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Microwave curing for producing cotton fabrics with easy care and antibacterial properties

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ABSTRACT

A new microwave curing system was used to affect crosslinking of cotton fabric with nonformaldehyde finishes, namely, glyoxal, glutaraldehyde and 1,2,3,4 butanetetracarboxylic acid (BTCA). Water soluble chitosan was incorporated in the finishing bath in order to impart antibacterial activity to the fabric in addition to the ease of care characteristics. Glyoxal proved to be the best finish and, hence, it was studied along with the chitosan under a variety of conditions including chitosan concentrations, power and time of microwave curing. Besides the crease recovery and strength properties of the finished fabrics, the latter were also monitored for *N*%, antibacterial activity and characterized using scanning electron microscope (SEM) and FTIR spectra when compared. With conventional curing system, the microwave curing system was found advantageous in production of cotton fabrics with easy care antibacterial properties without high losses in strength properties.

1. Introduction

Cotton fabrics are often subjected to chemical finishing using crosslinking agents in order to convey easy care properties. 1,2,3,4 Butanetetracarboxylic acid, glyoxal and glutaraldehyde (Fig. 1a–c) have been investigated as possible substitutes for formaldehyde-emitting N-methylol compounds (Kittinavarat, Kantuptim, & Singhaboonponp, 2006; Purwa & Joshi, 2005; Schramm & Rinderer, 2002).

Durable press finishing involves the use of chemical crosslinking agents that can covalently crosslink two adjacent cellulose polymer chains within cotton fibers. The crosslinks not only result in the fabrics wrinkle resistance but also in discoloration and impairment of fabric strength and other mechanical properties (Vukusic & Katovic, 2003). By and large increased crosslinking is associated with decreased fabric strength. Crosslinking between cellulose molecules stiffens the cellulosic macromolecular network and causes fiber embrittlement that reduces the treated fabrics mechanical strength (Kang, Yang, & Wei, 1998). A conventional curing system may also contribute to the decrease in crosslinking mechanical properties. This conventional curing system involves heat transfer to the fabric by convection, conduction and radiation. The surface of the cotton fabric becomes hotter than the interior leading to baking and non-uniform crosslink distribution throughout the fabric material.

Microwave curing is a possible alternative to conventional curing for improving the mechanical properties of crosslinked textile materials. This method can generate heat uniformly throughout the textile substrate. Microwave radiation is absorbed by molecules having resonant frequencies in the microwave spectral region. When an electric field is applied at microwave frequencies, polar molecules rotate to align their dipole moment with the charging electrical field. Energy is absorbed and heat is generated by the internal friction between the rotating molecules (Zhang, Chen, JI, Hung, & Chen, 2003).

One of the most outstanding advantages of microwave technology is volumetric heating which minimizes the damage from over drying. Since heat energy is transferred through the material electromagnetically, and not as a heat flux, it is not limited by the volume of the material (Vukusic, Katovic, & Grgec, 2004). Microwave heating of the textile fabric is almost instantaneous throughout the material, so that the fabric's environment insulates it against heat loss from the surface thereby allowing curing.

Chitosan, a polysaccharide, is obtained by deacetylating chitin (Shin, Yoo, & Jang, 2001; Shin, Yoo, & Min, 1999). The structure of chitosan is similar to that of cellulose except for the hydroxyl groups on the carbon atom number two have been replaced by amino group. It has been useful in many areas of applications, such as wastewater treatment, food and textile industry and recently in drug industry and as hydrating agent in cosmetics (Kumar, Bristow, Smith, & Payne, 2000; Martine & de la Orden, 2006; Vasiliu, Popa, & Rinaudo, 2005).

In this study, efforts are paid to use microwave technology for curing cotton fabrics treated with nonformaldehyde finishing

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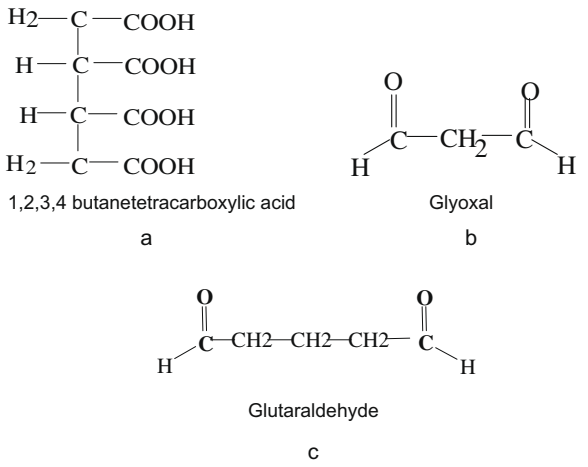

Fig. 1. Structure of nonformaldehyde-emitting crosslinking agents.

agents along with water soluble chitosan. With a view to impart ease care and antibacterial properties to the fabrics. Finishing agents comprised glyoxal, glutraldehyde and 1,2,3,4 butanetetracarboxylic acid.

