



Non-formaldehyde, crease resistant agent for cotton fabrics based on an organic–inorganic hybrid material



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ABSTRACT

1,2,3,4-Butanetetracarboxylic acid (BTCA) was reacted with (3-aminopropyl)triethoxysilane (APTES) to a poly(amic)acid (PAA). The molar ratios of BTCA and APTES were 1/1 (B/A-1/1), 1/2 (B/A-1/2), 1/3 (B/A-1/3), and 1/4 (B/A-1/4). The as-prepared precursor solution was applied to cotton substrates. After thermal treatment (180 °C) the physical–mechanical properties of the cotton samples were tested by means of dry crease recovery angle and tensile strength. For B/A-1/1 treated fabrics a significant improvement of the crease resistance was observed. FT-IR spectra revealed the formation of an imide group and an ester linkage, indicating the cross-linking of the cellulosic material. SEM images showed a smooth surface. As evidenced by TGA data the thermal stability of the cotton samples was not increased. No hydrophobicity could be observed. For B/A-1/3 and (B/A-1/4) modified cotton samples no crease resistant properties were detected. However, enhanced contact angle values were measured. A reaction mechanism for the formation of the ladder-like polysilsesquioxane and the cross-linking reaction is proposed.

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1. Introduction

Cellulosic material is chemically modified in an attempt to impart specific properties to cotton fabrics or garments. Thus, cotton textiles are reacted with crease resistant finishing agents, which react with the hydroxyl groups of the cellulose to improve the wrinkle resistance and the dimensional stability. As a consequence the mechanical strength is decreased. The most widely used crease resistant finishing agent is dimethyloldihydroxyethyleneurea (DMDHEU), since this finishing chemical is very effective and inexpensive. However, DMDHEU tends to release the potential human carcinogen formaldehyde during the curing process, the storage and the consumer use (Bajaj, 2002; Hewson, 1994). Polycarboxylic acids, such as 1,2,3,4-butanetetracarboxylic acid (BTCA) or citric acid (CA) in conjunction with sodium hypophosphite (SHP) as appropriate catalyst are considered to be capable of replacing DMDHEU. At elevated temperatures (180 °C, 90 s) the carboxyl groups of BTCA react with the hydroxyl groups of the cellulose via a five-membered cyclic anhydride forming an ester linkage (Harifi & Montazer, 2012; Welch & Andrews, 1989; Welch, 1990).

Different approaches have been used to substitute SHP as catalyst due to its adverse impact to the environment (Choi, 1993;

Choi, Li, Goodin, & Pratt, 1994). A reduction of the phosphorus-containing catalyst was obtained using nano-TiO₂ as co-catalyst (Nazari, Montazer, Rashidi, Yazdanshenas, & Anary-Abbasinejad, 2009; Yuen et al., 2007).

To produce a novel formaldehyde-free crease resistant finishing agent BTCA was reacted with APTES. The latter had been subjected to a hydrolysis reaction according to the sol–gel process. In general, the sol–gel technology is based on the hydrolysis and the ensuing condensation of tetraalkoxysilanes Si(OR)₄, such as tetraethoxysilane (R = CH₂CH₃, TEOS) or of organotrialkoxysilanes R¹Si(OR²)₃, such as methyltriethoxysilane (R¹ = –CH₃, R² = CH₂CH₃, MTEOS), thus forming a three dimensional siloxane network (Brinker & Scherer, 1990; Hench & West, 1990; Wright & Sommerdijk, 2001). This sol–gel method is also extensively applied to impart new properties to cotton textiles, such as antimicrobial function (Lee, Broughton, Akdag, Worley, & Huang, 2007; Montazer & Afjeh, 2007), flame retardancy (Blanchard & Graves, 2002; Yang & Wu, 2003), water repellency (Gashti, Alimohammadi, & Shamei, 2012; Wang, Ding, Xue, Wang, & Lin, 2011) or other functional properties (Mahlitig & Textor, 2008).

Various studies had been carried out to improve the non-formaldehyde crease resistant properties of cotton fabrics using sol–gel technology (Ibrahim, Refai, Ahmed, & Youssef, 2005; Lam, Kan, & Yuen, 2011; Schramm, Binder, & Tessadri, 2004).

The major objective of this paper is to present the results that were obtained, when cotton fabrics were treated with a BTCA/APTES solution. The as-treated cellulosic material was subjected to a curing process and subsequently evaluated in terms

