Indian Journal of Fibre & Textile Research
Vol. 45, March 2020, pp. 96-101

Effect of textile softeners on BTCA treated cotton fabric

Archana Mahapatra[1, a] , Sharmila Patil[1], A Arputharaj[1], V D Gotmare[2] & P G Patil[1]

[1] ICAR- Central Institute for Research on Cotton Technology, Adenwala Road, Matunga, Mumbai 400 019, India

[2] Textile Manufactures Department, Veermata Jijabai Technological Institute, Matunga, Mumbai 400 031, India

Received 30 January 2018; revised received and accepted 8 January 2019

The effect of two softeners (Sapamine® OC and Ultratex® ASG) on physical properties of BTCA finished cotton fabric has been studied. Bleached cotton fabrics are treated with 1, 2, 3, 4-butane tetracarboxylic acid (BTCA) followed by finishing with a cationic and a silicone softener by conventional pad-dry-cure method. Properties, such as crease recovery angle, absorbency, whiteness index, tensile strength, moisture regain, surface morphology and crystallinity of the finished fabrics, have been evaluated. The crease recovery angle of the finished fabrics increases from 154° to 257° after treatment with BTCA and further increases to 266° after BTCA+silicone softener based finishing. The absorbency of cationic softener finished fabric is found to be higher than that of silicone softener finished fabrics. Tensile strength reduces after BTCA finishing by more than 50%, and about 17% softeners is recovered. Scanning electron microscope images show smooth ridges and surface characteristics of cotton fibres in BTCA treated samples as well as deposition of softener on the fibre surface in cationic and silicone softener finished fabrics.

Keywords: 1, 2, 3, 4-butane tetracarboxylic acid, Absorbency, Anticrease agent, Cationic softener, Cotton fabric, Crease recovery angle, Silicone softener, Whiteness index

1 Introduction

Cotton is one of the most popular natural fibres and widely used in textile industry. It possesses excellent properties like high water absorbency, strength, durability, softness, dyeability and biodegradability. Being a cellulosic fibre, the hydrogen bond in cotton fabric tends to be weak. Upon coming in contact with humidity, sweat or moisture, new hydrogen bonds are formed between fibres and the location where these bonds are formed, come up as wrinkle. Formation of wrinkle depends on various factors, like type and size of fibre, thickness of fabric, fabric knit, warp- weft density and yarn twist[1].

Cotton fabrics are chemically treated using crosslinking agents to impart crease resistant finishing. The crease resistant or anti-wrinke finish further results in improved comfort and pilling performance, eases maintenance, reduces shrinkage and dries the fabric faster. Crosslinking of cellulose with formaldehyde-based compounds, like dimethylol dihydroxy ethylene urea, (DMDHEU), improves wrinkle resistance, but releases carcinogenic formaldehyde, restricting its use in textile industry[2, 3]. Polycarboxylic acids, such as citric acid and BTCA (1, 2, 3, 4-butanetetracarboxylic acid), are the most promising alternative to conventional formaldehyde based anti-wrinkle agents. Polycarboxylic acid esterifies cellulose through the formation of a five-membered cyclic anhydride intermediate by the dehydration of two adjacent carboxyl groups. The formation of cyclic anhydrides at lower temperature is usually a slow process which can be accelerated by using catalyst like sodium hypophosphite (SHP)[4, 5]. Finishing of cotton fabric using BTCA has been reported by various researchers [6-12]. Although polycarboxylic acids impart crease resistant property to cotton fabric, but it results in reduction of breaking and abrasion resistance, loss of tensile strength and yellowing. It also imparts a stiff harsh, uncomfortable feel[8, 13-15] to the fabric.

However, these effects of BTCA finishing can be overcome by using fabric softener to the wrinkle free garment. Softener modifies the hand or feel of a fabric. It also lubricates the fibres to increase abrasion resistance and tear strength. It improves soiling resistance, crease recovery, static protection and stretch, reduces pilling, color fastness, moisture absorbency, flammability, sewing thread breakage and needle cutting when the garment is sewn. Softeners function as a sewing lubricant, as high needle speeds can generate heat to melt and fuse synthetic fibres, causing rejection of fabric by customer[16-20].