2. Experimental

2.1. Material

Bleached 100% cotton fabric was kindly supplied by Misr Company for spinning and weaving Mehalla El Kobra, Egypt. Water soluble chitosan supplied by ChiPro Company. Glyoxal 40% solution, glutaraldehyde and BTCA were purchased from Merck, GmbH, Germany. Magnesium chloride and sodium hypophosphite was laboratory grade chemicals.

2.2. Testing and analysis

Crease recovery angle (CRA) of the treated samples were determined according to AATCC standard test Method 66-1996.

Tensile strength (TS) and elongation at break were determined according to ASTM standard test Method, D 1682-94, 1994.

FTIR spectroscopy was carried out on the sample in pellet form, made from finely chapped fiber (2 mg) and KBr (45 mg) using 400 kg/cm^2 pressure for 10 min, on a perkin Elmer Bx 11-FTIR spectrometer in transmittance made.

Scanning electron microscope (SEM) was used to monitor changes in surface characteristics of the cotton fabrics due to crosslinking.

2.3. Fabric treatment

Fabric samples were impregnated for 5 min in pad baths each containing 0.8% (owing of weight of bath) owb water soluble chitosan and 5% owb of different crosslinkers each of them with its compatible catalyst. For glyoxal, glutaraldehyde, Magnesium chloride (10 g/l) was used, and for BTCA, sodium hypophosphite 5% owb was used. Samples were then padded through laboratory padder with two dips and two nips to give a wet pick up of 100% (owing of weight of fabric) owf. Immediately after padding, half of the samples were simultaneously dried and cured in a microwave oven at 200 W/2 min. The other half fabric samples were dried at 85 °C for 5 min and cured at 140 °C for 3 min in case of glyoxal and glutaraldehyde and at 180 °C for 90 s in case of BTCA. After curing, the fabric samples were rinsed with warm water for 15 min and dried at room temperature.

3. Results and discussion

To start with, cotton fabrics were independently treated with three finishing agents, namely, glyoxal, glutaraldehyde and 1,2,3,4 butantetracarboxylic acid in presence and absence of chitosan. Thus treated fabrics were subjected to curing as per two systems: the conventional heat curing system and microwave curing system. After being washed and dried, the finished fabrics were monitored for CRA and TS. This was done to clarify differences in system performance among the microwave curing system and the conventional curing system when the finishing treatments were carried out with and without chitosan in the finishing baths. Results obtained are shown in Fig. 2.

Results of Fig. 2 signifies that in absence of chitosan the two curing systems under investigation bring about finished fabrics which exhibit significantly improved CRA but with the certainty that in case of the conventional curing system the CRA obtained with BTCA exceeds those of glyoxal and glutaraldehyde, on the contrary, glyoxal is the best candidate for finishing using the microwave curing system. Trends obtained with the two curing systems persist when the finishing treatments were conducted in presence of chitosan though the latter tends to marginally decrease the CRA.

Fig. 3 shows the tensile strength of cotton fabrics treated using the three finishing agents in presence and absence of chitosan as per the conventional curing system and the microwave curing system. As is evident fabrics treated in presence of chitosan and cured as per the microwave system display the highest tensile strength while those fabrics treated in absence of chitosan and cured according to the conventional system exhibit the least tensile strength. In both systems fabrics treated with glyoxal retain the highest tensile strength. This is observed in presence and absence of chitosan.

