Color Analysis on Natural Polyphenolic Dyed Cotton Cellulose Fibers

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Abstract: Natural polyphenolic dye extracted from green tea is used to dye cotton cellulose fibers. FTIR for the microstructure analysis of fibers, and SEM for the surface morphology observation have been used to characterize the changes induced by tea dyeing. Color shade, thermal stability and mechanical property of dyed fibers are tested. The results show that hydroxyl group of tea polyphenolic and cotton cellulose forms hydrogen bond in dyed fibers without mordant agent, while both coordinate bond and hydrogen bond are coexisted in meta-mordant dyed fibers. The deep shade is obtained in acid media of pH 4 for meta-mordant dyed fibers due to the mordant effect. The coordinate bond contributes to the thermal stability of meta-mordant dyed fibers. The formation of fiber-Fe complexation decreases tensile strength and increases elongation at break for fibers.

Keywords: Cotton cellulose fiber, natural polyphenolic dyestuff, green tea, iron ion, thermal stability, tensile property.

1. Introduction

Cotton cellulose fibers are popular materials used for apparel and home furnishing due to their natural and comfortable properties. There are increasingly used in dyeing of cotton fabrics with natural plant dyes on account of environmental compatibility, low toxicity and allergic reaction [1-4]. Green tea extraction is a natural dyestuff containing several polyphenolic catechins [5], and has been extensively used in medical fields [6] and beverages [7]. The green tea extraction is also reported to be a colorant for cellulose dyeing [8,9].

The above-mentioned researches on dyeing with green tea extraction mainly focus on quantitative aspects of dyed samples. Furthermore, most textile products are dyed after the fabrics have been constructed. The piece dyeing is always quite satisfactory for many users. There are, however, some disadvantages associated with piece-dyed fabrics such as incomplete penetration of dyestuff into fabric, single in fabric color and complicated dyeing control on fabrics made of different fiber types. One of the solutions is to dye fibers and then spin fibers with different colors into yarns.

One of the primary considerations in dyeing fibers with natural dyestuff before spinning is to ensure thermal stability of color and mechanical stability of dyed fibers. This study is to explore the dyeing of cotton cellulose fibers with green tea extraction as the natural dyestuff. The iron ion is selected as a mordant agent due to the least toxicity among the other commonly used mordant agents [10]. The changes in chemical structure and surface morphology induced by tea dyeing are investigated with FTIR and SEM. The thermal durability and tensile properties of dyed fibers are tested to study the color stability and fiber mechanical strength.

2. Experimental

2.1. Materials

Commercial green tea powder was from Hangzhou, China. The combed 100% cotton fibers were provided by Huafu Holding Pte. Ltd., China. $FeSO_4 \cdot 7H_2O$ was used as a mordant agent in this study. Acetate acid and sodium hydroxide were of the analytical reagents.

2.2. Dyeing cotton fibers with green tea

2.2.1 Extraction of green tea

Green tea powder of 2 g was added to distilled water of 100 mL. The mixture was boiled at 100 ºC for 60 min, allowed to be statically cooled at room temperature and then filtered. The filtrate was used as green tea extraction for dyeing.

2.2.2 Dyeing without a mordant agent

Cotton fibers were immersed into green tea extraction at a liquor ratio of 1:50, and dyed with a shaking dyeing machine (L24B-1, Taiwan) at 100 ºC for 60 min. The dyeing bath was cooled down to room temperature.

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After which, the dyed cotton fibers were removed from the dyeing bath and rinsed thoroughly with tap water, followed by squeezing and air drying under shade.

2.2.3 Meta-mordant dyeing of cotton fibers

In the case of meta-mordant dyeing, cotton fibers were immersed into a dyeing bath containing mordant agent and green tea extraction. The liquor ratio was set as 1:50. The other procedure was the same as described in Section 2.2.2.

2.3 Color measurement

Color values were evaluated by *K/S*, ΔE, ΔL, Δa and Δb tested with Spectraflash SF600 spectrophotometer (illuminated D65/10º observer). Each value was averaged from three samples.

2.4 Thermal stability

The meta-mordant dyed cotton fibers were dipped into boiled water for a certain time and rinsed thoroughly with tap water. After squeezing and air drying, the color values of fibers were tested to evaluate color thermal stability.

2.5 Characterization

Scanning electron microscope (SEM, JSM-5600LV) was used to study morphology of the cotton fiber samples before and after dyeing. The samples were sputter-coated with gold prior to examination. Secondary electron images were acquired using an accelerating voltage of 10 kV. Fourier transform infrared spectra were collected using a TENSOR37 FTIR Spectrometer (Bruker Company). The samples were analyzed in 800 ~ 4000 cm^{-1} range with a 4 cm^{-1} resolution.

2.6 Tensile property of cotton fibers

A model YG001A fiber tensile tester (Taicang, Jiangsu, China) was used to test tensile property of cotton fibers. The pretension was 10 cN. The gauge length was 1 cm and the loading speed was 10 mm/min. The test was carried out at room temperature with relative humidity of (65±5) %. Each value was averaged from 50 tests.

3. Results and discussion

3.1 FTIR analysis

The interaction between cotton cellulose fibers and dyestuff plays important role on stability of dyed fibers. The molecular structure change induced by tea dyeing could be identified by FTIR due to the vibration mode modification of the molecules involved [11].

Figure 1 shows the FTIR spectra of cotton fibers before and after dyeing. The band near 3368 cm^{-1} in the raw cotton fibers is the characteristic hydroxyl group peaks of cellulose. After dyeing, the peak intensity increases and shifts towards high wavenumber, indicating the change of hydrogen bond. This could be attributed to the damage of intramolecular hydrogen bond and formation of intermolecular hydrogen bond between oxygen of tea polyphenolic compounds and hydroxyl group of cellulose fiber [12]. New peaks representing the aromatic ring vibrations of tea polyphenolic are found around 1600~1450 cm^{-1}, especially in the spectrum of dyed fibers without mordant agent. For the meta-mordant dyed fibers, there appeared some peaks around 2400~2000 cm^{-1} which are attributed to the transition-metal carbonyls [13]. This indicates that both coordinate bond and hydrogen bond exists in the meta-mordant dyed fibers. The formation of coordinate bond between cellulose and iron ion is expected to improve color fastness.

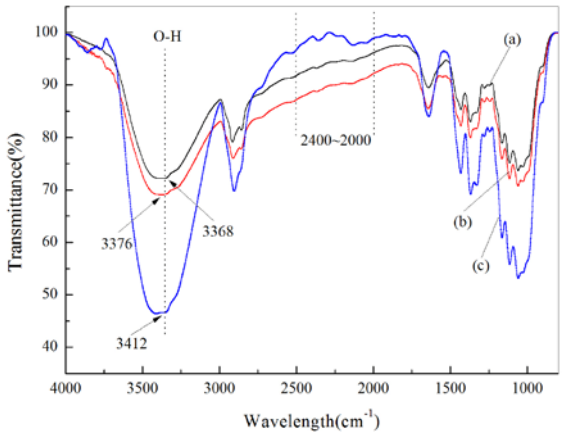

Figure 1 FTIR of cotton fibers. (a) Before dyeing. (b) Dyeing without mordant agent. (c) Meta-mordant dyeing.

3.2 Morphology analysis by SEM

The surface morphology of raw cotton fibers and dyed fibers is observed by SEM as shown in Figure 2. It is concluded from Figure 2 that the SEM micrograph of