[a]Corresponding author.
E-mail: archanamahapatra89@gmail.com

Many studies have been conducted on the effect of different types and concentrations of softeners as well as auxiliary treatments on various physical, thermal and functional characteristic of various fabrics [21-25]. However, effect of anti-wrinkle finishing in combination with softener on physical comfort property of fabric is still unexplored. The present study aims to compare the effect of cationic and silicone based softeners on physical properties of anti-crease finished cotton fabric.

2 Materials and Methods

2.1 Materials

Desized, scoured and bleached cotton fabric was used in this work. The specifications of the fabric were: plain weave, 74 picks/inch, 79 ends/inch, 124 g/m^2 weight, 32 Ne warp count and 34 Ne weft count. 1, 2, 3, 4- Butane tetracarboxylic acid and sodium hypophosphite were procured from Merck Ind Pvt Ltd. Sapamine® OC textile softener and Ultratex® ASG silicone softener were purchased from Huntsman International Pvt. Ltd.

2.2 Methods

Specimen fabrics of 45 cm × 30 cm were subjected to various treatments as shown in Table 1. For finishing, each sample was impregnated with finishing solution of the respective recipe for 5 min at 30°C, keeping material-to-liquor ratio at 1:15. Then, it was squeezed using laboratory scale padding mangle at a wet pickup of 80%. The fabrics were subsequently dried at 85°C for 5 min followed by curing at 180°C for 3 min (M/s RB Electronics, Mumbai).

2.3 Testing

Samples were tested according to standard test methods. The crease recovery angle was measured according to "Indian standard method for determination of recovery from creasing of textile fabrics by measuring the angle of recovery" IS: 4681-1981. Absorbency of fabric was measured by AATCC test method 79- 2010. The degree of whiteness expressed as whiteness index was determined using computer color matching system (Spectra Scan 5100). The instrument was first calibrated using the standard tile, followed by scanning of the samples. Whiteness index was calculated using CIE method for D65 and 10 degree observer function. Tensile strength (kg. force) was measured according to ASTM D503-06 method using Instron tensile testing machine (5566 AR 3947 Model, England), equipped with 2 KN load cell and the testing was conducted at a speed of 300 mm/min. The moisture regain of the untreated and treated fabric samples was determined as per "Indian Standard method for estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials" IS 199: 1989. The results obtained were the statistical average of ten tests for warp and weft direction each.

Table 1 — Experimental design for various fabric treatment

Sample code	BTCA %	Sodium hypophosphite %	Softener	
			Cationic g/L	Silicone g/L
Control	-	-	-	-
T1	8	4	-	-
T2	-	-	10	-
T3	-	-	-	10
T4	8	4	10	-
T5	8	4	-	10

Surface Morphology

The surface morphology was studied using scanning electron microscope (Philips XL30 SEM, Netherlands) with an accelerating voltage of 10 kV at ×2000 magnification.

X- ray Diffraction

X- ray diffraction of the fabric samples was carried out in PANalytical X'Pert PRO diffractometer. Samples were ground using a Wiley mill (Arthur H. Thomas Co., USA) with a 200 steel mesh screen prior to testing. Equatorial scans were performed on the X-ray diffractometer at 40 kV and 30 mA with 2θ varying from 5° to 60°, at 0.01° scan step.

The crystallinity index (C.I.) was determined as [26]:

$$C.I. = \frac{I_{22} - I_{18}}{I_{22}} \times 100$$

where I_{22} and I_{18} are the intensity at 2θ angle of 22° and 18° representing the crystalline material and amorphous material in cellulose respectively.

FTIR Spectroscopy Analysis

FTIR spectroscopy of the fabrics was done using IRPrestige-21 (M/s Shimadzu) spectrophotometer. The analysis was carried out at 30°C using ATR accessory in the range of 700-4000 cm^{-1}.