The favourable role of chitosan in retaining higher strength could be ascribed to the basicity created in the finishing bath as well as in the picked up liquor by the fabric. Such basicity would certainly detract from the severity of the acid catalyst thereby reducing the detrimental effect of the latter on the molecular degradation of cotton cellulose and, therefore on the tensile strength. It is as well

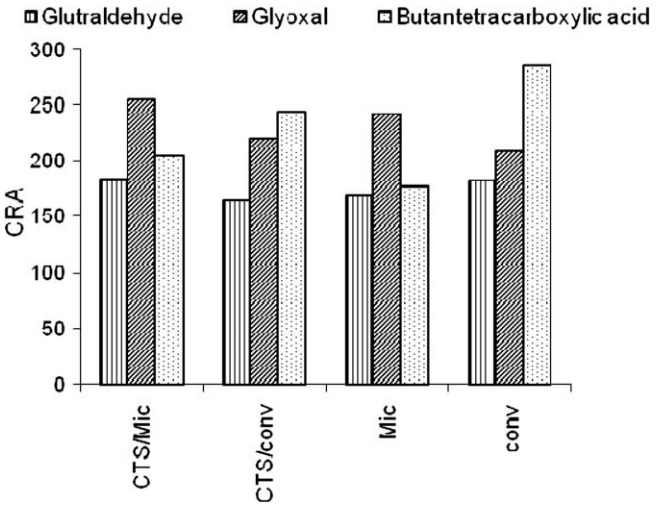

Fig. 2. Effect of application of different crosslinker, using microwave and conventional technique, on CRA. [Glutaraldehyde] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min, curing at 140 °C/3min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min. [Glyoxal] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min, curing at 140 °C /3 min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min. [BTCA] 5% owb, [SHP] 5% owb, drying at 80 °C/5 min, curing at 180 °C/1.5 min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min, 400 W/2 min, and 200 W/4 min.

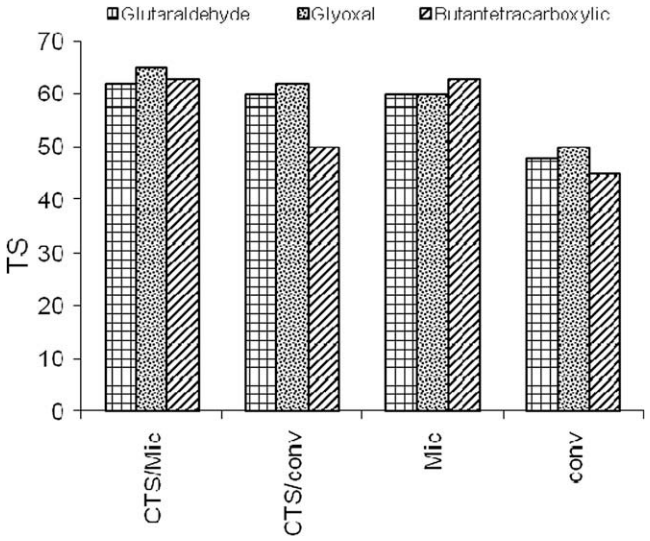

Fig. 3. Effect of application of different crosslinker, using microwave and conventional technique, on tensile strength (TS). [CTS] 0.8%. [Glutaraldehyde] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min, curing at 140 °C/3 min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min. [Glyoxal] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min, curing at 140 °C/3 min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min. [BTCA] 5% owb, [SHP] 5% owb, drying at 80 °C/5 min, curing at 180 °C/1.5 min, for treated and blank sample. In case of microwave, the same conc. was used but the curing was at 200 W/2 min, 400 W/2 min, and 200 W/4 min.

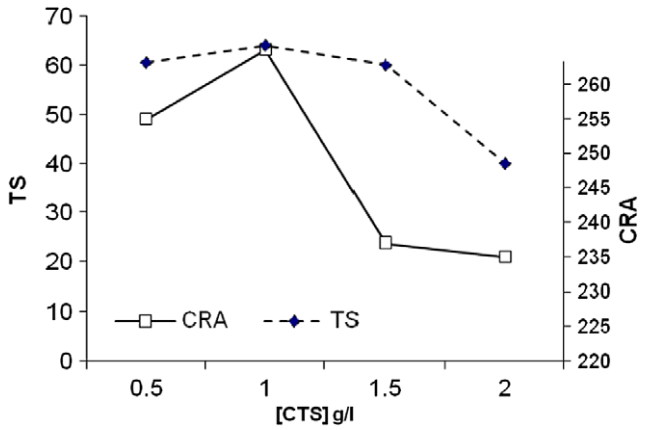

Fig. 4. Effect of chitosan concentration on CRA and TS by using the microwave technique. [Glyoxal] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min, microwave (200 W/2 min).

to emphasize that the mechanism of crosslinking of cotton fabrics occurs through esterification of the three hydroxyls, although not all the carboxylic acids will be able to react with cellulosic substrate (Ga & Yan, 1998; Hsieh, Chen, & Wei, 2003). The addition of water soluble chitosan in the finishing bath formulation provides the cellulose cationic properties, as in the case of BTCA, in the presence of sodium hypophosphite (SHP) as catalyst. While using glyoxal and glutaraldehyde as dialdehyde crosslinking agent, chitosan has a dual function; it acts as a catalyst as well as cationizing agent.

