Using Nano-TiO_2 as Co-catalyst for Improving Wrinkle-resistant of Cotton Fabric

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Abstract

Cotton contains cellulose molecules arranged linearly and passing through the crystalline and amorphous regions of the fibres. The cellulose molecules are held in position by hydrogen bonds between themselves. When a force of sufficient magnitude is applied onto the fibres, slippage occurs between the cellulose chains. The hydrogen bonds present between the cellulose molecules tend to resist or prevent the slippage. However, when slippage occurs, the hydrogen bonds reform at new locations and tend to maintain the fibre in a bent or wrinkle state. On the other hand, the absorption of water facilities the breaking of the hydrogen bonds and thus cotton wrinkles easily after laundering.

In order to prevent cotton from wrinkling, hydroxyl groups in the cellulose chain of cotton are partially crosslinked to keep the chain fixed relatively to each other. The popular crosslinking agent recently in use is the dimethylol dihydroxy ethylene urea (DMDHEU). However, DMDHEU suffers a disadvantage that its reaction product on the fabric tends to decompose and release formaldehyde which cause harm to human health. Recently, 1,2,3,4-butane tetracarboxylic acid (BTCA) has been explored as a new wrinkle-resistant agent providing similar performance to that of DMDHEU. In BTCA finishing, catalyst of inorganic phosphorus containing acids such as sodium hypophosphite was used. However, such phosphorus compounds have a highly adverse impact on the environment such as causing an increase in growth of algae in rivers and lakes, thus lowering the oxygen content in water. In this paper, nano-TiO_2 was used as a co-catalyst with the sodium hypophophite in the treatment of cotton with BTCA. The experimental results revealed that the wrinkle recovery of the cotton fabric was improved and the effectiveness of sodium hypophophite, with reduced amount, as a catalyst was improved by the addition of nano-TiO_2.

Keywords: Nanotechnology, wrinkle-resistant, nano titanium dioxide, co-catalyst

1. Introduction

Cotton is composed of cellulose which is a kind of polysaccharide. The cellulose molecules are arranged linearly and pass through the crystalline and amorphous regions of the cotton fibres. The cellulose molecules are held in position by hydrogen bonds between themselves. When a force of sufficient magnitude is applied onto the fibres, slippage occurs between the cellulose chains or between larger structural units of the fibre. The hydrogen bonds present between the cellulose molecules tend to resist or prevent the slippage. However, when slippage occurs, the hydrogen bonds reform at new locations and tend to maintain the fibre in the bent or wrinkle state. In addition, cotton fibre is hydrophilic and can absorb water easily. The absorption of water breaks the hydrogen bonds and allows the fibre or fabric to shrink. As a result, 100% cotton wrinkles easily and has the potential to shrink after laundering.

Cellulose is made up of repeating anhydroglucose units. Each anhydroglucose unit contains two secondary and one primary alcohol groups. In order to resist wrinkles, alcohol groups on the adjacent cellulose chains are partially crosslinked to keep the chains fixed relative to each other. The chemical principle of the wrinkle resistance and durable press treatments in the cellulose-containing textiles is related to the crosslinking of cellulose molecules, generally by the reaction of di- or poly-functional agent with the cellulose. With regard to these two treatments, the most popular agents recently in use are the methylol amide compounds formed by the reaction of formaldehyde with polyfunctional organic amides.

Dimethylol ethylene urea, dimethylol ethyltriazone, trimethylol melamine and methylol ureas are some of the examples. The other examples of crosslinking agents used in the wrinkle-resistant treatment include isocyanates, epoxides, divinylsulphones, aldehydes, chlorohydrins, N-methylol compounds and polycarboxylic acids. Of these crosslinking agents, N-methylol compounds are the most commonly used. Examples of these compounds include dimethylol urea, dimethylol ethylene urea, trimethylol trazine, dimethylol methyl carbamate, uron, triazone and dimethylol dihydroxy ethylene urea. Dimethylol dihydroxy ethylene urea (DMDHEU) is the most commonly used durable-press finish agent today. These methyolamide crosslinkiing agents are quite effective in imparting wrinkle resistance. However, they suffer from a number of disadvantages. One of the disadvantages is that the agent and its reaction product on the fabric tend to decompose and release formaldehyde. Formaldehyde is very irritating and even small amounts are undesirable. In addition, a small amount of free formaldehyde may also be a hazard.