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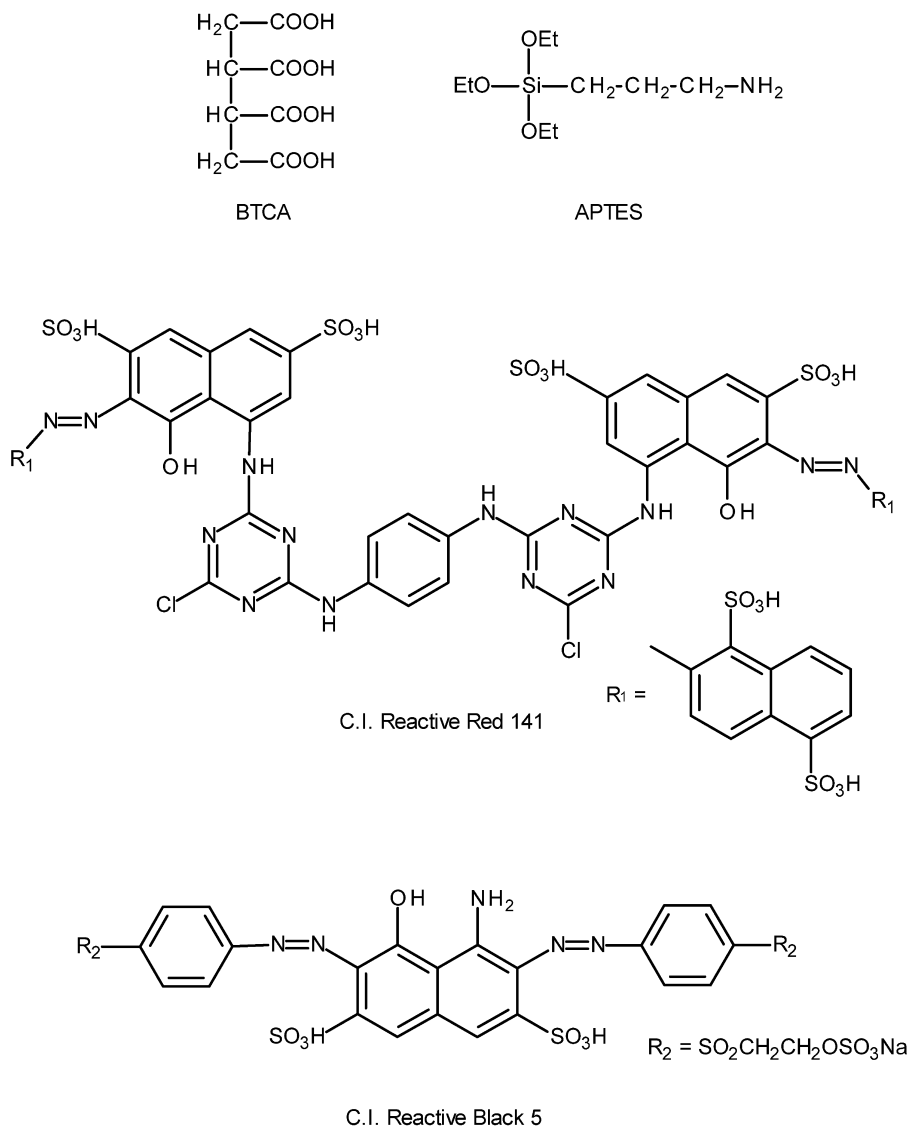


Fig. 1. Formula of substances of interest.

of crease resistant properties, hydrophobic behavior and thermal stability. This approach had been selected since a previous investigation has shown that the reaction of BTCA with APTES results in the formation of a poly(amic) acid, which gives rise to the formation of a cyclic imide when cured at elevated temperature (Schramm et al., 2012).

2. Experimental

2.1. Materials

Desized, scoured, bleached and mercerized 100% cotton fabric, weighing 109 g/m^2 , was used throughout the investigation. The dyes: C.I. Reactive Red 141 and C.I. Reactive Black 5 (Fig. 1) as well as the textile auxiliaries were supplied by BEZEMA AG, Montlingen, Switzerland. 3-Aminopropyltriethoxysilane (100%, APTES, Fig. 1) was obtained from Wacker Silicone, Burghausen, Germany. 1,2,3,4-Butanetetracarboxylic acid (>99%, BTCA) (Fig. 1), was purchased from Merck GmbH, Germany. Deionized water was used throughout the investigation. The chemicals were used without further purification.

2.2. Preparation of the precursor solution

The standard procedure for the synthesis of a BTCA/APTES solution (molar ratio 1/1) in an aqueous medium was as follows: APTES (7.07 mL, 30 mmol) was hydrolyzed with HCl ($c = 0.05 \text{ mol/L}$, 2.70 mL) in deionized water (20 mL) under magnetic stirring (500 rpm) in a polyethylene beaker (100 mL) for 15 h at room temperature (RT). Subsequently, 20 mmol of BTCA (7.02 g, 30 mmol) were added under vigorous stirring. Stirring was continued for 30 min and the solution was filled to 100 mL with deionized water.

2.3. Finishing of the cotton fabrics

The pre-weighed cotton fabrics (undyed and dyed) were impregnated with the corresponding BTCA/APTES solution applying a two-roll laboratory padder (HVL 500 Mathis AG, Niederhasli, Switzerland; air pressure 1 bar, rotary speed 3 m/min). After drying (5 min, 105°C) the as-treated fabric was cured in a lab dryer (LTE, W. Mathis AG, Switzerland) at a specified temperature and for a specified time.

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