At any event, it is very likely that microwave energy heats the fabric uniformly from inside out. As a consequence a uniform distribution of crosslinks and polymerization occur throughout the microstructure. In addition, such heating uniformity prevents serious losses in strength without impairing the desired crease resistant durable press properties. This, indeed, affords considerable advantages compared to conventional surface heating, which is a non-uniform outside-in heating and might cause the surface, edges and corners to be much hotter than the inside of the fabric. Consequently, the quality of conventionally heated materials varies and frequently does not reach the desired level.

Based on the foregoing, glyoxal along with chitosan selected for further studies using the microwave curing system. Research was designed to investigate factors affecting such finishing treatments. Variable examined include concentration of chitosan, microwave power and time of exposure.

3.1. Chitosan concentration

Fig. 4 shows the effect of chitosan concentration (0.5–2% owb) on the performance properties, viz CRA and TS of the treated fabric. The finishing baths were prepared containing 5% owb glyoxal and $MgCl_2$ 1% owb, thus treated fabrics with 100% pick up where dried then exposed to microwave power 200 W for 2 min.

It is clear (Fig. 4) that the CRA of the treated fabrics which was cured using the indicated microwave condition is pronounced as chitosan concentration increases up to 1% then decreases sharply. The enhancement in CRA of the finished fabrics by increasing chitosan concentration suggests that chitosan performed two functions: (1) it reacts with glyoxal in the fiber molecules; (2) chitosan undergoes crosslinking with the fabric to form a network matrix. The water soluble chitosan with its low molecular weight penetrate the fiber more easily promoting anticreasing in the treated fabrics. Water soluble chitosan generates ether reaction with the hydroxyl groups in the fibers, forming a two dimensional structure that improved the crease resistance of the fabrics (Hsieh et al., 2003). Decrement in CRA by increasing the chitosan concentration above 1% could be associated with increased basicity of the finishing environment at higher chitosan concentrations. It is logical that basicity would stand as an inverse function to the acidity of the catalytic system of the crosslinking cotton cellulose with glyoxal. Lower catalysis would certainly lead to decreased CRA.

With respect to tensile strength (TS), on the other hand, penetration or encapsulation of chitosan molecules would improve the strength properties of the treated fabrics. As shown in Fig. 4, the tensile strength increases by increasing chitosan concentration up to 1.5% owb then tends to decrease thereafter. Rigidity conferred on the structure of cotton by inclusion of chitosan through various interactions with cotton and glyoxal may account for the decrease in TS at higher chitosan concentrations. It is also probable that higher concentrations of chitosan create more fibers bridging and are more likely to cause stress accumulation thereby decreasing the TS.

3.2. Curing time and power

Improvement in textile properties of fabrics finished with glyoxal 5% owb and 1% owb chitosan would be anticipated to rely greatly on the curing power and time. This expectation is, indeed, valid when the results of Table 1 are considered. Here curing was carried out using different microwave power (200 and 400 W) for 1, 2 and 3 min. As is evident the power acts in favour of CRA and TS only when the time of curing is short (1 min). Longer times decrease the TS without significant enhancement in CRA of treated fabrics. The elongation at break of treated fabrics does not affected much by the magnitude of power or time of curing. The same holds true for N% which is a measure of the amount of chitosan reacted with the fabrics.

Under microwave conditions, crosslinking occurs by making use of the energy generated by water molecule vibration and other ionic components, e.g. crosslinking agent and catalyst. Meanwhile, the deformation and breaking of some crosslinking may also occur. As treatment time increases, crosslink deformation may dominate over crosslink formation. Both the rate of crosslink formation and

Table 1
Effect of curing power and time on the cotton performance properties.

Power (W)	Time/min	CRA (°)	TS (kg)	*E* %	*N* %
200	1	252	60	20	0.167
	2	273	58	18	0.17
	3	271	55	18	0.165
400	1	269	54	18	0.169
	2	273	51	17	0.18
	3	275	49	16	0.168

[CTS] 1%, [glyoxal] 5% owb, [$MgCl_2$] 10 g/l, drying at 80 °C/5 min.

deformation increases as the power increases. Therefore, the final crease recovery angle depends on the rate of crosslink formation vs the rate of crosslink deformation at high power, a more stable crossslinked structure may have formed at shorter times, therefore minimizing deformation conversely the crease recovery angle of the treated fabrics was better, because with higher treatment power or longer times cotton fibers molecules swell more and finishing solution can enter more easily.

