



Effect of aqueous glycine treatment on the fine structure and dyeing ability of cotton

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ABSTRACT

Exhaustion dyeing of cotton means a significant proportion of dyestuff is not fixed onto the material and is drained out when the dye bath is emptied. This waste presents issues for dye houses in terms of remediating water quality and the loss of expensive dye chemicals. Work to improve the dyeing ability of cotton has been limited to modifying cotton's cellulose structure by, notionally, environmentally hazardous treatments, e.g., concentrated caustic soda. Here we use a buffered, aqueous glycine treatment to improve the dyeing ability of cotton. The treated cotton showed improved dye absorption compared with control samples. An investigation of the structure of the treated cotton using Fourier Transform Infrared spectroscopy (FTIR) and ^{13}C solid-state nuclear magnetic resonance (NMR) showed there was no appreciable change in crystallinity. However, there were changes in the chemical structure of the glycine-cellulose complex that were related to the pH of the buffered glycine treatment.

1. Introduction

In developing chemical process treatments for cotton textiles, understanding the effects of chemical reactions on the fine structure of cotton cellulose is important. The building block in cotton fibre is β -1,4-D glucopyranose and the blocks are linked by 1,4-glycosidic bonds. There are three hydroxyl groups attached equatorially to each anhydroglucose (at the C_6 , C_2 and C_3 atoms) molecule and these form both inter and intra-molecular hydrogen bonds with surrounding molecules (Anderson & Kerr, 1938; Lewin, 2006; Mitchell, Carr, Parfitt, Vickerman, & Jones, 2005; Tripp & Rollins, 1952). Intramolecular hydrogen bonds (within the same glucopyranose unit) are formed between the C_2 -OH and C_6 -OH groups as well as the C_3 -OH and endocyclic oxygen. Intermolecular hydrogen bonds (with adjacent glucopyranose units) are formed between the C_3 -OH and C_6 -OH groups (Baptista, Ferreira, & Borges, 2013; Lewin & Pearce, 1998).

A variety of chemical methods have been explored for improving the physical and chemical properties, e.g., strength, lustre and dye uptake of cotton. Treatments with strong alkalis such as sodium hydroxide, ammonia and ethylene diamine are well known (Kalyanaraman, 1985; Orr, Burgis, Andrews, & Grant, 1959; Rousselle, Nelson, Hassenboehler, & Legendre, 1976; Sameii, Mortazavi, Rashidi, & Sheikhzadah-Najar, 2008; Zhang, Zhu, & Sun, 2014). In these,

modification of the intra and inter-molecular hydrogen bonding occurs with the degree dependent on the reagents, its concentration and the effect of any pre-treatments, e.g., scouring, which typically involves a light alkaline and soap treatment.

Mercerisation is one widely used treatment of cotton to affect improved dyeing ability and strength (Sameii et al., 2008). In conventional mercerisation, untreated cotton (cellulose I) is treated with a highly concentrated sodium hydroxide solution to form alkali cellulose, also commonly known as soda-cellulose. After removing the alkali residue via a wash and neutralisation, the cellulose structure turns to cellulose II. The reaction with the concentrated alkali starts in the amorphous regions of the cotton cellulose, and if the concentration of sodium hydroxide is high enough, progresses into the crystalline regions to transform the cellulose I structure via soda-cellulose into cellulose II.

Liquid ammonia treatment is another swelling/mercerizing treatment for cotton. It has been used commercially since the mid-1960s. During the ammonia-cellulose interaction, ammonia penetrates the cellulose and reacts with the hydroxyl groups. The reaction first occurs in the amorphous region followed by the crystalline regions (Hu & Hsieh, 1996; Sreenivasan, Iyer, & Patel, 1993). First an intermediate ammonia-cellulose complex is formed, which is then changed into cellulose III (Greenwood, 1987). The unit cell dimensions for the

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cellulose crystal are different again for cellulose III. The intermediate ammonia-cellulose complex breaks down in several ways, depending on the way ammonia is removed, with evaporation giving rise to a cellulose III form, while washing with water forms cellulose I (Bertoniere & King, 1989; Lewin, 2007).

The effects of caustic versus liquid ammonia mercerisation on cotton dyeing ability have been compared. Cotton fibres mercerized with sodium hydroxide exhibited large pores and higher dye absorption compared to ammonia-treated cotton (Rowland, Wade, & Bertoniere, 1984). Other workers observed improved dye absorption, fabric lustre and mechanical properties with similar treatments (Greenwood, 1987; Jones, Turner, & Luparello, 1980; Nelson, Hassenboehler, Andrews, & Markezich, 1976; Saapan, Kandil, & Habib, 1984). However, a key challenge in cotton dyeing and in the ability of cotton to more fully exhaust dyestuff from the dyebath has been the availability, or unavailability, of an effective, non-corrosive mercerizing/swelling agent.

Recently it was shown that cotton fibres could be swollen in aqueous glycine solution to improve the fibre's mechanical properties (Remadevi, Gordon, Wang, & Rajkhowa, 2017). The objective of this current work was to examine the changes in the structure and dyeing ability of cotton fibre and fabric after various glycine treatments. Specifically, crystallinity and structural changes in glycine treated cellulose (cotton fibres) were examined using attenuated total reflection (ATR) FTIR and solid state NMR. ^{13}C NMR cross polarization magic angle spinning (CP-MAS) was used to reveal changes in the crystallinity and structure variation of the cotton cellulose.

