Effect of Plasma Pretreatment on Enhancing Wrinkle Resistant Property of Cotton Fiber Treated with BTCA and TiO_2 System

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ABSTRACT: 1,2,3,4-butanetetracarboxylic acid (BTCA) with titanium dioxide as a catalyst, was used to crosslink cotton fibers for the purpose of enhancing wrinkle recovery angle (WRA). To enhance the BTCA treatment with TiO_2, surface modification of cotton fiber is required; atmospheric pressure plasma jet pretreatment was used in experiments reported in this article. In this study, optimum conditions for plasma pretreatment were analyzed using orthogonal array testing strategy (OATS) technique, on the basis of WRAs achieved after BTCA treatment with and without TiO_2 as catalyst. It was found that (i) longer duration of plasma pretreatment provides enough time for the substrate to be impacted by the concentrated active species produced in plasma gas and therefore, modifies the material surface effectively and offers the best balance between enhancement of WRA and minimization of fiber damage, (ii) high oxygen flow rate producing a severe etching effect that alters the material's surface characteristics. However, when concentration of O_2 increased during the plasma pretreatment, the active species might react with the oxygen also, besides the cotton surface, and (iii) when the distance between the plasma jet nozzle and the substrate surface is too large, plasma gas from the nozzle is unable to hit the fabric surface, which means no surface modification is achieved. As a result, plasma treatment with 2 mm/s treatment speed, 0.1 L/min oxygen flow rate, and 2 mm jet-to-substrate distance was the most effective plasma pretreatment. J Appl Polym Sci 124: 3341–3347, 2012

Key words: wrinkle-resistant finishing; titanium dioxide; plasma pretreatment; optimization; cotton; BTCA

INTRODUCTION

Cotton is the most important natural fiber used in the textile industry because of its inherent built-in performance properties.[1] For enhancing wrinkle resistant property of cotton fabric, 1,2,3,4-butanetetracarboxylic acid (BTCA), a promising nonformaldehyde crosslinking agent, has been applied to crosslink amorphous regions of cotton fibers.[1–3] In addition, previous studies have utilized titanium dioxide (TiO_2) as a catalyst to improve the crease recovery property that can enhance the finishing effect and minimize side effects.[4–9] To enhance the BTCA treatment with (or without) TiO_2, modification of surface of cotton fabric is required. Among various types of surface treatments, atmospheric pressure plasma jet (APPJ) is widely used in the textile industry to modify the fabric surface in an environment friendly process that reduces the use of wet chemicals and energy consumption.[10,11] Generally speaking, plasma treatment affects only the outer surface of the substrate, which is directly bombarded by plasma gas; the depth of penetration of plasma determines the quality of plasma treatment of textile fabrics.[11–14] Effectiveness of APPJ treatment depends on treatment speed (related to treatment time), gas flow rate, and jet-to-substrate distance, as well as substrate's pore size or structure.[10,12] However, detailed information concerning the effect of plasma pretreatment on wrinkle-resistant properties of cotton fabric, after BTCA treatment (with or without using TiO_2 as catalyst), has been seldom reported. In addition, optimization of effectiveness of plasma pretreatment on wrinkle resistant properties of treated fabric has also lacked attention. In this study, wrinkle resistant properties of cotton specimens pretreated with plasma, and then with BTCA, with and without using TiO_2 as catalyst, are investigated. The optimum conditions are investigated with orthogonal array testing strategy (OATS) technique, taking into consideration treatment speed (treatment time), oxygen flow rate, and jet-to-substrate distance.[15–18] Wrinkle recovery angle (WRA) of the fabric after BTCA treatment is used as the measure of optimality.