3 Results and Discussion

3.1 Crease Recovery Angle

The fabrics evaluated for wrinkle free proportion in terms of crease recovery angle are shown in Fig. 1. Higher value of CRA depicts more wrinkle free properties of the fabric. The CRA increases from 154° to 257° after treatment with BTCA (T1). Similarly, the CRA of fabric treated with both softener and BTCA (T4 & T5) is bound significantly higher than those treated with only softener. CRA increases by 64% and 59% when fabrics are treated with both as compared to only softener treatment. During the curing process, the poly carboxylic acid reacts with the cellulose molecules of cotton fabric most probably through the formation of cyclic anhydrides as reactive intermediates, which, in turn, esterify cotton cellulose. Due to increase of ester carbonyl band intensity ratio the crease recovery property also increases[8]. CRA of cationic softener treated sample is found to be less than that of original untreated sample, which may be due to the lubricating effect of the cationic softener. Highest CRA has been recorded for BTCA with silicone softener treatment, which can be attributed to the combined anti-wrinkle function of BTCA as well as silicone.

3.2 Water Absorbency

Water absorption quantifies the rate at which a fabric can absorb sweat generated by the body. In order to prevent wet clinging, absorption should be low at the surface of the fabric which makes contact with the skin. The result of the water absorbency test is given in Table 2. The absorbency slightly decreases after treatment with BTCA, although the change is not huge. Similarly, there is no difference found between absorbency of samples T3 and T5, as both are treated with silicone softener.

The cellulose present in cotton fibre consists of both crystalline and amorphous regions, of which the former does not contribute to water absorption significantly. The dipole force between water molecules and hydroxyl group of cellulose facilitates absorption of water by cotton fabric. Crosslinking of the free hydroxyl group with BTCA inhibits water absorption by cellulose, decreasing the water absorption capacity of fabric.

The absorbency decreases in cationic softener finishing, and it further reduces in BTCA- cationic softener treated fabric sample. Depending on the ionic nature of the softener molecule and the relative hydrophobicity of the fibre surface, cationic softeners orient themselves. The positively charged ends are oriented towards the partially negatively charged fabrics due to zeta potential, creating a new surface of carbon chain. The so formed carbon chain is hydrophobic in nature and provides the characteristic softening and lubricity with cationic softeners. The hydrophobicity thus leads to longer wetting time of the fabric. Silicone softener although is non-ionic in nature, but still the water droplet is not absorbed by the fabric even after 5 min. The methyl group is oriented and attached to the fibre surface by silicone links leading to water repellency of silicone [27].

3.3 Whiteness Index

Whiteness index (WI) is the most important requirement of finished fabric and is used to evaluate the whiteness of fabric after anti crease finishing. A higher whiteness index indicates a lighter shade of the fabric. The effect of different treatments on whiteness of cotton fabric is shown in Table 2. It is found that the samples finished with both BTCA and softeners are slightly whiter than untreated fabric. However, the whiteness index value decreases in fabrics treated with only softeners. The reduction in whiteness index

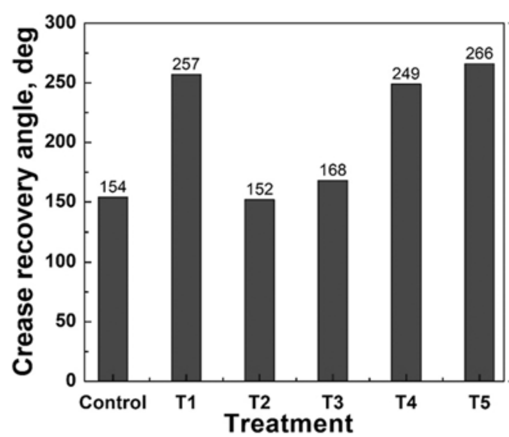

Fig. 1 — Effect of BTCA and softeners on crease recovery angle of cotton fabric samples

Table 2 — Effect of BTCA and softeners on absorbency, whiteness index and moisture regain of cotton fabric

Treatment	Absorbency s	Whiteness index	Moisture regain %
Control	1±0.1	65±0.10	7.5±0.05
T1	2±0.1	65±0.16	7.25±0.03
T2	9±0.4	55±0.21	7.4±0.08
T3	>300	51±0.11	7.4±0.12
T4	39±1.3	68±0.26	7.2±0.01
T5	>300	68±0.13	7.1±0.07

of samples treated with cationic and silicone softeners may be attributed to the high curing temperature[28].

