



Effects of acid diffusibility and affinity to cellulose on strength loss of polycarboxylic acid crosslinked fabrics



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Benzophenone-3,3',4,4'-tetracarboxylic dianhydride (PubChem CID: 75498)

1,2,3,4-Butanetetracarboxylic acid (PubChem CID: 15560)

Sodium hypophosphite monohydrate (PubChem CID: 23708894)

2,4,6-Trihydroxybenzoic acid monohydrate (PubChem CID: 2723793)

3,4-Dihydroxybenzoic acid (PubChem CID: 72)

Bromophenol blue (PubChem CID: 8272)

Collodion (PubChem CID: 44135439)

ABSTRACT

1,2,3,4-Butanetetracarboxylic acid (BTCA) imparts good anti-wrinkle property to cotton fabrics and results in significant strength loss due to cross-linking and acid degradation of cellulose simultaneously. However, benzophenone-3,3',4,4'-tetracarboxylic acid (BPTCA), an aromatic acid, crosslinks cellulose effectively but causes less strength loss to the products under similar conditions. The difference in damages to cellulose fibers was analyzed by using diffusibility and corresponding affinity of the acids to cellulose fibers, which were estimated by their molecular sizes and Hansen solubility parameters (HSP). Both experimental results and theoretical speculations revealed consistent agreement, indicating that smaller acid molecules could diffuse into cellulose fiber more rapidly and deeply, resulting in more acid degradation. Besides, the aliphatic acid such as BTCA has higher molecular affinity than BPTCA to cellulose, causing additional more degradation of cellulose. Both factors are potential reasons of the observed more severe tensile strength loss of the BTCA treated cotton fabrics.

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1. Introduction

Esterification of polycarboxylic acids with cellulose can form cross-linking bridges between celluloses and can be applied in anti-wrinkle finishing of cotton fabrics (Welch, 1988; Yang, 2001). It has been proved that 1,2,3,4-butanetetracarboxylic acid (BTCA), an model aliphatic polycarboxylic acid, reacts with cellulose by first formation of its intermediate anhydrides (Yang and Wang, 1996) and then esterification of the anhydrides with cellulose. The roles

of catalysts in the reactions were also clarified in the previous studies (Ji, Qi, Yan, & Sun, 2015; Ji, Tang, Yan, & Sun, 2015; Peng, Yang, & Wang, 2012; Yang, 2001). However, the severe strength loss of the treated fabrics is a main hurdle to its practical application. There are two major reasons accounting for the strength loss in the crosslinked cellulose by polycarboxylic acids: ester cross-linking between acid and cellulose and acidic degradation of cellulose macromolecules (Kang, Yang, Wei, & Lickfield, 1998).

In our previous studies, the catalytic actions of alkaline salt catalysts were investigated in details (Ji, Qi et al., 2015; Ji, Tang et al., 2015). Results indicated that large alkaline metal ion accelerates the formation of anhydrides by BTCA, and catalyst anion assists the esterification between anhydrides and cellulose. On the other side,

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pH value of finishing baths, which affects concentrations of corresponding acid anion and metal cation of the catalyst salt, shows important impact on both reactions of anhydride formation and esterification. Obviously, pH of the finishing bath will affect the strength loss of the treated fabrics by damaging cellulose. Yang et al. have investigated and identified the effects of pH and molecular flexibility of finishing reagents on the strength loss (Chen, Lickfield, & Yang, 2004; Kang et al., 1998; Yang, Wei, & Lickfield, 2000). Higher pH and larger molecular flexibility could be beneficial to strength retention of the treated fabrics.

However, recent studies revealed that aromatic acids, such as benzophenone-3,3',4,4'-tetracarboxylic acid (BPTCA), could achieve the similar anti-wrinkle property on the fabrics but always cause lower tensile strength losses than aliphatic polycarboxylic acid does under similar conditions. Furthermore, aromatic acids are more acidic than aliphatic ones due to the chemical structural features under the same concentration in solutions. This disparity in damaging cellulose by the two types of acids is difficult to understand if only the impact of pH value is considered, but is worth of further investigation. One difference of the polycarboxylic acids lies at their molecular volumes or sizes, which could affect the diffusion of the chemicals into the cellulose fiber, one kind of polymers (Hansen and Hansen, 1988). Besides, both acids are dissolved in water, and are absorbed onto cotton fibers and finally diffuse into the fiber (Hou and Sun, 2013; Ji, Tang et al., 2015). Here, acids with great affinity to cellulose may diffuse rapidly into cellulose and also closely interfere with cellulose. Therefore, the interactions between the acids and cellulose should also have an impact on the strength loss of the treated fabrics, while the affinity of the acids with cellulose could be evaluated by analyzing their structural similarity.