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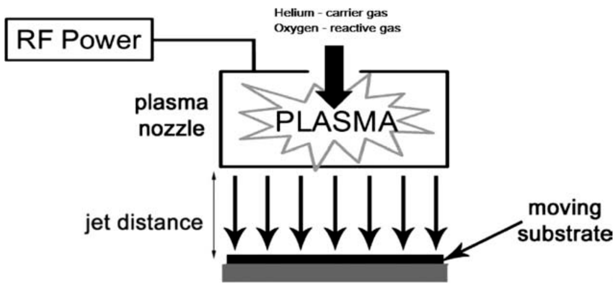

Figure 1 Schematic diagram of plasma pretreatment.

EXPERIMENT

The data collected from the experiment was within 5% analytical tolerance limit.

Material

A 25 cm × 25 cm piece of 100% semibleached plain weave cotton fabric (warp: 58 picks/cm, yarn count 40 tex; weft: 58 ends/cm, yarn count 38 tex; weight: 175 g/m^2) was used, which was purchased from Tai Tung Textiles Company, Hong Kong. The crosslinking agent was BTCA (98% purity), supplied by International Laboratory Ltd., San Burno, USA. The catalyst used was micro-titanium dioxide (TiO_2, average diameter 2 μm, purity over 99.5%) obtained from UniChem Ltd., North Carolina, USA. The particle size was further verified with a particle size analyzer (LS13320 Beckman Coulter, Beckman Coulter Inc., Pasadena, USA). All other chemicals used in the study were reagent grade.

Plasma pretreatment and its optimization

Plasma pretreatment of cotton fabric was carried out by an APPJ apparatus manufactured by Surfx Technologies, Culver City, USA. The cotton fabric was moved according to the specified treatment speed; the experimental set-up is shown in Figure 1. The machine produced a stable discharge at atmospheric pressure with radio frequency of 13.56 MHz at 80 W. The treatment was carried out using a rectangular nozzle which covered an active area of 25.4 mm × 1 mm, and was mounted vertically, above the cotton fabric. Helium (15 L/min) and oxygen were used as carrier and reactive gas, respectively.

The effect of plasma pretreatment on the final fabric surface properties depends on treatment parameters namely, treatment speed (treatment time), oxygen flow rate, and jet-to-substrate distance, because they are interrelated to each other. Three levels were set for each parameter,[19] as shown in Table I. According to the rules of the OATS technique, nine test runs were conducted (nine combinations of the three parameters). Levels of individual parameters and the arrangement of experimental trials are summarized in Table II.

TABLE I
Parameters and Levels Used in Plasma Pretreatment

Level	Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)
I	50	0.1	2
II	10	0.2	4
III	2	0.3	6

TABLE II
Combinations of the Three Parameters for Plasma Pretreatment Used in the Experiment

Test run	Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)
1	50	0.1	2
2	50	0.2	4
3	50	0.3	6
4	10	0.1	4
5	10	0.2	6
6	10	0.3	2
7	2	0.1	6
8	2	0.2	2
9	2	0.3	4

BTCA two-bath pad-dry-cure treatment

After plasma pretreatment, cotton fabric samples were treated with three different compositions of crosslinking agent: (i) 5% BTCA, (ii) 5% BTCA and 0.1% TiO_2, and (iii) 5% BTCA and 0.2% TiO_2. In this study, TiO_2 was used for replacing sodium hypophosphite (SHP) as the catalyst for BTCA treatment because TiO_2 can achieve better wrinkle resistance effect on cotton fabric.[4,8,9] A two-bath method was used for BTCA treatments. In the first bath, the fabrics were padded with BTCA until wet pick up of 80% was achieved at 25°C. The fabrics were then dried at 85°C for 5 min. In the second bath, the padding processes were performed, by using TiO_2 solution dispersed in 10% Matexil DN-VL (dispersing agent). Subsequently, padded fabrics were dried at 85°C for 5 min and were then cured at 170°C for 2 min. Finally, the fabrics were conditioned at (21 ± 1)°C and (65 ± 5)% RH for 24 h, prior to any further treatment.

