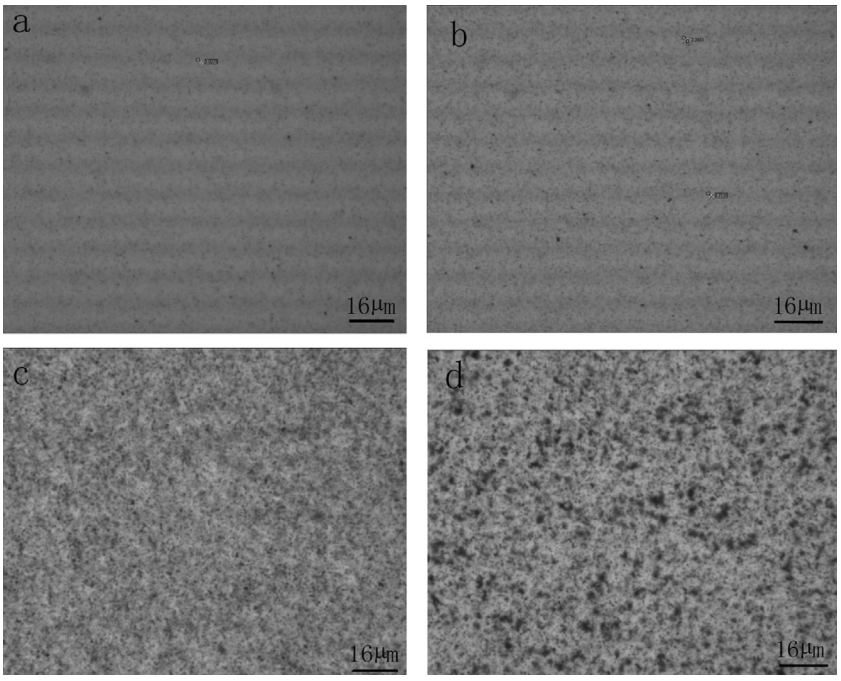

Fig. 3. Microscopic images of regenerated cellulose fiber spinning solution with the addition of different dispersions (mass ratio of CB and fiber 3.5%), (a) CB/latex composite dispersion, (b) CB/latex composite dispersion and stored for 3 days, (c) DNS86-dispersed CB dispersion and (d) DNS86-dispersed CB dispersion and stored for 3 days.

sodium ions compressed the double electric layer of the CB/latex, resulting in a decrease in zeta potential as shown in Fig. 2b.

3.2. Stability of CB/latex composite dispersion in regenerated cellulose fiber spinning solutions

Spun-dyeing of regenerated cellulose fiber requires that the CB/latex composite dispersion has a good stability in the spinning solution. The stability of the CB/latex composite dispersion in spinning solution of regenerated cellulose fiber was qualitatively assessed by microscopy. As shown in Fig. 3, no apparent agglomerate was observed in the mixture of the CB/latex composite dispersion and the spinning solution of regenerated cellulose fiber even for 3-day storage while a large amount of CB agglomerates was observed in the mixture of DNS86-dispersed CB dispersion and the spinning solution. This result indicates that the CB/latex composite dispersion had better stability in the spinning solution of regenerated cellulose fiber. The reason was that, in CB/latex composite dispersion, DNS86 was bonding to the latex through the covalent bond and thus hardly peeled off from the CB surface.

Additionally, the stability of the CB/latex composite dispersion in the spinning solution of regenerated cellulose fiber can also be quantitatively assessed by the change in apparent viscosity for various storage time as given in Eq. (3),

$$M_t = \frac{|v_t - v_0|}{v_0} \times 100\% \quad (3)$$

where v_0 and v_t are the apparent viscosities of the mixture of 5% dispersion and 95% spinning solution before and after storage for a target time, respectively. A smaller value of M_t indicates a better stability of the dispersion in the spinning solution. As shown in Fig. 4, the mixture of the CB/latex composite dispersion and the spinning solution exhibited a value of M_t less than 4% over the 10-h storage time, indicating that the CB/latex composite dispersion had an excellent stability in the spinning solution. However, the mixture of the DNS86-dispersed CB dispersion and the spinning solution exhibited an increasing value of M_t with an increase in storage time, indicating an unstable mixture in which CB tended to agglomerate with an extension of the storage time. These results were consistent with the microscopic observation shown in Fig. 3.

