



# Multifunctional finishing of cotton with 3,3',4,4'-benzophenone tetracarboxylic acid: Functional performance



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## ABSTRACT

3,3',4,4'-Benzophenone tetracarboxylic acid (BPTCA) can directly react with hydroxyl groups on cotton cellulose to form ester bonds, which could crosslink cellulose and provide wrinkle-free functions to the cotton fabrics. BPTCA, as a derivative of photo-active benzophenone, can absorb ultraviolet lights and offer ultraviolet (UV) protective and photo-sensitive functions on the treated materials. Finishing conditions such as agent concentration, curing temperature and time, could affect ester bond formation and crosslinking of cellulose. Wrinkle-free and UV protective functions in relationship to the functional agent, as well as surface morphology of BPTCA treated cotton were investigated as well.

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## 1. Introduction

Clothing has been considered and actually has acted in many aspects as a "second skin", a protective outer layer, to human. Real human skin is the largest organ that has various protective functions against almost all natural hazards. When novel protective functions are added onto clothing materials, the new products are imaged as a second skin to wear. Based on general expectations, such clothing materials should at least possess several functional properties including comfort, easy-care, moisture and heat regulating, self-cleaning against biological and chemical toxins, sun or UV-protective functions. For example, cotton fabric is the most popular material used in various consumer and professional apparels due to its comfort, moisture absorbing and excellent wearing properties. Additional wrinkle-free, UV blocking, and antimicrobial properties are considered necessary to be added for human convenience and health demands, which could make the clothing more resemble a second skin. Wrinkle-free treatment of cotton fabrics is generally achieved by chemically crosslinking cotton cellulose by using traditional formaldehyde containing N-methylol compounds or non-formaldehyde 1,2,3,4-butanetetracarboxylic acid (BTCA) (Lam, Kan, & Yuen, 2011; Montazer & Afjeh, 2007; Yang & Wei, 2000a, 2000b). The UV protective function on the fabrics, which is important for protecting both materials and wearers

(Gouda & Keshk, 2010; Lu, Fei, Xin, Wang, & Li, 2006), can be obtained by incorporating UV absorbents or UV blockers onto the fabric (Czajkowski, Paluszkiwicz, Stolarski, Kaz'mierska, & Grzesiak, 2006; Hatch & Osterwalder, 2006; Hou, Zhang, & Wang, 2012; Ibrahim, E-Zairy, & Eid, 2010a; Wang & Hauser, 2010). To obtain multiple functions, multi-step chemical treatments of cotton fabrics are normally required, which could consume large quantity of water and energy and consequently increase costs and environmental impacts. Thus, development of energy efficient and multi-functional finishing processes of textiles is extremely important (Ibrahim, Eid, Hashem, Refai, & EL-Hossamy, 2010).

In a previous study, 3,3',4,4'-benzophenone tetracarboxylic dianhydride (BPTCD) was employed in a direct reaction with cotton cellulose by using N,N-dimethylformamide (DMF) as a solvent (Hong & Sun, 2011). The reactive anhydride groups can crosslink cellulose and bring durable press and photo-active functions to the fabrics, though the treatment process was not practical due to the use of DMF. In a more recent study, 3,3',4,4'-benzophenone tetracarboxylic acid (BPTCA), which is hot water soluble, was directly used to react with cellulose without formation of anhydride structures during the treatment process (Hou & Sun, 2013). Such a process is commercially practical, which leads to further evaluation of functional properties, including UV-protection, wrinkle resistance, and more importantly mechanical properties, of the treated fabrics. Surface morphological changes of the treated fabrics were also examined by a scanning electron microscope (SEM). The results indicate that BPTCA could bring multiple functions onto cotton fabrics in a simple one step wet finishing process.

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## 2. Experimental

### 2.1. Materials

Desized, scoured, and bleached cotton plain weave fabrics (#400) were purchased from Test Fabrics Inc. (West Pittston, PA, USA). 3,3',4,4'-Benzophenone tetracarboxylic dianhydride (BPTCD) and sodium hypophosphite monohydrate were purchased from Sigma Chemical Co. (Louis, MO, USA). All other chemicals were purchased from Fisher Scientific (Pittsburgh, PA, USA). All reagents were used as received without any further purification.

### 2.2. Treatment of cotton fabrics

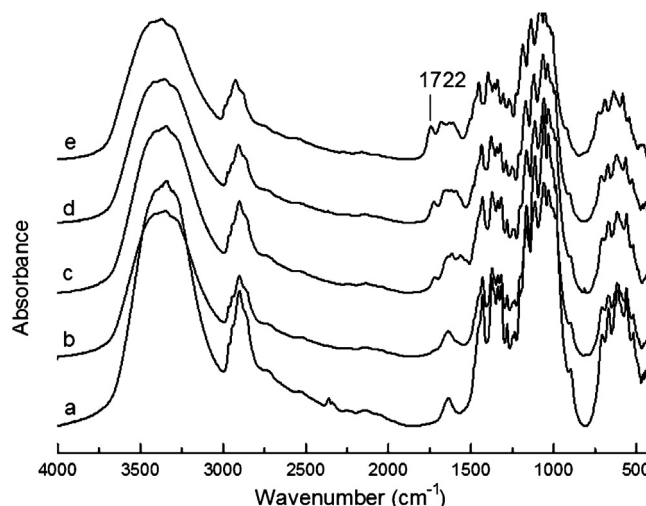
3,3',4,4'-Benzophenone tetracarboxylic dianhydride (BPTCD) was dissolved in hot water to completely hydrolyze to 3,3',4,4'-benzophenone tetracarboxylic acids (BPTCA), and the solution was directly applied in a wet finishing process on cotton fabrics. Sodium hypophosphite monohydrate was added as a catalyst to the BPTCA solution in a mole ratio of catalyst:BPTCD = 1:2. The cotton fabric was first impregnated in the solution and then padded through two dips and two nips to reach an average wet pickup of 120%, dried at 90 °C for 3 min, and cured in a curing oven (Roaches International Ltd., England) at a specified temperature for 3 min. And finally the treated fabrics were washed with water and air-dried in a conditioning room (25 °C, 65% R.H.) for 24 h.