Wrinkle resistant property

The wrinkle resistant property of cotton fabric was determined by measuring its WRA, in accordance with the AATCC Test Method 66-2003. WRA of cotton fabric was measured in both warp and weft directions, i.e., the higher the WRA value, the better the wrinkle recovery property will be. In each direction, six fabric specimens were tested, and the average WRA was calculated based on the 12 readings, within a tolerance of 5%.

Scanning electron microscopy

Surface morphology of cotton fibers was examined by the JEOL JSM-6490 scanning electron microscope (SEM), Tokyo, Japan, with an accelerating voltage of 20 kV and a current of 10 μA at a high magnification power of up to ×2000.

RESULTS AND DISCUSSION

Effects of plasma pretreatment parameters on wrinkle resistant property of cotton fabric

Tables III–V show WRAs of plasma pretreated cotton fabrics after treatment with 5% BTCA, 5% BTCA and 0.1% TiO_2, and 5% BTCA and 0.2% TiO_2. WRAs were analyzed with the OATS technique. The higher the WRA value, the better the wrinkle recovery property will be. Therefore, plasma pretreatment conditions that result in the highest WRA values represent the optimal conditions for plasma pretreatment, whereas the greatest difference between WRA values produced, in terms of individual pretreatment conditions, is the dominating factor.[15] Results of experiments, measured in terms of WRA values, are shown in Tables III–V, and Table VI summarizes the optimum conditions for plasma pretreatment preparatory to different BTCA treatments.

The WRA of untreated cotton fabric was 67.8°; different BTCA treatments (after plasma pretreatment) resulted in significant improvement in WRA. Previous studies have proved that BTCA molecules were able to crosslink the hydroxyl groups of cellulosic macromolecules effectively, contributing to the increase of WRA.[8,9] In general, the structural units of cellulose are oriented into three different regions, namely crystalline region, amorphous region as well as intermediate region between them, which the chemical reactivity of cellulose is strongly influenced by the hydroxyl groups in between.[20] To minimize the wrinkle formation conditions, the chain displacement is limited by replacing the weak interaction forces between cellulose chains with stronger bonding that will link them together that give a smooth appearance. In the industry, easy-care properties are commonly imparted to cellulosic fabric by increasing the elastic recovery of the fiber through the formation of crosslinks between adjacent cellulose chains.[20,21]

Without plasma pretreatment, WRA of 5% BTCA treated fabric was 86.1° but it increased by about 3.14–3.95% further after addition of TiO_2. The presence of TiO_2 particles enhances the crosslinking effect between BTCA and the cellulosic molecular chain and also restricts molecular movement of cellulose. It was found that 0.1% TiO_2 added in the BTCA treatment could slightly improve the WRA. As shown in Table VI, addition of 0.2% TiO_2 did give the overall

TABLE III
WRA Values Obtained with Plasma Pretreatment Under Different Conditions, Followed by 5% BTCA Treatment

Test run	Parameters			WRA (°)
	Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)	
1	50	0.1	2	92.8
2	50	0.2	4	87.2
3	50	0.3	6	89.5
4	10	0.1	4	90.3
5	10	0.2	6	86.5
6	10	0.3	2	94.6
7	2	0.1	6	93.0
8	2	0.2	2	91.3
9	2	0.3	4	89.4

∑WRA (°)	Parameters		
	Treatment speed	Oxygen flow rate	Jet-to-substrate distance
∑I	269.5	**276.1**	**278.7**
∑II	271.4	265.0	266.9
∑III	**273.7**	273.5	269.0
Difference[a]	*4.2*	*11.1*	*9.7*

[a] Dominating factors are in the order: Treatment speed > Oxygen flow rate > Jet-to-substrate distance.

Figures in **bold** exhibit the highest values among values of levels of different factors used, while figures in ***Italics*** show the level of importance of each factor.

