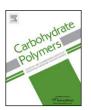
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# Properties of alginate fiber spun-dyed with fluorescent pigment dispersion



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#### ABSTRACT

Spun-dyed alginate fiber was prepared by the spun-dyeing method with the mixture of fluorescent pigment dispersion and sodium alginate fiber spinning solution, and its properties were characterized by SEM, TGA, DSC, and XRD. The results indicate that fluorescent pigment dispersion prepared with esterified poly (styrene-alt maleic acid) had excellent compatibility with sodium alginate fiber spinning solution, and small amount of fluorescent pigment could reduce the viscosity of spun-dyed spinning solutions. SEM photo of spun-dyed alginate fiber indicated that fewer pigment particles deposited on its surface. TGA, DSC, and XRD results suggested that thermal properties and crystal phase of spun-dyed alginate fibers had slight changes compared to the original alginate fibers. The fluorescence intensity of spun-dyed alginate fiber reached its maximum when the content of fluorescent pigment was 4%. The spun-dyed alginate fiber showed excellent rubbing and washing fastness.

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#### 1. Introduction

Alginate fiber derived from brown algae consists of  $\beta$ -(1-4)-linked-D-mannuronic acid (M) and  $\alpha$ -L-guluronic acid (G) segments (Chart 1) (de Kerchove Alexis & Elimelech, 2006; Khan & Ahmad, 2013; Turco et al., 2011). Presently, alginate fibers are extensively used in wound care due to their potential bioactivity, non-toxicity, and biocompatibility (Knill et al., 2004; Lian, Wu, Zhou, & Wang, 2011; Mikolajczyk, Wolowska-Czapnik, & Bogun, 2004; Mikolajczyk, Boguń, Kurzak, & Szparaga, 2009; Yimin, 2008; Lian et al., 2011). During the wound care process, the moist gel formed by ions exchange between Ca<sup>2+</sup> and Na<sup>+</sup> when alginate fibers interact with the wound exudates, thereby preventing fiber entrapment in the wound (Lihong et al., 2005, 2006; Murakami et al., 2010; Thomas, Harding, & Moore, 2000).

With the increasing emphasis on functional textile coupled with the clarion call for eco-friendly textile materials and production processes (Dangelico, Pontrandolfo, & Pujari, 2013; Faqeer, 2014; Kate, 2014), alginate fibers applicable to the manufacture of textile products such as underwear, sportswear and bed clothes (Bogu, Mikolajczyk, Szparaga, & Kurzak, 2010; Senthil Kumar, 2013) have attracted more research attention. As a result, suitable, sustainable and eco-friendly coloration of these products is very crucial

for their sustenance in the ever-changing world of fashion. However, coloration of alginate fibers presents huge challenge because these fibers easily get damaged in salt solution due to ion exchange. Using the traditional method of dyeing (piece/bath dyeing) makes it impossible because large amount of salts are required to ensure complete dye exhaustion, fixation and levelness during the dyeing process (Lv, Zhu, & Wang, 2002). Therefore, it is necessary to find a method for coloring these novel fibers.

Spun-dyeing presents a very unique opportunity in this regard because it yields excellent color fastness, low coloration costs and less environment impact compared to the tradition bath dyeing (Fujikawa, Kushino, & Yamamoto, 1988; Maniana, Ruef, & Bechtold, 2007; Munukutla & Bajaj, 1990; Nava, Bianchi, & Pitea, 1983; Wang, Du, Tian, Fu, & Xu, 2014), yet there are still few publications for successful dyeing of alginate fibers (Lv et al., 2002). Pigment as one of the most commonly used colorant in spun-dyeing also has its downsides because improperly dispersed pigment could lead to agglomeration, resulting in large particle sizes and unequal distribution in the spinning solution which can significantly affect the spinning process, microstructure, surface morphology, color performance and the mechanical properties of the spun-dyed fibers (Munukutla & Bajaj, 1990; Wang et al., 2014). Even though a lot of success has been achieved in the use of pigments for spun-dyeing over the years in terms of good dispersion and dope compatibility, this has been limited to fibers, such as viscose rayon, polyester fibers, acrylic fibers, lyocell fibers (Chen, Lai, & Sun, 2006; Hirota, Kado, & Shosuke, 1989).

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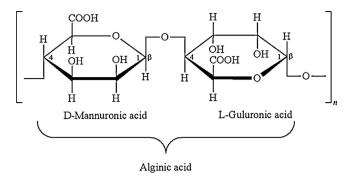


Chart 1. Chemical structure of alginate fiber derived from brown algae.

The compatibility of pigment dispersions and alginate fiber spinning solution has not been reported yet hence the need for this research. In this article, we first prepared fluorescent pigment dispersion and then investigated the compatibility of fluorescent pigment and alginate fiber spinning solution. Properties of spundyed alginate fibers were also studied by various techniques. This research provides further insight into spun-dyeing of alginate fibers with pigment dispersions.

