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Modification of wool fiber using steam explosion

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Abstract

Wool fiber was modified by steam explosion in this study. SEM results show that some scales on the fiber surface were cleaved and tiny grooves generated during the explosion. FTIR results suggest no evident changes in the chemical composition of the fiber after the explosion treatment. However, the crystallinity of the fiber decreased slightly as the steam pressure increased based on the X-ray results. In the thermal analysis, DSC results show that the temperature corresponding to vaporization of absorbed water and cleavage of disulfide bonds respectively decreased as the steam pressure increased. The reduction in thermal decomposition energy of the treated fiber indicates that steam explosion might have destroyed some crystals and crosslinks of macromolecular chains in the fiber. The treatment also led to some alterations of the fiber properties, including reduction in strength, moisture regain and solubility in caustic solution.

Keywords: Wool; Steam explosion; Modification; X-ray; SEM; TG; DSC

1. Introduction

Steam explosion is a physical treatment that involves an instant discharge of high-pressure steam in a sealed container. It was originally applied in the separation of plant fibers and starch. Recently this method was used in the modification of celluloses fibers such as cotton and flax [1]. Three types of cellulose (softwood pulp, hardwood pulp, cotton linters) were investigated by scanning electron microscopy, nuclear magnetic resonance and mass spectrometry after the explosion [2]. Steam explosion was also found to be effective for delignification of wood and cleaning of recovered cellulosic materials to produce the pulp for dissolving in *N*-methylmorpholine oxide [3]. To date, steam explosion has been used to modify the cellulosic materials mainly. We have recently applied this technique to protein materials such as silk, and found that silk was separated into more than 10 small fibroins filaments when treated with 0.8 MPa steam explosion [4]. Steam explosion may be a very effective method in modifying the properties of other protein materials such as wool.

Wool is a high-quality protein fiber and is widely used as a high-quality textile material. There has been considerable interest also in finding new applications for wool through developments of new wool products, such as wool film [5] and wool powder [6].

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As for wool powder, different methods have been developed for its production, including mechanical method and chemical–mechanical method. To obtain super fine wool powder, special chemical pretreatment is usually employed to destroy the chemical bonds and decrease the crystallinity of the parent wool fiber. This would also improve the powder productivity by fiber grinding. Some researchers have employed steam explosion to produce wool powder directly [6]. This paper presents comprehensive results on the properties of wool after steam explosion treatment.

2. Experimental

2.1. Materials

Clean wool tops (e.g. a thick bundle of wool fibers) were used in the experiments. The average diameter of the wool is about 20 μm. The wool tops were treated by steam explosion under different pressures in a purpose-built machine. Sample 0# was the control sample, samples 1#, 2#, 3#, 4# represented the samples treated under steam pressure of 0.2 MPa, 0.4 MPa, 0.6 MPa, and 0.8 MPa, respectively.

2.2. SEM analysis

Scanning electron microscopy (SEM) analysis was carried out with a X-650 microscope, at 10 kV acceleration voltage, after gold coating.

2.3. FTIR analysis

A FTIR instrument (Nicolet 20 SXB) was used to analyze the spectra of the samples. Resolution for the infrared spectra was 4 cm^{-1} and there were 32 scans for each spectrum. KBr pellet sampling method was used to prepare the thin film for testing. Transmittance of the infrared in the film between 400 cm^{-1} and 4000 cm^{-1} was collected by the instrument.

2.4. X-ray diffraction analysis

To determine the crystallinity of the treated samples under different conditions, the sample (powder) was dispersed onto a stub and placed within the chamber of analytical X-ray powder diffractometer (Japanese Dmax-rA, wavelength = 1.54 Å, CuKα radiation). Generator intensity was 40 kV, generator current was 50 mA. The sample was then scanned from $2\theta = 5–70°$, in step of 0.02°. The resultant graphs were printed out on the Origin graph plotting package.

2.5. Thermal analysis

Thermal gravity (TG) was performed on DSC/TG STA-449C, and the testing was carried out in flowing nitrogen atmosphere (5 mg/min), at a heating rate of 10 °C/min. Differential scanning calorimeter (DSC) was performed on model Perkin–Elmer DSC-2, and the testing was carried out in flowing nitrogen atmosphere (5 mg/min), at a heating rate of 10 °C/min, end temperature of the testing was 300 °C.

2.6. Test of moisture regain and solubility

Moisture regain of the samples were tested under standard conditions (RH 65% and 25 °C). The moisture regain of the samples was determined by

$$\text{Moisture regain } (\%) = (W_2 - W_1)/W_1 \times 100$$

where W_1 and W_2 represented the dry weight and conditioned weight of the samples, respectively.

To measure the solubility of the treated wool fiber, the samples were dissolved in an aqueous solution of 2.5% NaOH at 80 °C, because the disulfide bonds in the wool macromolecular is very sensitive to the caustic solution. Dissolving time of the sample was then determined.

2.7. Tensile properties of the treated wool fiber

Tensile property of single fiber was measured on a single fiber tensile tester (model LLY-06A), under the conditions of 0.3 cN pretension, 10 mm gauge length and 20 mm/min extension. For each sample 60 fibers were tested and the mean value and the deviation were calculated.

3. Results and discussion

3.1. Surface morphology

The surface morphology of wool is characterized by the scales, which play an important role in protecting the wool from damage. In addition, the scales have great influence on other important properties of wool, such as luster and shrinkage. The