3.3. Properties of spun-dyed regenerated cellulose fiber

3.3.1. Tensile properties

Fig. 5 shows the effect of the CB content on the tensile strength and breaking elongation of spun-dyed regenerated cellulose fiber. As can be seen from the figure, the tensile strength and breaking elongation of the spun-dyed regenerated cellulose fiber increased and reached a relatively constant level as the CB content increased when the CB/latex composite dispersion was used as a colorant, but exhibited no apparent change over the initial range of CB content and reduced significantly as the CB content continuously increased when the DNS86-dispersed CB dispersion was used as a colorant. It

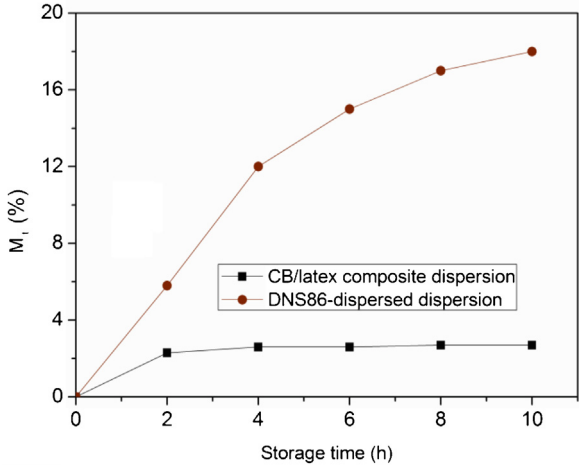

Fig. 4. Effect of storage time on apparent viscosity of mixture prepared from regenerated cellulose fiber spinning solution and dispersion, mass ratio of CB and fiber 3.5%.