2. Experimental section

2.1. Materials

The fibre examined in this study came from Australian cotton variety Sicot 71BRF (*Gossypium hirsutum*), which was grown under full irrigation in season 2012/13 at the Australian Cotton Research Institute (ACRI) in Narrabri (30.3°S 149.8°E). The cotton was machine-harvested and ginned with minimal pre-cleaning using a narrowed (40 saw) Continental Eagle 119 saw gin with one lint cleaning passage.

The cotton fibre used in this study was a Strict Middling grade cotton, i.e., reflectance (Rd) > 83 and yellowness value (+b) < 8.0, with low trash and a staple length (upper-half mean length) of 1.20 in., breaking tenacity of 29.8 g/tex and micronaire value of 4.3; suggesting reasonable maturity and fineness. It is noted the maturity component of micronaire has a strong effect on dye uptake and specular reflectance, and thus on the dyed appearance of the cotton (Pal & Esteve, 1959; Smith, 1991).

The cotton fabric used for the dyeing ability examination was a commercial, un-scoured, plain weave cotton fabric (142 g/m²) woven from 30 tex (Ne 20) yarn with 650 turns per metre.

Sodium hydroxide (NaOH), glycine (Reagent plus 99%, HPLC grade) and hydrochloric acid (32%) were purchased from Sigma Aldrich and used as received. All reagents were analytical grade and all aqueous solutions were prepared with deionised water.

2.2. Fibre and fabric sample preparation

Raw cotton fibre specimens (1 g) were scoured using a conventional scouring method to remove the impurities and wax from the surface of fibres. A Datacolor AHIBA IR Pro laboratory rotary dyeing machine (Datacolor, USA) was used to treat fibre specimens in an aqueous NaOH (4%) bath at 100 °C for 90 min. At the conclusion, specimens were washed with a large volume of distilled water to remove scouring agent and dried overnight in a laboratory convection oven at 50 °C. The fabric used to examine dyeing ability was also pre-treated using the same scouring procedure.

Table 1
Fibre Treatments and Codes.

Number	Sample Treatment	Code
1	Untreated	UTC
2	Scoured	SC
3	Treated at pH 3 (buffer control)	W3
4	Glycine treated at pH 3	CG3
5	Glycine treated at pH 7	CG7
6	Treated at pH 11 (buffer control)	W11
7	Glycine treated at pH 11	CG11

2.3. Treatment with glycine

Glycine treatments were performed on scoured cotton samples. Specimens of scoured cotton fiber and fabric samples were treated in aqueous solutions of glycine (20% w/v) adjusted for pH using either sodium hydroxide (10% w/v) or hydrochloric acid (32% w/v) in a 1:40 material-to-liquor ratio at 100 °C for 24 h under reflux with constant stirring. Three pH treatments were examined: pH 3, 7 and 11. Notably, the alkali and acid concentrations in these solutions were low (0.2–0.4 M) compared with the molarity of a typical mercerizing solution (at 3–5 M). Control aqueous treatments at different pH without glycine were also applied. At the end of each treatment, fibers were thoroughly washed with distilled water and dried overnight in a convection oven at 50 °C.

A long 24 h reflux period was used to allow the reaction between the glycine and cellulose to reach full equilibrium without the introduction of additional reagents such as surfactants, which could have reduced the equilibration time to as low as 2 h. By excluding other reagents the effects observed were more clearly attributable to glycine. Table 1 lists the treatments applied to the cotton fibre and fabric samples in this examination.

2.4. Dyeing of fabric samples

Dyeing of the samples was performed using a Datacolor AHIBA IR Pro laboratory rotary dyeing machine (Datacolor, USA). Dyeing was carried out using 0.1% w/w and 1% w/w Procion Blue HERD. Dyeing was performed using 0.1% and 1% dye concentrations to verify shade variation between samples noting that sometimes higher dye concentrations create difficulties in identifying differences in colour variation. Fabric samples were added to a solution of NaCl (70 g/L), Albalflow FFW (0.5 g/L) and Verolan NBO (1 g/L), and held at 30 °C for 10 min before dye was added. The temperature was then raised to 90 °C (at 2 °C/min) and held for 30 min. The bath was then cooled to 70 °C (at 2 °C/min) before soda ash (10 g/L) was added. Samples were held for a further 35 min in this state. (The temperature profile used for dyeing is illustrated in Fig. S1 of the ESI). Dyed samples were rinsed in cold water, spun dry and then further dried in a 105 °C convection oven for 60 min.

2.5. Examination of dye uptake

The colour of the dyed samples was measured with a Gretag Macbeth Color-Eye 7000A UV/VIS reflectance spectrophotometer (X-rite, USA) using a D65 light source and a 10° collection angle with the spectral component included. Samples were conditioned for 24 hs under standard conditions (20 ± 2 °C and 65 ± 2% relative humidity) prior to colour measurement. Sample fabrics were measured using the instrument's large (25.4 mm) aperture, whilst folded twice in half to eliminate interference from background light. Colour was measured in the CIELab colour space using an average of three readings. ΔE values, which measure the extent of the colour difference between a sample and a nominated control; in this case a scoured, untreated sample, were calculated using Eq. (1) below. (ΔE values are tabulated in Table S1 of

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