TABLE IV
WRA Values Obtained with Plasma Pretreatment Under Different Conditions, Followed by 5% BTCA + 0.1% TiO_2 Treatment

Test run	Parameters			WRA (°)
	Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)	
1	50	0.1	2	95.7
2	50	0.2	4	96.1
3	50	0.3	6	97.5
4	10	0.1	4	91.8
5	10	0.2	6	89.0
6	10	0.3	2	91.7
7	2	0.1	6	100.0
8	2	0.2	2	99.6
9	2	0.3	4	97.8

∑ WRA (°)	Parameters		
	Treatment speed	Oxygen flow rate	Jet-to-substrate distance
∑I	289.3	**287.5**	**287**
∑II	272.5	284.7	285.7
∑III	**297.4**	287.0	286.5
Difference[a]	*24.9*	*2.8*	*0.5*

[a] Dominating factors are in the order: Treatment speed > Oxygen flow rate > Jet-to-substrate distance.

Figures in **bold** exhibit the highest values among values of levels of different factors used, while figures in ***Italics*** show the level of importance of each factor.

TABLE V
WRA Values Obtained with Plasma Pretreatment Under Different Conditions, Followed by 5% BTCA + 0.2% TiO_2 Treatment

Test run	Parameters: Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)	WRA (°)
1	50	0.1	2	98.9
2	50	0.2	4	93.1
3	50	0.3	6	97.1
4	10	0.1	4	105.6
5	10	0.2	6	101.8
6	10	0.3	2	104.7
7	2	0.1	6	103.8
8	2	0.2	2	104.2
9	2	0.3	4	105.4

∑ WRA (°)	Parameters: Treatment speed	Oxygen flow rate	Jet-to-substrate distance
∑I	289.1	**308.3**	**307.8**
∑II	312.1	299.1	304.1
∑III	**313.4**	307.2	302.7
Difference[a]	*24.3*	*9.2*	*5.1*

[a] Dominating factors are in the order: Treatment speed > Oxygen flow rate > Jet-to-substrate distance.

Figures in **bold** exhibit the highest values among values of levels of different factors used, while figures in ***Italics*** show the level of importance of each factor.

best results, meaning that higher concentration of TiO_2 could contribute to better wrinkle recovery ability. On the other hand, with plasma pretreatment, WRA of cotton fabrics increased significantly. It was also found that WRA of cotton fabrics was further enhanced by the subsequent BTCA treatment in the presence of TiO_2. This phenomenon could be explained by the etching effect on the fabric surface, obtained after plasma pretreatment; it provides a new pathway for the finishing agent to enter into the fibers, resulting in increased WRA. In addition, the increase in wettability of cotton fibers, caused by oxygen plasma pretreatment, might enhance the effect of the BTCA treatment due to introduction of hydrophilic functional groups.[22,23] The etched fabric surface also allows better attachment of TiO_2 particles which implies a higher catalytic effect on BTCA and the cellulosic molecular chain which consequently restricts molecular movement of cellulose. Moreover, results shown in Table VI demonstrate clearly that different BTCA treatments on cotton fabric, after plasma pretreatment under optimum conditions, result in the highest WRA values, compared with those shown in Tables III–V. Therefore, it can be concluded that plasma pretreatment could be an effective pretreatment for enhancing wrinkle resistant property of cotton fabric treated with BTCA, with or without addition of TiO_2.

Effect of speed of the plasma pretreatment

SEM images of untreated cotton fibers and cotton fibers after plasma pretreatment at different treatment speeds, while maintaining oxygen flow rate at 0.1 L/min and jet-to-substrate distance of 2 mm, are shown in Figures 2 and 3(a–c). SEM images indicate more topographical changed to the surface of the fibers and therefore more effective surface modification and etching effect of plasma treatment at slower speed (longer treatment time). Therefore, longer duration of plasma pretreatment modifies the material surface severely. However, careful control of treatment time and/or treatment speed is important to avoid damage to the fibers. Movement speed of substrate, i.e., treatment speed, is directly proportional to the treatment time, i.e., the faster is the treatment speed, the shorter is the treatment time for the substrate. When the treatment speed is too slow, high concentration of active species generated from the plasma jet would accumulate on the fabric surface. The active species carry high energy, causing a severe sputtering or etching effect that alters the material surface or internal characteristics (penetration of plasma into the fabric[10]). On the other hand, when the treatment speed is too fast, there is insufficient time for the substrate to be reacted with the active species in plasma gas.