Hitherto, numerous researches have studied the application of carboxylic acids, especially 1,2,3,4-butane tetracarboxylic acid (BTCA), as the new crosslinking agents for wrinkle-resistant treatment. The compound BTCA was a desirable reactant when catalysed with certain phosphorous compounds, providing the performance is similar to DMDHEU but at somewhat higher cost [1,2]. Researchers also studied the non-formaldehyde crease-resist finishing of cotton fabrics by using carboxylic acid as cross-linking agent with the compound catalyst of nanometre grade titanium dioxide (TiO_2), Ag, $MgCl_2$, SiO_2 and ZrO_2 [3].

In the traditional textile industry, BTCA has been explored to be a new wrinkle-resistant agent while the nano TiO_2 has commonly been used as a finishing agent for UV protection treatment. However, very little research has been conducted to study the wrinkle-resistant finishing of cotton fabrics using BTCA crosslinking agent combined with sodium dihydrogen hypophosphite ($NaH_2PO_4\cdot 6H_2O$) catalyst and nano TiO_2 co-catalyst. The aim of this paper is to investigate the effect of crosslinking treatment on the wrinkle recovery, tensile strength, tearing strength and bending length performance of the treated cotton fabrics.

2. Experimental

2.1. *Material*

100% combed cotton 128 x 60/20 x 16 Z-twill fabrics with fabric weight of 250g/m^2 and size of 20 x 20cm^2 were used. The fabric specimens were semi-bleached, washed and dried before crosslinking treatment.

2.2. *BTCA Treatment Finishing Agents*

Two-bath method was adopted for treating the cotton fabric specimens. BTCA and nano TiO_2 solutions were prepared separately for the crosslinking treatment. In order to study the effect of nano TiO_2 and BTCA solution on the performance of the treated fabrics, different concentrations of nano TiO_2 and BTCA solutions were prepared according to the conditions tested in Table 1.

Table 1 Different concentrations of nano TiO_2 and BTCA solutions used for the crosslinking treatment

Amount of nano TiO_2 added	10g of BTCA added	15g of BTCA added
0g	Sample A	Sample B
0.05g	Sample C	Sample D
0.1g	Sample E	Sample F

•10g $NaH_2PO_4\cdot 6H_2O$ catalyst was added to the BTCA solution

The BTCA solution was prepared by adding different amounts of BTCA, i.e. either 10 or 15g BTCA, together with 10g $NaH_2PO_4\cdot 6H_2O$ catalyst into 100ml water. Both the BTCA and $NaH_2PO_4\cdot 6H_2O$ chemicals were supplied by the Advanced Technology and Industrial Co., Ltd., with the purity of 98%. The nano TiO_2 solution was prepared by adding different amounts of nano TiO_2, i.e. either nil, 0.05 or 0.1g nano TiO_2, into 100ml non-ionic dispersing agent Matexil DN-VL manufactured by Uniqema. The nano TiO_2 was manufactured by Advanced Technology and Industrial Co., Ltd. with the purity of 99.5%.

2.3. *BTCA Two-Bath Pad-Dry Cure Treatment*

For each condition, the treated fabric was prepared by two-bath method. In the first bath, the specimen was dipped and padded with 100ml BTCA solution twice until the wet pick up percentage of 75% was reached at room temperature. In the second bath, the dipping and padding processes were performed again by using 100ml nano TiO_2 solution. The padded fabrics were then exposed to a 0.378mW/cm^2 intensity of UV light source for 1 hour followed by curing in an oven at 170℃ for 3 minutes. Finally, the fabrics were conditioned before testing.

2.4. *Traditional Resin Treatment*

In order to evaluate the performance of the BTCA treated fabrics, the conventional resin treated sample (Sample G) was prepared for comparison. The resin used for comparison was a modified dimethyloldihydroxyethylene urea manufactured by BASF. It is used as a pre-catalysed crosslinking agent for extremely low-formaldehyde easy-care finishing of textiles composed of cellulosic fibres or their blends with synthetic fibres. Sample G was prepared by padding it with a 100ml traditional resin solution containing 70g/L resin and 13g/L magnesium chloride until the wet pick up was equal to 70%. The padded sample was then dry at 100℃ for 5 minutes followed by curing at 150℃ for 3.5 minutes. Finally, Sample G was conditioned before testing.

2.5. *Wrinkle Recovery*

The wrinkle recovery of the treated fabrics was evaluated according to the AATCC Test Method 66-1998.