3.4 Tensile Strength

The tensile strength describes the response of a textile material when an external force is exerted on the cellulosic specimen until it breaks. This mechanical property is influenced by numerous parameters, such as molecular structure, fibre properties as well as the structure of the fabric. Especially, cross-linking is a very contributing factor to tensile properties, since cross-linking prevents the dissipation of the strain energy and thus worsen tensile properties. The tensile strength of untreated and treated fabrics is shown in Fig. 2. The strength reduces from 20.2 kgf in untreated cotton fabric to 8.3 kgf in BTCA treated fabric, reducing by more than 50%. The strength of fabrics treated with either type of softener also decreases marginally. The tensile strength further decreases in fabrics treated with both BTCA and softener. Softener combined with anti-wrinkle finishing slightly improves the tensile strength of fabric in the weft direction as compared to BTCA treatment only. The crosslinking of cellulose inhibits distribution of tear stress over many molecules which can slightly shift the external forces [29]. Further, the acidity of the finishing solution results in a reduction of the tensile strength[29].

3.5 Moisture Regain

The moisture regain of the untreated and finished fabric samples is depicted in Table 2. The moisture regain of untreated cotton fabric is 7.5% which decreases in the treated fabrics. The moisture regain decreases in the softener treated fabrics (T2 & T3). Also, the reduction in moisture regain is found more in fabric treated with both BTCA and softener (T4 & T5). The amount of moisture absorbed by a fabric from the atmosphere depends mostly on the structure of yarn and fabric, type of fibre and ambient humidity. The moisture regain of cotton fibres is a function of the internal surface area of the fibre. The hydroxyl groups in the accessible surface of fabric usually absorbs water. Upon cross linking of fabrics as in anti-wrinkle finishing, the hydroxyl group is cross lined with BTCA, reducing the moisture regain of fabric. Further, softener finishing results in decreased internal surface area of fabric due to deposition of softener in the fabric [30].

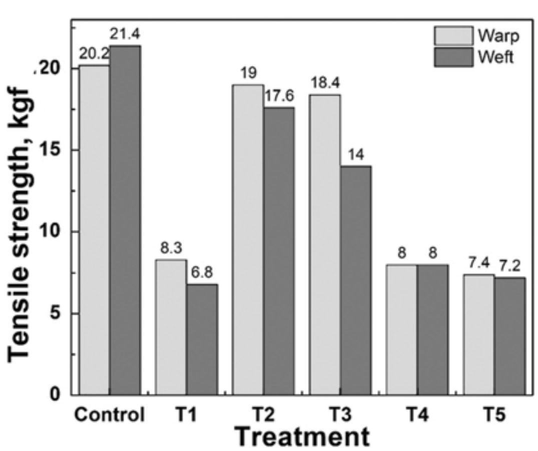

Fig. 2 — Tensile strength of treated and untreated fabric samples

3.6 Surface Morphology

Apart from the possible changes in chemistry, crosslinking reaction may also alter the morphological form of the fibre and SEM could serve as an effective tool to characterize such changes. The scanning electron microscope images of untreated and treated samples are shown in Fig. 3. Figure 3(a) represents the SEM image of the control sample which is scoured and bleached and not undergone any damaging treatment. The SEM shows the normal spiral structure of the cotton sample, which is clearly defined. It shows typical fibres with twisted, wrinkled and harsh surfaces that are produced when fibres from the boll dehydrate upon boll opening [31]. The characteristic parallel ridges fibril with smooth ridges, concave grooves spiraling around the fibre at an acute angle to its axis and protruding fibrils can also be seen. After the cotton samples are treated with 8% BTCA, as shown in Fig. 3(b), smooth ridges and smooth surface characteristic are observed although concave grooves still appeared. Figures 3 (c) and (d) show deposition of softener over the fibre. The primary function of a fabric softener is to lubricate the surface by coating the fibres with a thin film layer. Figures 3(e) and (f) show deposition of softener as well as a smooth fibre surface due to combined effect of softener and BTCA.