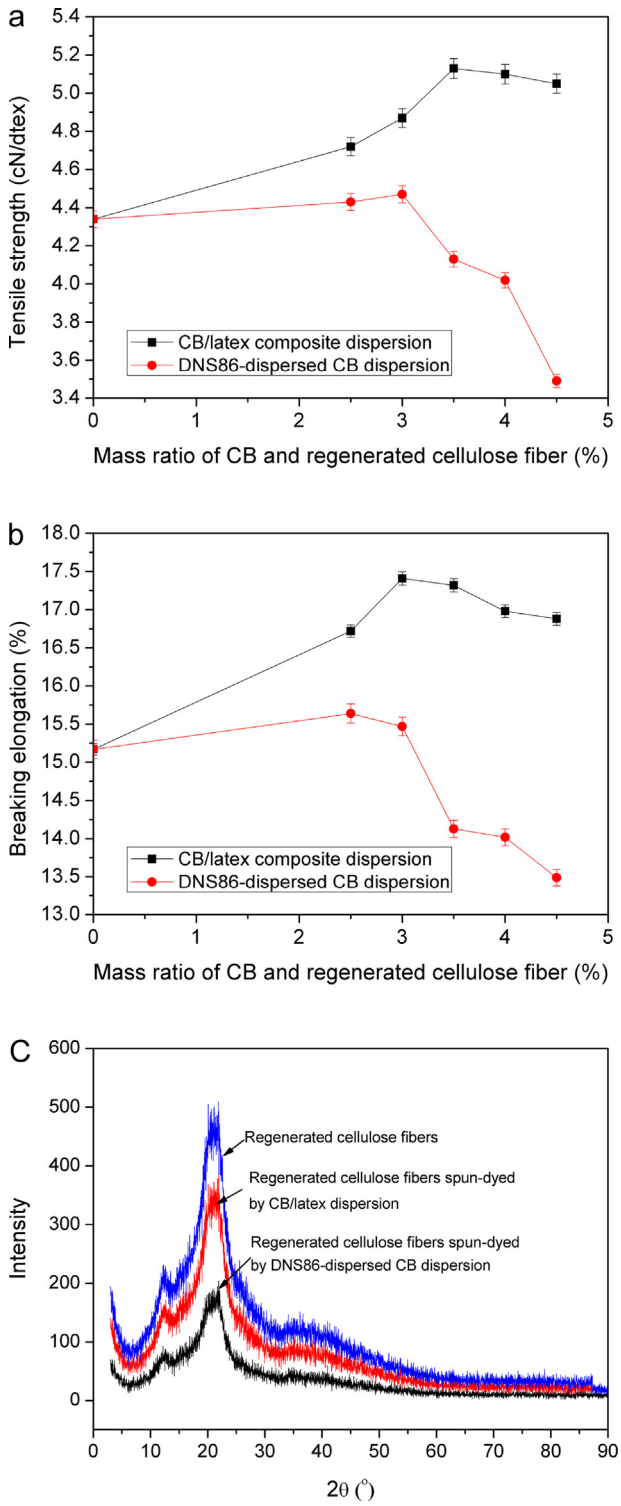

Fig. 5. Effect of mass ratio of CB and fiber on (a) tensile strength, (b) breaking elongation of the regenerated cellulose fibers and (c) the XRD of the spun-dyed regenerated cellulose fibers.