2.6. *Tensile Strength*

The tensile strength of the treated cotton fabrics was evaluated according to the BS EN ISO 13934-1:1999 using an Instron Tensile Tester.

2.7. *Tearing Strength*

The tearing strength of the treated cotton fabrics was tested according to the ASTM D1424-96 Standard Test Method for Tearing Strength of Fabrics by Falling-Pendulum Type (Elemendorf) Apparatus.

2.8. *Bending length*

The bending length of the treated cotton fabrics was measured according to the ASTM D1388-2002 Standard Test Method for Stiffness of Fabrics.

3. Results and Discussion

3.1. *Wrinkle Recovery*

The reaction between the polycarboxylic acid and cellulose hydroxyl group is composed of a two-stages reaction. The first stage is the formation of a five member cyclic anhydride intermediate, while the second stage is the formation of the ester bond between the cyclic anhydride ring and the hydroxyl group on the cellulose. The mechanism of this is illustrated in Fig. 1.

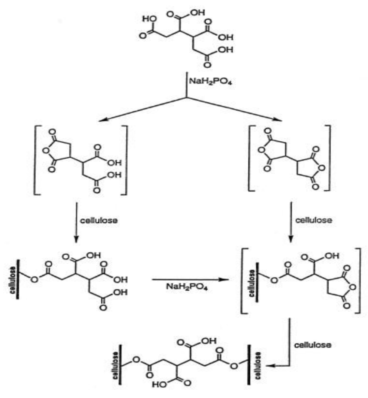

Fig. 1. Mechanism of BTCA reaction

During the curing process, the polycarboxylic 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. Since the anhydrides are reactive and able to esterify cotton cellulose without a catalyst, thus the chief role of catalyst is to accelerate the formation of anhydrides from polycarboxylic acids [4]. A lot of salts of phosphorus-containing acids are examined as catalysts for the finishing of cotton with polycarboxylic acids. The use of sodium hypophosphite as catalyst, however, has several disadvantages. The high cost of chemical and its tendency to cause shade changes in the dyed fabrics are the associated problems. Moreover, the use of phosphorus compounds in textile finishing raises the environmental concern. When waste solutions of these agents are discharged into streams and lakes, the phosphorous compounds are likely to serve as nutrients promoting the growth of algae [1]. In order to diminish the disadvantages of using phosphorous compound as catalyst, some research works were conducted to investigate the probability of decreasing the amount of phosphorous compound by using nano TiO_2 as the co-catalyst.

The results of wrinkle recovery of the BTCA treated cotton fabrics are shown in Table 2. After treating the cotton fabric with BTCA without the use of nano TiO_2, it was clearly observed that the wrinkle recovery of the BTCA treated cotton fabric was significantly increased. The increment was proportional to the increased amount of BTCA used in the treatment bath. However, the wrinkle recovery obtained was still worse than the traditional resin treatment.

Table 2 Wrinkle recovery of BTCA and resin treated fabrics

	Wrinkle Recovery Angle (˚)		
Sample	Warp	Weft	Warp Plus Weft
Control	79	90	169
A- 0g nano TiO_2 with 10g BTCA[a]	85(↑7.6%)[b]	116(↑28.9%)[b]	201 (↑18.93%)[b]
B - 0g nano TiO_2 with 15g BTCA[a]	100(↑26.6%)[b]	116(↑28.9%)[b]	216 (↑27.81%)[b]
C - 0.05g nano TiO_2 with 10g BTCA[a]	118(↑49.4%)[b]	126(↑40.0%)[b]	244 (↑44.38%)[b]
D - 0.05g nano TiO_2 with 15g BTCA[a]	112(↑41.8%)[b]	129(↑43.3%)[b]	241 (↑42.60%)[b]
E - 0.1g nano TiO_2 with 10g BTCA[a]	96(↑21.5%)[b]	115(↑27.8%)[b]	211 (↑24.85%)[b]
F - 0.1g nano TiO_2 with 15g BTCA[a]	122(↑54.4%)[b]	130(↑44.4%)[b]	252 (↑49.11%)[b]
G - Traditional Resin	106(↑34.2%)[b]	114(↑26.7%)[b]	220 (↑30.18%)[b]

[a] For Samples A to F, 10g NaH_2PO_4 was used during the crosslinking treatment.
[b] The symbol "↑%" represents the percentage of enhancement in wrinkle recovery angle after the treatment.