3.7 XRD Study

The X-ray diffraction results, as depicted in Fig. 4, show that the crystallinity of treated and untreated samples ranges between 80% and 84%. There is no significant reduction in the crystallinity of treated and control fabric samples. Since crosslinking chemicals mostly react with –OH groups of amorphous region without affecting the crystalline part of the cellulose, the crystallinity of the BTCA and softener treated fabrics do not change.

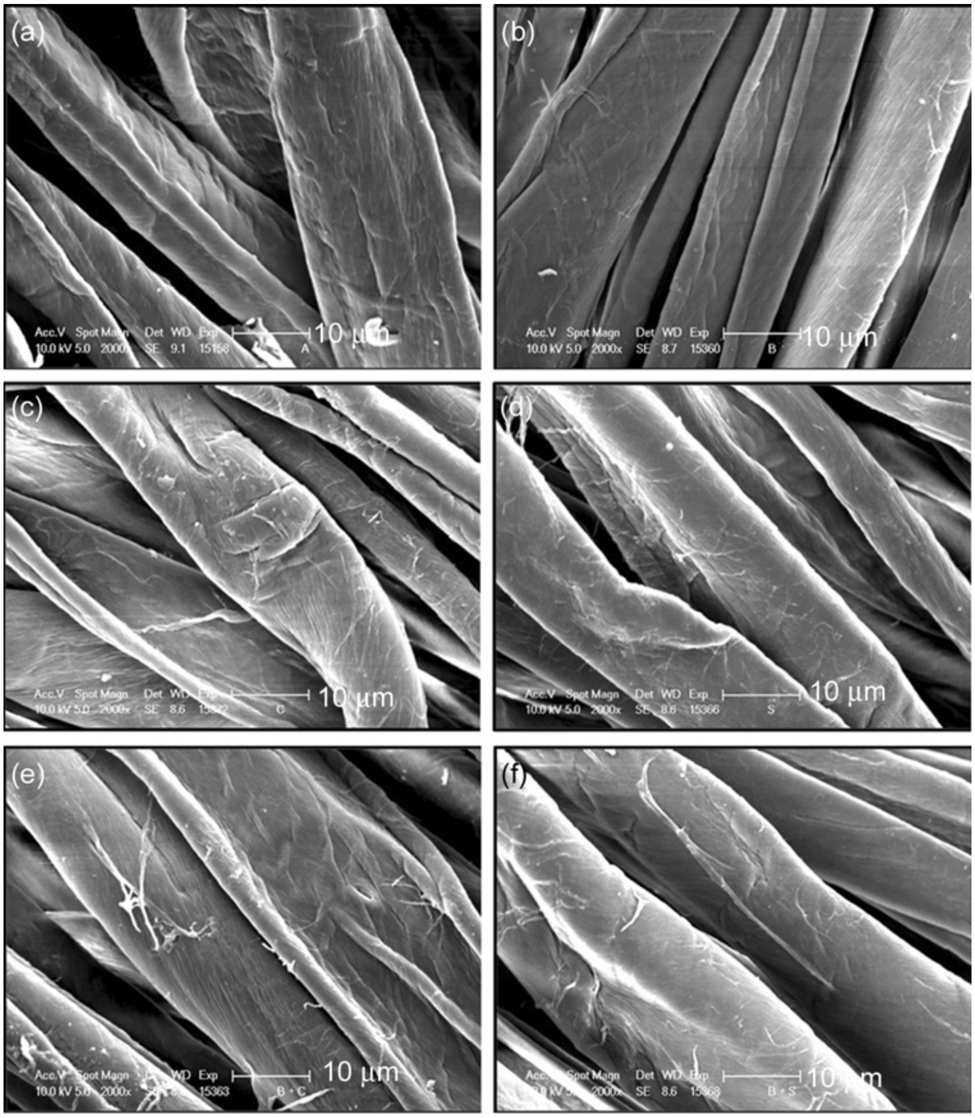

Fig. 3 — SEM images of (a) control, (b) BTCA, (c) cationic softener, (d) silicone softener, (e) BTCA + cationic softener and (f) BTCA + silicone softener treated fabric samples