The effectiveness of plasma pretreatment is highly dependent on the treatment speed, which is proved by the OATS analysis. According to the results, plasma pretreatment speed is the dominating factor when plasma pretreated fabric is subjected to BTCA treatment in the presence of 0.1% or 0.2% TiO_2. Table

TABLE VI
Optimized Plasma Pretreatment Conditions for Different BTCA Treatments

BTCA treatment system	Treatment speed (mm/s)	Oxygen flow rate (L/min)	Jet-to-substrate distance (mm)	Dominating Factor	WRA of plasma untreated fabric (°)	WRA of plasma pre-treated fabric (°)
5% BTCA	2	0.1	2	Oxygen flow rate	86.1	95.3
5% BTCA + 0.1% TiO_2	2	0.1	2	Treatment speed	88.8	111.5
5% BTCA + 0.2% TiO_2	2	0.1	2	Treatment speed	89.5	114.3

WRA for control cotton fabric = 67.8°.

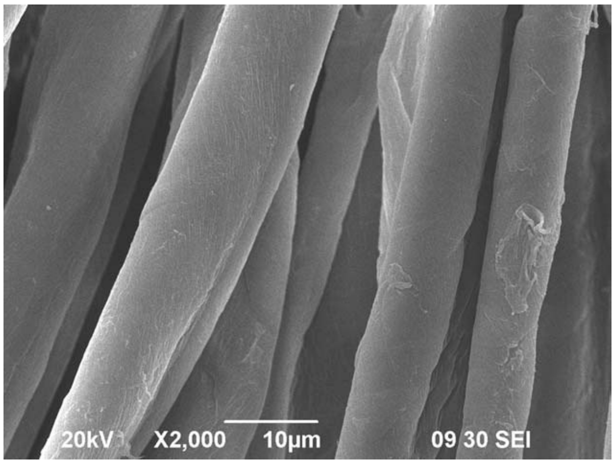

Figure 2 SEM of untreated cotton specimens.

VI shows that of the conditions examined, a speed of 2 mm/s plasma treatment, offers the best balance between enhancement of WRA and minimization of fiber damage. In general, a large number of active species or particles will be generated during plasma pretreatment, such as electrons, ions, free radicals, photons, excited atoms, or molecules. High-speed electrons are the most contributing species that react with the substrate. Once the concentration of the active species increases to a critical level, 2 mm/s speed of plasma pretreatment allows them to diffuse through the fabric, before being neutralized. It was evident that such treatment speed provided enough time for the substrate to be impacted by the concentrated active species produced in plasma gas.[10,24,25]

Effect of oxygen flow rate in plasma pretreatment

In this study, oxygen is the reactive gas carried by helium gas, in plasma pretreatment. Figure 4(a,b) show SEM images of cotton fibers pretreated with plasma, at different oxygen flow rates, at a constant speed of 2 mm/s and constant jet-to-substrate distance of 2 mm. High oxygen flow rate implies high concentration of active species in the plasma jet, producing a severe etching effect that alters the material's surface characteristics. SEM images presented in Figures 3(c) and 4(a,b) show that the etching effect increased with increase in oxygen flow rate. Therefore, increase in flow rate enhanced the surface modification of cotton fibers, until it reached the peak. The effectiveness of the treatment drops when the flow rate is too high, due to diminished production of active species. The oxygen flow rate is related to concentration of oxygen used in plasma pretreatment. Generally speaking, the higher the oxygen flow rate, the higher the concentration of oxygen. Surprisingly, a flow rate of 0.1 L/min produced the best result in the optimum conditions determined. When concentration of O_2 increased during the plasma pretreatment, the supply of active species also increased. However, if a large amount of oxygen is supplied continuously, the active species might react with the oxygen also, besides the cotton surface. As a result, the amount of active species would be decreased and hence the surface reaction will be lowered. When plasma pretreated fabrics were subjected to 5% BTCA treatment, oxygen flow rate was the critical factor affecting the effectiveness of