When the nano TiO_2 was used as a co-catalyst in the BTCA treatment, it was obvious that the wrinkle recovery obtained of the treated fabrics was higher than the untreated cotton. Moreover, when compared with the traditional resin treatment, Sample G showed a less significant effect on wrinkle recovery when compared with Samples C, D and F. With regard to the effect of various amounts of nano TiO_2 used on the wrinkle recovery of the fabrics, the amount of BTCA used was fixed to be 10g and 15g respectively and the corresponding two curves were plotted as shown in Fig. 2. When 15g BTCA was used, the increased amount of nano TiO_2 in the treatment bath would further enhance the wrinkle recovery of the cotton fabric whereas different results were noted in the case of 10g BTCA used in the treatment bath. As for the case of 10g BTCA, optimum wrinkle recovery could be achieved when 0.05g nano TiO_2 was added. Further increase in the amount of nano TiO_2 to 0.1g could not enhance the wrinkle recovery. This reduction suggested that the use of nano TiO_2 as a co-catalyst with NaH_2PO_4 could improve the catalytic reaction of the BTCA. When 10g BTCA was used, both the nano TiO_2 and NaH_2PO_4 enhanced the catalytic reaction through the formation of ester bond between the cyclic anhydride ring and the hydroxyl group of cellulose at a faster rate. Once the BTCA was used up, further addition of nano TiO_2 would possibly have no effect on the catalytic reaction. On the contrary, as the particle size of the TiO_2 is in nano-scale, the nano TiO_2 could fill the amorphous region of the cellulose and hence the presence of nano TiO_2 inside the fibre would probably restrict the molecular movement of cellulose. Hence, the reduction of the wrinkle recovery ability of the cotton fabric would possibly be the result. Furthermore, the increase of nano TiO_2 would promote the reversible crosslinking of cellulose molecules and so it might diminish the wrinkle recovery effect.

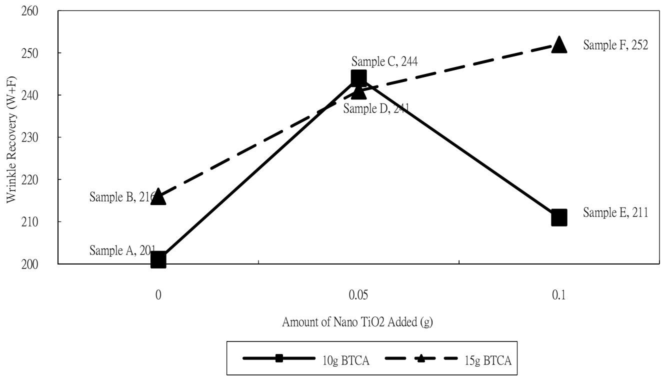

Fig. 2. Wrinkle recovery curves for 10g and 15g BTCA treated cotton fabrics

In the case of 15g BTCA with sufficient amount of BTCA provided for the catalytic reaction, increasing the amount of nano TiO_2 could further enhance the wrinkle recovery of the cotton fabric. As for the case of 10g BTCA, the additional amount of nano TiO_2 would give an adverse effect on the wrinkle recovery performance of the cotton fabric as the amount of BTCA required was insufficient.

As a result, it was concluded that when the nano TiO_2 was used as a co-catalyst of NaH_2PO_4, the catalytic reaction of the BTCA treatment was increased. The amount of BTCA used in the treatment bath would play an important role in determining the final wrinkle recovery of the cotton fabric. If the amount of BTCA was too low, this co-catalyst method would not be so effective to produce a desired wrinkle recovery effect on cotton fabric.

Fig. 3 compares the wrinkle recovery of the treated cotton fabrics with reference to the same amount of BTCA. In the case of 10g BTCA, Sample C had higher wrinkle recovery when compared with Sample E. As for the case of 15g BTCA, the wrinkle recovery performance of Sample F was higher than that of Sample D. This illustrated that at lower concentration of BTCA, smaller amount of nano TiO_2 would give better result but opposite effect was obtained at higher concentration of BTCA. Based on this result, it was suggested that when both the nano TiO_2 and NaH_2PO_4 were used as catalyst for the BTCA treatment, the competitive effect would occur between these two chemicals during the BTCA treatment.