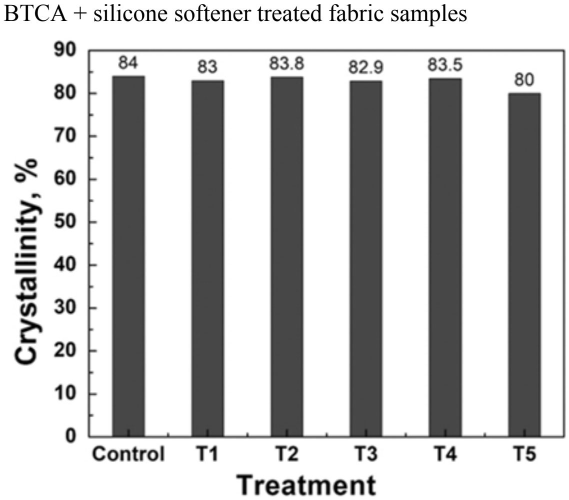

Fig. 4 — X- Ray diffraction study showing crystallinity of fabric samples

3.8 FTIR Study

The ATR spectra of the characteristic peaks of the control and treated cotton fabrics are shown in Fig. 5. The band at the wavelength of 1728 cm^{-1} corresponds to the ester carbonyl group (which confirms the covalent bond between the cellulose and the BTCA in case of BTCA treated samples. This band is also present in the spectra of samples finished with both BTCA and softener. However, the ester-carbonyl stretching cannot be observed in the spectrum of the untreated fabric. The intensity of the H-bonded hydroxyl (O-H) stretching band of cellulose at the wavelength remarked in the untreated cotton spectrum decreases in the spectra of BTCA treated cotton samples. This peak gets narrow because of the decrease

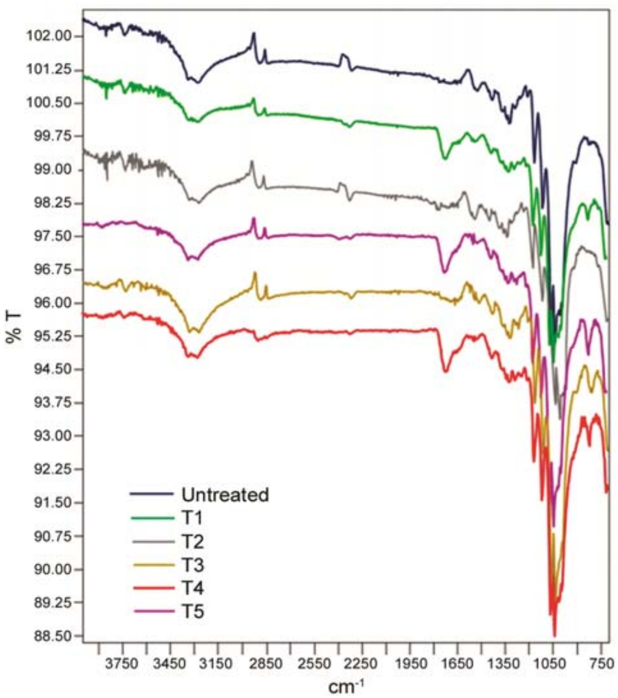

Fig. 5 — FTIR image of control and treated cotton fabric samples

of H-bonded -OH groups of cross-linked cellulose. It proves the esterification reaction between carboxylic acid groups of poly carboxylic acid and hydroxyl groups of cellulose. Based on these results, it is concluded that the cellulose macromolecules are cross-linked by the esterification reaction of the BTCA.

4 Conclusion

The effect of anti-wrinkle treatment and cationic and silicone softener finishing on cotton fabric has been studied. The crease recovery angle is improved significantly by BTCA as well as silicone softener finishing. Both anti-crease finishing and softening treatment decreases the absorbency of cotton fabric. Silicone softener imparts hydrophobicity, also known as lotus effect to the fabric. Strength reduces drastically in BTCA treated fabric with and without addition of softener by more than 50%. The strength of fabrics treated with either type of softener also decreases marginally. Crease resistant finishing results in whiter fabric. Further, the anti-wrinkle and softening treatments do not affect the crystallinity of fabric significantly. The FTIR spectra illustrates effective crosslinking of BTCA with the fibre.

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