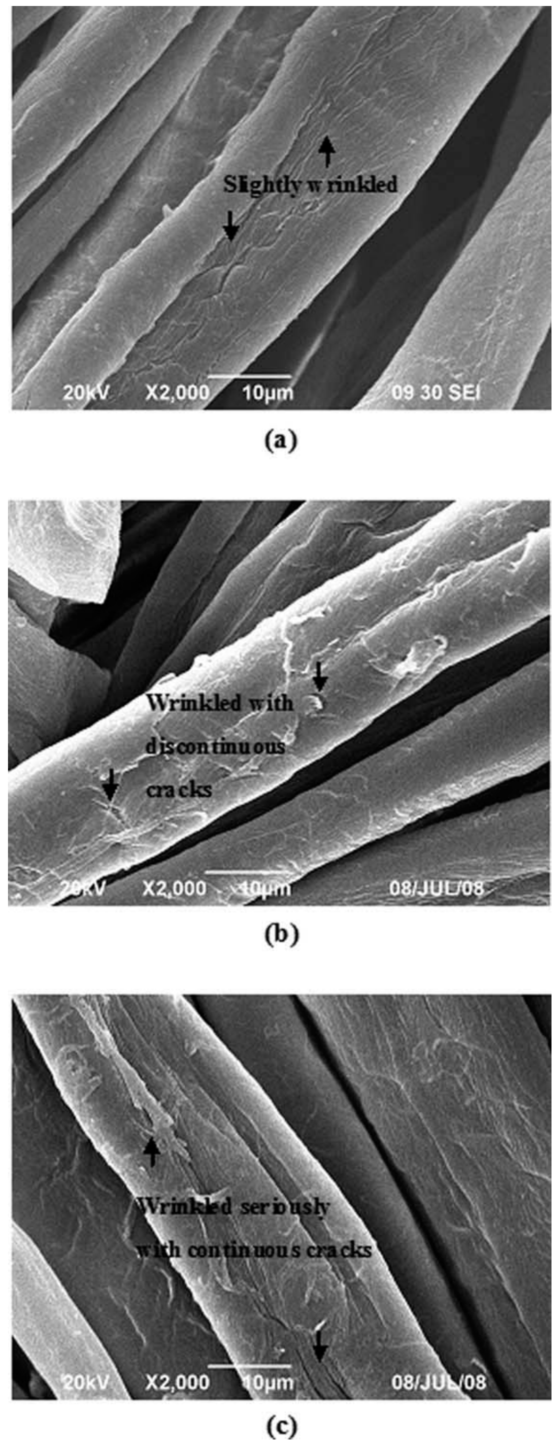

Figure 3 SEM of cotton specimens with plasma pretreatment at speeds of (a) 50 mm/s, (b) 10 mm/s, and (c) 2 mm/s.