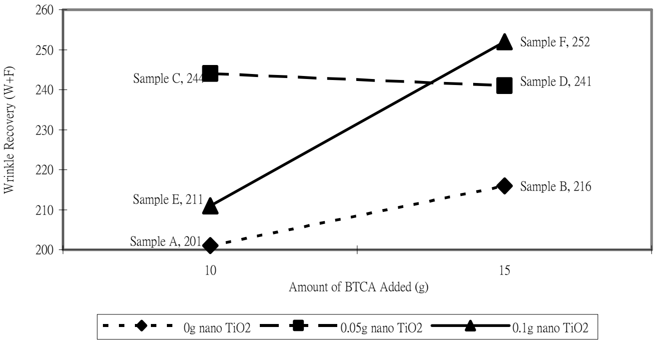

Fig. 3.Comparison of wrinkle recovery of the fabrics treated with different amounts of nano TiO_2 co-catalyst.

The enhancement of wrinkle recovery by the addition of nano TiO_2 in the BTCA treatment is probably due to the unique photocatalytic property of the nano TiO_2 which is a kind of N-type semi-conductor. When TiO_2 is exposed to the ultra-violet radiation whose energy is higher than the band gap of TiO_2 of around 3.2eV, the electrons of TiO_2 will be excited from the valence band to the conduction band, resulting in the formation of two kinds of opposite charged carriers, i.e. hole (h^+) and electron (e^-). The electron (e^-) is changed to superoxide anion ($\bullet O_2^-$) by oxygen, while the formed hole (h^+) is changed to hydroxyl radical (•OH) by the moisture present in the air. These superoxide anion and hydroxyl radical are responsible for the redox reaction. The hydroxyl radical (•OH) has strong oxidising power for oxidising the other materials. On the other hand, the superoxide anion ($\bullet O_2^-$) has strong reducing power to reduce other materials such as the heavy metal ions inside the water [12]. The mechanism of it is illustrated in Fig. 4.

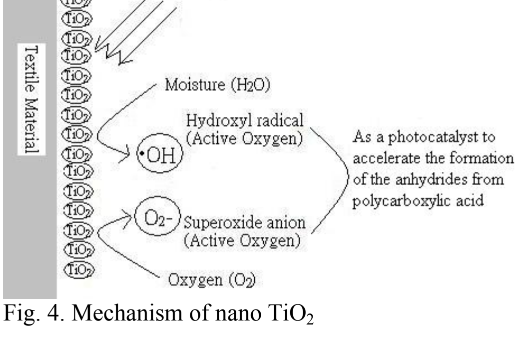

Fig. 4. Mechanism of nano TiO_2

The reaction mentioned is illustrated by the following equations:

TiO_2 (Anatase) + hv → $TiO_2(h^+) + TiO_2(e^-)$

Oxidation reaction: $TiO_2(h^+) + H_2O \rightarrow H^+ + \bullet OH$

$TiO_2(h^+)$ + Reactant → Product

•OH + Reactant → Product

Reduction reaction:

$TiO_2(e^-) + O_2 \rightarrow \bullet O_2^-$

$TiO_2(e^-)$ + Reactant → Product

$\bullet O_2^-$ + Reactant → Product

where heavy metal ions is an example of the reactant. In this paper, the Na^+ metal ion from the catalyst NaH_2PO_4 was used as the reactant for promoting this reaction.

The hydroxyl radical (•OH) and superoxide anion ($\bullet O_2^-$) formed may act as a catalyst to accelerate the formation of anhydrides from polycarboxylic acids. Moreover, the effect of hydroxyl radical (•OH) and superoxide anion ($\bullet O_2^-$) on the increase of the charge localization of the solid cellulose medium in which the esterification and crosslinking take place may also be significant.

When the size of the TiO_2 is reduced to nano-scale, the photocatalytic characteristic of TiO_2 will be greatly enhanced due to the advent of nanotechnology. At nano-scale, not only the surface area of TiO_2 particle increases dramatically but also it exhibits other effects on the optical properties and size quantisation. An increased rate in photocatalytic reaction is observed as the redox potential increases and the size decreases. The overall result is that the presence of nano TiO_2 as co-catalyst may enhance wrinkle recovery performance of the BTCA treated fabrics.

3.2. *Tensile and Tearing Strength*

In the previous research, it was found that loss of fabric mechanical strength was attributed to two main factors, i.e. irreversible acid-catalysed depolymerisation and reversible crosslinking of cellulose molecules [4,5].