It is also noteworthy that since more crosslinking takes place when the reaction between the fibers and glyoxal is more active with higher power or longer time, the treated fibers become hardened and straightened, resulting in a greater loss of tensile strength of fabrics. Furthermore, long time enhances the hydrolysis of fibers in acid catalysts reducing the tensile strength of the fabric.

3.3. Antibacterial efficacy on the finished fabrics

Cotton fabric with good antibacterial activity could be obtained by crosslinking of cotton with glyoxal in presence of water soluble chitosan and catalyst. As detailed.

Different concentrations of water soluble chitosan (0.5–2% owb) were applied to cotton fabrics with 5% owb glyoxal, $MgCl_2$ 1% owb using microwave power 200 W/2 min. The antibacterial activity of the treated cotton fabrics are evaluated against *E. coli* and *Micrococcus luteus* according to TTC-test method (El-Shafei, Fouda, Knittel, & Schollmeyer, 2008). In Figs. 5 and 6 the absorbance of formazan is directly proportional to the number of active cells. The activity of the cells of both microorganisms decreases by increasing chitosan concentrations. There are two proposed mechanisms of antibacterial activity by chitosan. In one mechanism, the poly cationic nature of chitosan interferes with the bacterial metabolism by stacking at the cell surface (Xie, Liw, & Chen, 2007). The other mechanism is the binding of chitosan with DNA to inhibit mRNA synthesis. The latter is favourable because water soluble chitosan can easily penetrate into the cells (Lim & Hudson, 2004). But by increasing chitosan concentration its viscosity becomes larger and its penetration becomes difficult.

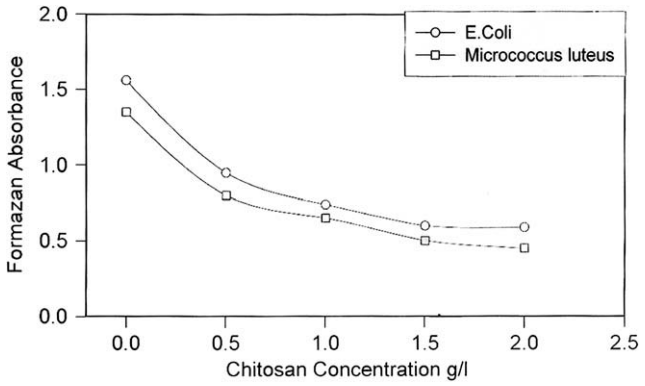

Fig. 5. Effect of chitosan conc. on the antibacterial properties of crosslinked cotton. [Glyoxal] 5%, microwave power, 200 W/2 min.

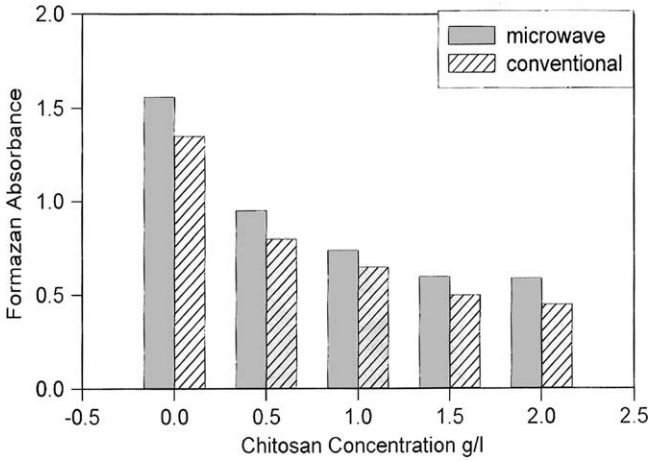

Fig. 6. Effect of chitosan conc. on the antibacterial properties of crosslinked cotton using microwave and conventional curing technique. [Glyoxal] 5%, microwave power, 200 W/2 min, conventional 130 °C/3 min.

Since the structure of glyoxal is similar to formaldehyde. Therefore, the glyoxal–chitosan treated fabric might decompose under certain condition and release the formaldehyde to inhibit the growth of bacteria (Kittinaovarat & Kantuptim, 2005). Also microwave technique is promising than conventional because of the easy of fixation of glyoxal–chitosan combination which lead to improved antibacterial efficiency.