### 2.3. Characterization of treated fabrics

Add-ons of BPTCA on the treated cotton fabrics were obtained by measuring weight changes of the fabrics before and after the treatments. Ultraviolet protection factors (UPF) of the fabrics were measured by using an Ultraviolet Transmittance Fabric Analyzer, UV-1000F (Labsphere Co., USA). Fourier transform infrared (FTIR) spectroscopy was performed with a Nicolet 6700 FTIR spectrometer (Thermo Electron Co., USA) with a resolution of 4 cm<sup>-1</sup>, and the measurements were carried out by using KBr pellets.

Wrinkle recovery angle (WRA) and tensile strength of the cotton fabrics were measured according to AATCC Standard Test Method 66-2008 and ASTM Standard Test Method 5035-2006, respectively. Twelve specimens (six for warp and six for filling) were tested for WRA. Ten specimens (five for warp and five for filling) were employed in tensile strength tests on an Instron 5566 (Instron, Norwood, USA).

Washing durability of the fabric functions was evaluated following AATCC Standard Test Method 61-2009 (2A). The fabric used for the washing durability tests was treated with 50 g/L BPTCD, and cured at 160 °C for 3 min. The samples were placed in a Launder-O-meter using an accelerated laundering cycle at 49 ± 2 °C in a detergent solution. Then the fabric samples were washed one, two and three times, respectively. Each accelerated washing is equivalent to five home launderings. The wrinkle properties (WRA) were then evaluated again. The surface morphologies of cotton fabrics were examined using a scanning electron microscope (Philips XL30, USA).



**Fig. 1.** FTIR spectra of untreated and treated cotton fabrics with different amounts of BPTCD curing at 160 °C for 3 min: (a) untreated (control) cotton fabric; (b) fabric treated with 10 g/L BPTCD; (c) fabric treated with 30 g/L BPTCD; (d) fabric treated with 50 g/L BPTCD; (e) fabric treated with 70 g/L BPTCD.

## 3. Results and discussion

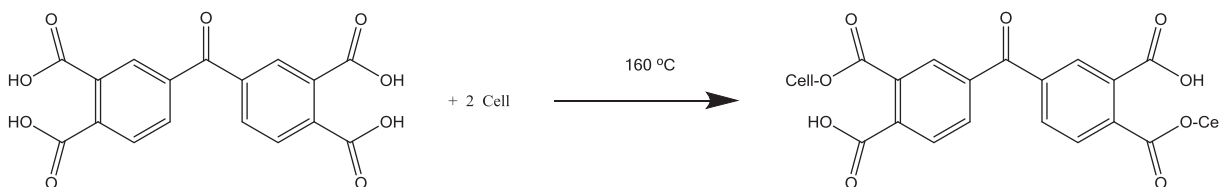
### 3.1. BPTCA treatment of cotton fabrics

BPTCA was applied onto cotton fabrics by using a two-dip-two-nip wet treatment process. The BPTCA treated fabrics were dried at 90 °C for 3 min to remove water, and cured at an elevated temperature for certain time to accelerate the esterification between the carboxylic acids and hydroxyl groups (Hou & Sun, 2013) shown in Scheme 1.

The structural changes of BPTCA treated cotton fabrics were examined by using FTIR. Fig. 1 shows FTIR spectra of the untreated (control) cotton fabric and the fabrics treated with different concentrations of BPTCA solutions. The peak at 1722 cm<sup>-1</sup> is a corresponding band of ester bond formed after curing, confirming the direct esterification reaction between BPTCA and cotton cellulose. The intensities of 1722 cm<sup>-1</sup> band on the cotton fabrics increased with the increase of BPTCA concentration, clearly noticeable in the FTIR spectra and consistent with the results of weight increases of the treated fabrics (Fig. 2).

### 3.2. Conditions on esterification reaction

The primary goal of this work was to achieve proper crosslinking effects and consequently desired wrinkle-free functions on the BPTCA treated cotton. According to the reaction mechanism discussed in the previous paper (Hou & Sun, 2013), amount of BPTCA in finishing baths and reaction temperature could significantly affect the formation and amount of ester bonds. Thus, different concentrations of BPTCA (10, 30, 50, 70 g/L measured in amount of solid BPTCD) were used to treat the fabrics at selected curing temperatures (140, 160, 170, 180 °C). Increasing BPTCA concentration in the



**Scheme 1.** Chemical reaction of cellulose with BPTCA.

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