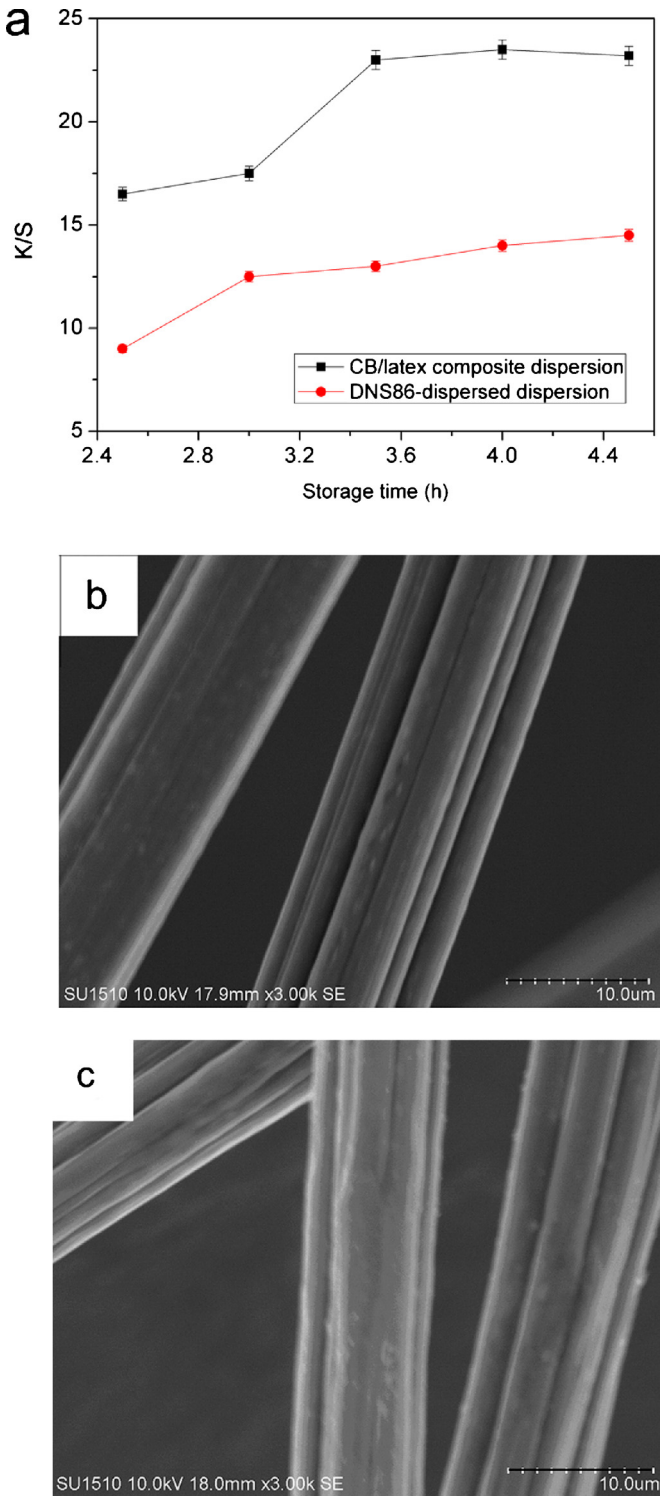

Fig. 6. (a) Effect of CB content on *K*/*S* value of the fabrics weaved by spun-dyed regenerated cellulose fibers; SEM photo of spun-dyed regenerated cellulose fibers by (b) CB/latex composite dispersion (mass ratio of CB and fiber 3.5%) and (c) DNS86-dispersed CB dispersion, mass ratio of CB and fiber 3.5% (mass ratio of CB and fiber 3.5%).

was thought that the particle morphology and the particle amount in fibers would be responsible for the change of tensile properties of the spun-dyed regenerated cellulose fibers. The CB/latex composite dispersion brought a certain amount of particles in smaller size that most likely produced a plasticizing effect on the spun-dyed regenerated cellulose fibers while the DNS86-dispersed CB dispersion brought a greater amount of particles in larger size that might actually destroy the degree of crystalline and orientation of the fibers so as to reduce the attractive forces among the molecular chain (as shown in Fig. 5c). Therefore, the CB/latex composite dispersion was superior to the DNS86-dispersed CB dispersion as a colorant for use in spun-dyeing of regenerated cellulose fibers for enhancing the tensile properties.

Table 1
Color fastness of fabrics weaved from spun-dyed regenerated cellulose fibers with CB/latex composite dispersion and DNS86-dispersed CB dispersion.

Samples spun-dyed by	Rubbing fastness (grade)		Washing fastness (grade)	
	Dry	Wet	Color changing	Staining
CB/latex composite dispersion	4–5	4	4–5	5
DNS86-dispersed CB dispersion	3–4	3	4	5

3.3.2. Color strength

Fig. 6a shows the color strength (K/S) of the fabrics woven from the spun-dyed regenerated cellulose fibers with a various amount of CB content. It is indicated that the CB/latex composite dispersion was superior to the DNS86-dispersed CB dispersion in producing a deeper color when they were used for the same CB content. This may be caused by the excellent stability of CB/latex composite dispersion in the regenerated cellulose fiber spinning solutions. It can be concluded from Fig. 6 that a 3.6% mass ratio of CB to fiber was adequate to give the maximum color strength.

Fig. 6b and c shows the surface morphology of spun-dyed regenerated cellulose fibers. It can be seen that the regenerated cellulose fiber spun-dyed with the CB/latex composite dispersion exhibited a smoother surface morphology than that spun-dyed with the DNS86-dispersed CB dispersion. This is most likely due to the excellent stability of the CB/latex composite dispersion in the spinning solution which ensured the carbon particles to be uniformly introduced onto the surface of regenerated cellulose fiber during the spun-dyeing. Additionally, the latex coated onto CB surface could take part in the film-forming, which also helps to produce a smooth surface for the regenerated cellulose fibers.

3.3.3. Color fastness

Table 1 shows the rubbing and washing fastnesses of spun-dyed regenerated cellulose fibers. Compared to the DNS86-dispersed CB dispersion, the CB/latex composite dispersion used as a colorant for spun dyeing of regenerated cellulose fibers provided textiles with enhanced color fastnesses. An important reason is that the latexes coated onto CB surface might form a film around fiber surface so as to improve the color fastness.

4. Conclusions

The CB/latex composite dispersion was prepared by miniemulsion polymerization method using a polymerizable dispersant. The CB/latex composite dispersion showed an outstanding stability to pH and to the spinning solution of regenerated cellulose fiber, and was suitable to be used as a colorant for spun-dyeing of regenerated cellulose fiber. The spun-dyed regenerated cellulose fiber by CB/latex composite dispersion had enhanced tensile properties and the fabrics woven from the spun-dyed regenerated cellulose fiber showed excellent color fastnesses and color strength.

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