With the purpose of further understanding the reasons of mechanical strength loss of the anti-wrinkle fabrics treated by polycarboxylic acids, the effects of molecular volumes (MV) and Hansen solubility parameters (HSP) of the acids were investigated in this research. MV and HSP of acids were calculated by ChemDraw14.0 software to compare their diffusibility in the cellulose fibers and by HSPiP 4.1.07 software to compare their affinity to the cellulose, respectively. Besides, Fourier transform infrared (FTIR) spectroscopy was also used in the analysis of the relationship between wrinkle recovery angle and ester cross-linking.

2. Experiment

2.1. Materials

Desized, scoured, and bleached pure cotton fabrics (#400) were obtained from Test Fabrics, Inc. (West Pittston, PA). Succinic acid (SUA), dichloroacetic acid (DCAA), benzophenone-3,3',4,4'-tetracarboxylic dianhydride (BPTCD), sodium hypophosphite monohydrate (SHP) and glycerol were purchased from Sigma-Aldrich Co. (St Louis, USA). 1,2,3,4-Butanetetracarboxylic acid (BTCA), 2,4,6-trihydroxybenzoic acid monohydrate (2,4,6-THBA, 95%), 3,4-dihydroxybenzoic acid (3,4-DHBA) and bromophenol blue were all purchased from Acros (New Jersey, USA). Sodium hydroxide (NaOH) was from EMD chemical Inc. (New Jersey, USA). Collodion (5%) was purchased from Electron Microscopy Sciences (Hatfield, USA). All chemicals were directly used as received.

2.2. Fabric treatment

Fabrics were soaked in a solution of acid with or without catalyst and were padded twice with a wet pickup of ~120%. The carboxyl concentrations were kept same for comparison, and catalyst was used at a 0.5 mole ratio to the amount of the acid. After being dried at 80 °C for 5 min, they were cured at 160 °C for 3 min. And then

they were washed in tap water at the room temperature for 10 min to eliminate the residual finishing agents and catalysts.

2.3. Wrinkle recovery angle (WRA) and tensile strength retention (TSR)

WRA and TSR of fabrics were measured according to the American Association of Textile Chemists and Colorists (AATCC) 66-1990 method and the American Society for Testing and Materials (ASTM) D 5035-06 method, respectively. WRA was measured by a wrinkle recovery tester (T.J. Edwards Inc., Boston, USA), and TSR was measured by an Instron 5566 instrument (Instron Corporation, MA, USA). For samples to be alkaline washed, treated fabrics were soaked in a 0.1 mol/L NaOH solution for 24 h at 50 °C to completely destroy the cross-linking between acid and cellulose.

2.4. FTIR

Before FTIR evaluation, treated fabrics were washed in a 0.1 mol/L NaOH solution for 4 min at room temperature, and dried at 80 °C for 5 min. They were cut into powders, weighed accurately for 2.0 mg, and then mixed with 200.0 mg potassium bromide (KBr) to be pressed into a pellet. FTIR spectroscopy was measured by a Nicolet 6700 FTIR spectrometer (Thermo Electron Co., USA) in absorbance mode for all the samples in the range of 4000–400 cm⁻¹, and scan times and resolution were 64 and 4 cm⁻¹, respectively. Ester bond absorbance intensity at 1724 cm⁻¹ (Yang, 1991; Yang, 1993a) was normalized against the absorbance intensity of 2900 cm⁻¹, due to the C–H stretching vibration (Hou and Sun, 2013; Lam, Kan, & Yuen, 2011).

2.5. Optical microscopy

Treated fabrics were cut by fiber slicing machine (Delarue Hyattsville, USA), and cross-section samples were put on the microscope slide with a drop of glycerol. And then a drop of bromophenol blue solution was dropped onto the sample. Samples were measured by a Motic microscope and the pictures were captured by a Dinoeye eyepiece camera AM-4023 (The Microscope Store, LLC, VA, USA) with a Dinocapture 2.0 software.

2.6. Molecular size by ChemDraw14.0

ChemDraw14.0 software was used to calculate the molecular volume (Álvarez and Aznar, 2008). Firstly, the molecules were processed by MM2 Minimize Energy program, and then by the Connolly solvent excluded volume program. The radius (r) of the acid was calculated based on

$$V = \left(\frac{4}{3}\right) \pi r^3 \quad (1)$$

where V is the Connolly solvent excluded volume of an acid.

2.7. Hansen solubility parameters (HSP) (Hansen, 2007)

HSP of various acids were calculated by HSPiP 4.1.07 software according to Y-MB method, and HSP of water or cellulose were obtained from the database in the software. The distance between acid and water or cellulose was calculated based on

$$Ra = [4(\Delta\delta_D)^2 + (\Delta\delta_P)^2 + (\Delta\delta_H)^2]^{1/2} \quad (2)$$

where δ_D is the energy from dispersion forces between molecules, δ_P is the energy from dipolar forces between molecules, and δ_H is the energy from hydrogen bonds between molecules.

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