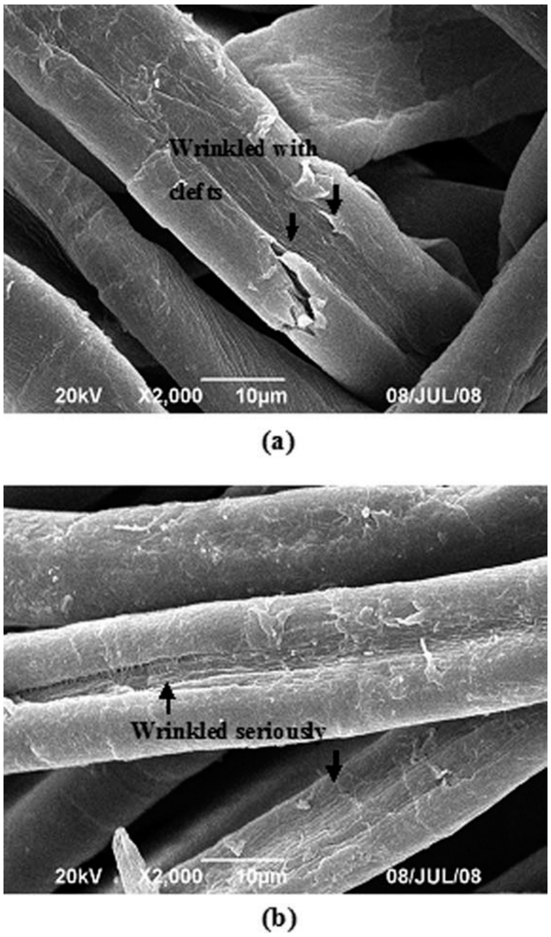

Figure 4 SEM of cotton specimens pretreated with plasma at oxygen flow rates of (a) 0.2 L/min and (b) 0.3 L/min.

BTCA treatment in generating better WRA. The difference between the highest and the lowest WRA generated at different oxygen flow rates was 11.1°. When the fabric is to be treated with BTCA in the presence or absence of TiO_2, after plasma treatment, it requires a lower oxygen flow rate (0.1 L/min) to modify its surface. In general, the catalytic effect enhanced when the surface area of catalyst increases. The surface of cellulosic fabrics is roughened by plasma gas, and TiO_2 particles fill up the etched surface,[8] providing more surface area for enhancing the catalytic effect between BTCA and the cellulose molecular chain. More crosslinkages are then formed between BTCA and cellulose, restricting the molecular movement of cellulose. In this study, 0.1 L/min oxygen flow rate produced the best plasma pretreatment in terms of the final WRA achieved.

Effect of jet-to-substrate distance in plasma pretreatment

Figure 3(c) shows SEM images of cotton fibers after plasma pretreatment with 2 mm jet-to-substrate distance, 0.1 L/min oxygen flow rate, and 2 mm/s speed, while Figure 5(a,b) presents SEM images of cotton fibers after plasma pretreatment with oxygen flow rate of 0.1 L/min, at 2 mm/s speed, but with different jet-to-substrate distances. When Figure 3(c) is compared with Figure 5(a,b), the etched effect is more obvious for cotton fibers plasma pretreated with 2 mm jet-to-substrate distance.

The active species were blown from a jet nozzle by helium flow reaching the fabric surface. The plasma jet was positioned perpendicular to the fabric, such that the surface was close to the point of emission of plasma gas. Generally speaking, when the distance between the plasma jet nozzle and the substrate surface is too close, flow of plasma gas from the nozzle is almost blocked by the fabric, and the gas can only be bounced off the surface; it then flows parallel to the fabric surface, which greatly reduces the effectiveness of the treatment.[10] Moreover, when the distance between the plasma jet nozzle and the substrate surface is too large, plasma gas from the nozzle is unable to hit the fabric surface, which means no surface modification is achieved. Plasma pretreatment of fabric with 2 mm jet-to-substrate distance constitutes the optimum condition for