#### 2. Experiments

#### 2.1. Materials

Fluorescent pigment (WLX-15) was purchased from fine chemical North American industry Co. Ltd., China. Alginate sodium was supplied by Qingdao chemical industry Co. Ltd., China. The esterified poly (styrene-alt maleic acid) (SMAE, Mn = 5363, acid value = 163) was prepared in our lab. Sodium hydroxides (NaOH, AR) and calcium chloride (CaCl<sub>2</sub>, AR) were purchased from Guoyao chemical reagent Co. Ltd., China. Distilled water was used in all the experiments.

#### 2.2. Preparation of fluorescent pigment dispersion

Six grams SMAE was dissolved in 264 g distilled water containing with 0.7 g NaOH, followed by the addition of 30 g fluorescent pigment. The mixture was stirred at 600 r/min for 30 min, and then homogenized with bead mill (Minizeta 03E, Netzsu, Germany) at 2000 r/min for 3 h. Finally, the prepared dispersion was filtered (pore size 1000 nm) to obtain the fluorescent pigment dispersion.

#### 2.3. Preparation of spun-dyed alginate fiber spinning solutions

A 10% mass concentration of sodium alginate acid solution was prepared. Different amounts of fluorescent pigment (0–4%, on the weight of alginate fiber) and corresponding amount of water were added to sodium alginate acid solution to prepare the spun-dyed alginate fiber spinning solutions, and kept the mass concentration of sodium alginate acid at 5%. The spun-dyed alginate fiber spinning solution was stirred with strong shear force for 30 min and then filtered using 155 mesh filter cloth under pressure of 0.3–0.5 mPa. The filtered spun-dyed alginate fiber spinning solution was degassed in spinning tank for 24 h.

#### 2.4. Spinning

The prepared spun-dyed alginate fiber spinning solution was heated to  $55\,^{\circ}\text{C}$  and then extruded through 20 hole (90  $\mu$ m diameter) spinneret plate into coagulating bath (CaCl<sub>2</sub> concentration 5 wt%) to produce alginate fiber. The spun-dyed alginate fibers were

washed and drafted in distilled water (draft ratio 1:1.2), and then dried at room temperature.

#### 2.5. Characterization

#### 2.5.1. Properties of fluorescent pigment dispersion

The sample was diluted 2000 times with distilled water. Particle size and its distribution were measured by dynamic light scattering method (DLS) using Malvern Zetasizer Nano ZS90 instrument at 25  $^{\circ}\text{C}.$ 

The sample was centrifuged at 3000 r/min for 30 min, and the particle size of the dispersion in the upper and bottom of the centrifugal tube was tested, respectively. The centrifugal stability was evaluated by the changing rate of particle size ( $S_c$ ) as given by Eq. (1).

$$S_{\rm c} = \frac{\left| d_{\rm u} - d_{\rm b} \right|}{d_{\rm b}} \times 100\% \tag{1}$$

where  $d_{\rm u}$  is the particle size of the dispersion in the upper centrifugal tube,  $d_{\rm b}$  is particle size of the dispersion in the bottom of the centrifugal tube.

The sample was sealed and stored at  $-10\,^{\circ}$ C for 24 h and then put in an oven at  $60\,^{\circ}$ C for another 24 h. The above method was repeated for different cycles. The freeze-thaw stability was evaluated by the change rate of particle size ( $S_{F-T}$ ) as given by Eq. (2).

$$S_{F-T} = \frac{\left| d_0 - d_T \right|}{d_0} \times 100\% \tag{2}$$

where  $d_0$  is the particle size before treatment,  $d_T$  is particle size after freeze-thaw treatment.

### 2.5.2. Compatibility of fluorescence pigment and alginate fiber spinning solutions

The distribution of fluorescent pigment particles in alginate fiber spinning solutions was observed under Digital Scanning Microscope (Zeiss DSM 955). DSM photos were taken at magnification 2000 times.

The apparent viscosity against time of spun-dyed alginate fiber spinning solutions was measured using DV-III Rheometer.

The spun-dyed alginate fiber spinning solutions was placed in an oven at  $60\,^{\circ}$ C, and its apparent viscosity was measured by DV-III rheometer for every 2 h. The stability of the spinning solution was evaluated by the changing rate of apparent viscosity according to Eq. (3)

$$S_{\eta} = \frac{\left| \eta - \eta_0 \right|}{\eta_0} \times 100\% \tag{3}$$

where  $\eta$  is the viscosity before treatment,  $\eta_0$  is the viscosity after storage at 60 °C for different times.

#### 2.5.3. Color measurement

The color strength (K/S) of fabric prepared from spun-dyed alginate fiber was measured using X-rite 8400 spectrophotometer under illuminant D65 and  $10^{\circ}$  standard observers. The color strength was calculated from the reflectance at 540 nm using the Kubelka–Munk equation as given in Eq. (4).

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-R_0)^2}{2R_0} \tag{4}$$

Where R and  $R_0$  are the reflectance of the colored and uncolored fabrics made from the spun-dyed alginate fiber.

The fluorescence intensity was measured on Shimadzu RF-5301PC luminescence spectrometer at excitation-emission wavelength 272 nm.

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