3.4. Characterization of treated fabrics

The fourier transform infrared (FTIR) spectra of untreated and cotton treated ith glyoxal and chitosan under conventional and microwave curing are shown in Fig. 7).

A decrease in absorbance intensity at 3350, 1630, 1372 and 1161 cm^{-1} is observed in treated samples cured with conventional and microwave systems as compared with untreated cotton sample. A decrease in intensity at 3350 and 1630 cm^{-1} could be

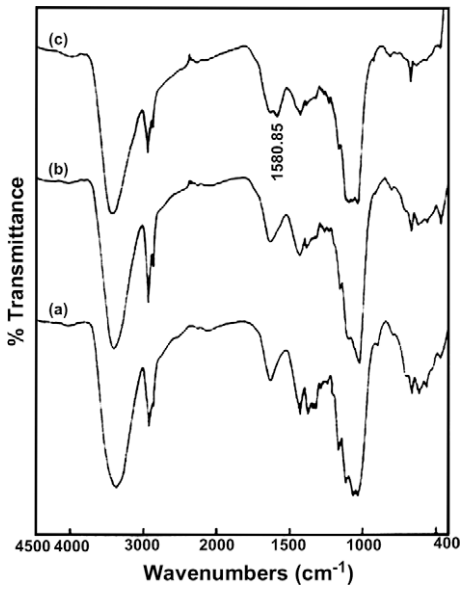

Fig. 7. FTIR spectra of cotton: (a) untreated, (b) conventionally cured (dried at 80 °C/5 min and cured at 140 °C/3 min, and (c) microwave cured 200 W/2 min.

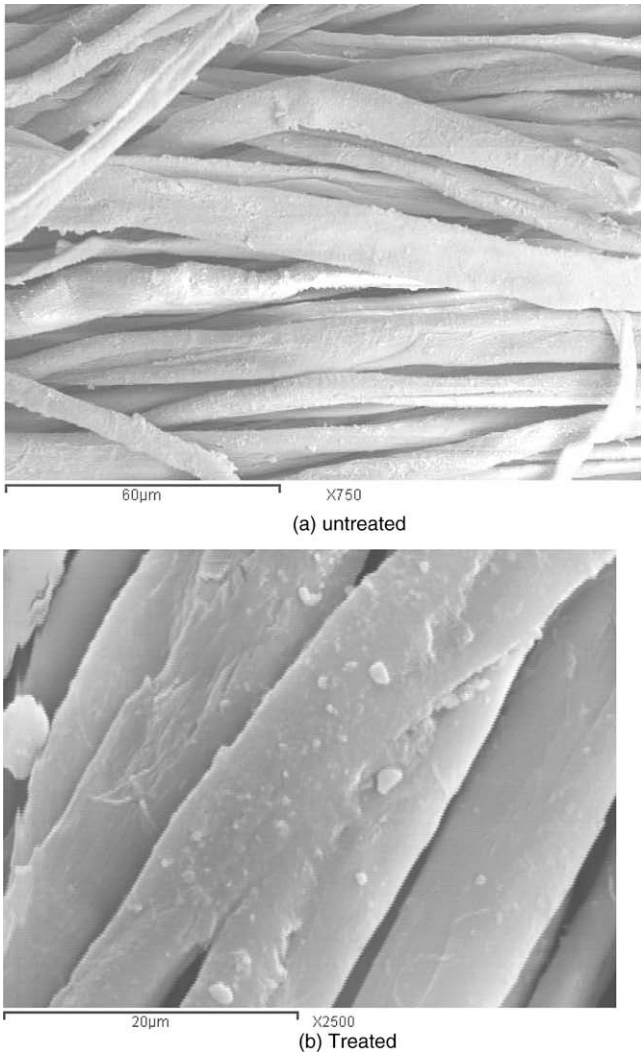

Fig. 8. (a and b): SEM of untreated and treated cotton under microwave curing.

attributed to a decrease in the total numbers of hydroxyl groups through crosslink formation between cotton and glyoxal.

The decrease in absorbance is higher in case the conventionally cured samples as compared to the microwave cured sample. This indicates that there are fewer hydroxyl groups associated with covalent bond formation, i.e. crosslinking, for microwave cured sample than for the conventionally cured sample (Bang, Lee, Kim, Yu, & Bae, 2007).

Fig. 8a and b shows the SEM pictures of fabric surface. The surface of treated cotton has a few white particles compared with blank this might be chitosan absorbed on the surface, indicating that the reaction has taken place on the surface.

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