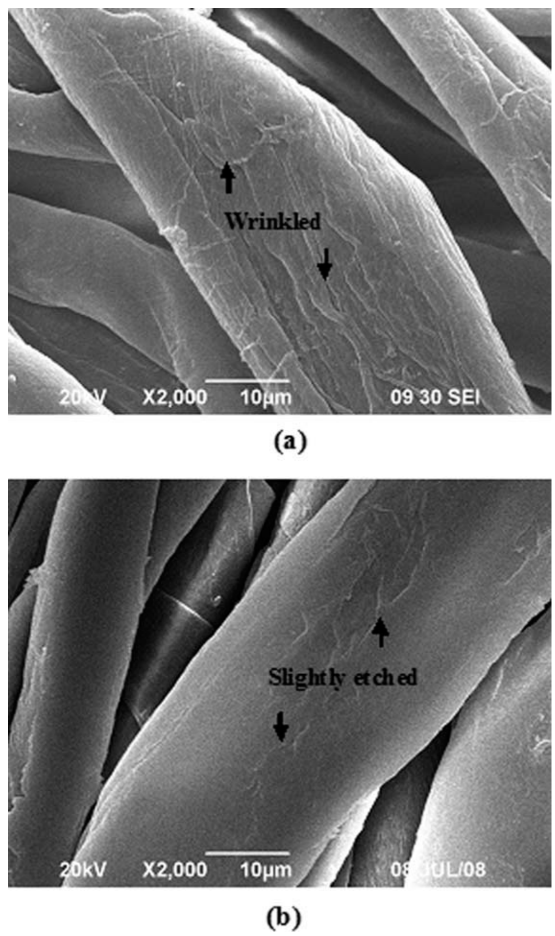

Figure 5 SEM of cotton specimens plasma pretreated with 4 mm and 6 mm jet-to-substrate distance.

the subsequent 5% BTCA treatment, with or without TiO_2, as proved by the OATS tests. However, OATS experiments reveal that the jet-to-substrate distance in plasma pretreatment was the less important parameter affecting the WRA.

Dominating factor

In this study, three plasma pretreatment parameters were used and their effects on wrinkle resistant property of cotton fiber treated with different combinations of BTCA and TiO_2 were studied through the OATS technique. With the use of OATS technique, we can not only determine the optimum condition for a specific process but also determine the process parameter having the dominating effect on the outcome. From the experimental results, it was noted that the optimum condition for applying plasma pretreatment for obtaining wrinkle property in cotton fiber treated with different combinations of BTCA and TiO_2 was (i) treatment speed = 2 mm/s; (ii) oxygen flow rate = 0.1 L/min; and (iii) jet-to-substrate distance = 2 mm. Although the same optimum conditions were obtained for different combinations of BTCA and TiO_2, the dominating factor were different. According to Table VI, the dominating factor for BTCA treatment without TiO_2 was "oxygen flow rate" but the dominating factor for BTCA treatment with TiO_2 was "treatment speed." This deviation may have occurred because of effect of interaction between BTCA and TiO_2 on the plasma pretreated cotton surface.

Without the use of TiO_2, i.e., the catalyst, BTCA can itself provide a certain degree of wrinkle resistant effect on cotton (Table VI) but plasma pretreatment can activate the cotton surface to provide a certain degree of catalytic effect on BTCA to form higher crosslinkages with the cellulose molecules; WRA changed from 86.1° to 95.3° for plasma untreated and plasma pretreated cotton fabrics, respectively. Therefore, the oxygen flow rate played an important role in activation of cotton surface for enhancing catalytic effect of TiO_2 on crosslinkages between BTCA and the cellulose molecular chain. With the use of TiO_2, the treatment speed played an important role in allowing sufficient time for the active species in the plasma to penetrate into the cotton fiber and hence provided more active surface area for the TiO_2 to fill. As a result, the catalytic reaction triggered by TiO_2 could be enhanced, resulting in improved WRA results.

CONCLUSIONS

In this study, it was found that more effective modification of fabric surface and higher etching effect of plasma pretreatment can be achieved with slower speed, lower oxygen flow rate, and lower jet-to-substrate distance, i.e., as investigated with OATS, 2 mm/s treatment speed, 0.1 L/min oxygen flow rate, and 2 mm jet-to-substrate distance were the most effective method for improving wrinkle-resistant property of fabrics treated with BTCA, with or without TiO_2 acting as catalyst. It is because these plasma pretreatment conditions allow the substrate to be impacted by the concentrated active species produced in plasma gas efficiently and therefore, the material surface will be modified effectively and offers the best balance between enhancement of WRA and minimization of fiber damage. In plasma pretreatment, oxygen flow rate and treatment speed were the dominating factors with respect to treatment with BTCA and BTCA-TiO_2.

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