The acidity of polycarboxylic acid has a significant effect on the reduction of tensile strength of the treated cotton. The loss in tensile strength of cotton fabric treated with polycarboxylic acid, is a result of the acid-catalysed depolymerisation of cellulose molecules [6]. This phenomenon was coincided with our experimental results. Table 3 shows the comparison of the tensile strength of Samples C and E with Samples D and F. When equal amount of nano TiO_2 was added together with a greater amount of BTCA (15g) in the treatment bath, the loss in tensile strength of Samples D and F would be greater than Samples C and E which were treated with fewer amounts of 10g BTCA. Since the chemical used in the traditional resin treatment (Sample G) was not acidic, the catalysed depolymerisation problem of cellulose molecules did not occur causing no severe reduction in tensile strength. As a result, the tensile strength loss of the treated Sample G was not as severe as those of the BTCA treated fabrics.

When comparing the tensile strength of Samples B, D and F, it was found that the fabrics treated with 15 g BTCA treated fabrics suffered the tensile strength loss with respect to the increasing wrinkle recovery angle and the large amount of nano TiO_2 added. For the fabrics (Samples A, C and E) treated with 10g BTCA, there was an increase in their tensile strength loss with respect to the increasing wrinkle recovery angle following the order of the tensile strength loss and wrinkle recovery of Samples C > E > A.

At elevated temperatures, polycarboxylic acids depolymerise cellulose. Crosslinking of cellulose molecules increases the brittleness of cotton fibres and in turn reduces the strength of the crosslinked fabrics. The magnitude of the tensile strength loss is enhanced with the increase in degree of crosslinking. As a result, for most durable press treatments of cotton, there will be a decrease in the tensile strength of the fabric as the wrinkle recovery angle is increased.

Table 3 also illustrates the comparison of the tearing strength of Samples A and B with Samples C and D. It was found that the tearing strength of Samples C and D treated with 0.05g nano TiO_2 added was smaller than Samples A and B which were treated with BTCA but without the addition of nano TiO_2. The reduction of tearing strength might be due to the result of the increase in crosslinking of cellulose molecules, which in turn reduced the tensile strength of the crosslinked fabrics as mentioned before. However, when the amount of nano TiO_2 added was increased to 0.1g, the tearing strength of the treated Samples E and F were higher than Samples C and D treated with 0.05g nano TiO_2. This enhancement might be due to presence of excess amount of nano TiO_2 within the fibres and yarns. This nano TiO_2 might induce inter fibre and inter yarn friction or make the yarns adhere more to one another which would resist the yarn slippage resulting in the reduction of tearing strength.

3.3. *Bending Length*

Fig. 5 shows that the BTCA and traditional resin treated fabrics generally had smaller bending length meaning that all the treated fabrics were less stiff when compared with that of the control fabric. The BTCA treated fabrics had even smaller bending length when compared with that of the traditional resin treated fabric. The decrease in stiffness of the fabrics might be due to the reduction of cross sectional area of the cotton fibres after the BTCA treatments. When the treated fibres were dried during the curing step, the BTCA molecules present inside the fibres would react with the cellulose surfaces. As the crosslinks introduced between the hydroxyl groups were covalent bonds that held the cellulose molecules together, they would function as a swelling restraint. When the fibre cross-sectional increases and other factors remain constant, the stiffness will increase [7,8]. The swelling restraint induced by the crosslinks caused the reduction of the cross-sectional areas of the cotton fibres, which consequently led to the decrease in stiffness of the fabrics after the BTCA treatment.

Moreover, the addition of nano TiO_2 generally decreased the bending length of the BTCA treated fabrics with the exception of Sample D treated with 15g BTCA and 0.05g nano TiO_2 co-catalyst. This might be due to the presence of nano TiO_2 between the fibres which restricted the swelling of the fibres, thereby reducing the stiffness of the fabrics.

Table 3. Wrinkle Recovery, Tensile and Tearing Strength of the BTCA Treated Samples

	Wrinkle Recovery Angle (˚)(W+F)	Tensile Strength (N) (W+F)	Tearing Strength (N) (W+F)
Control	169	1264.6	7743
A – 0g nano TiO_2 with 10g BTCA	201	761.9	4608
B – 0g nano TiO_2 with 15g BTCA	216	778.2	4416
C – 0.05g nano TiO_2 with 10g BTCA	244	734.2	3648
D – 0.05g nano TiO_2 with 15g BTCA	241	715.1	3776
E – 0.1g nano TiO_2 with 10g BTCA	211	758.6	4544
F – 0.1g nano TiO_2 with 15g BTCA	252	713.9	3904
G - Traditional Resin	220	1025.5	3157

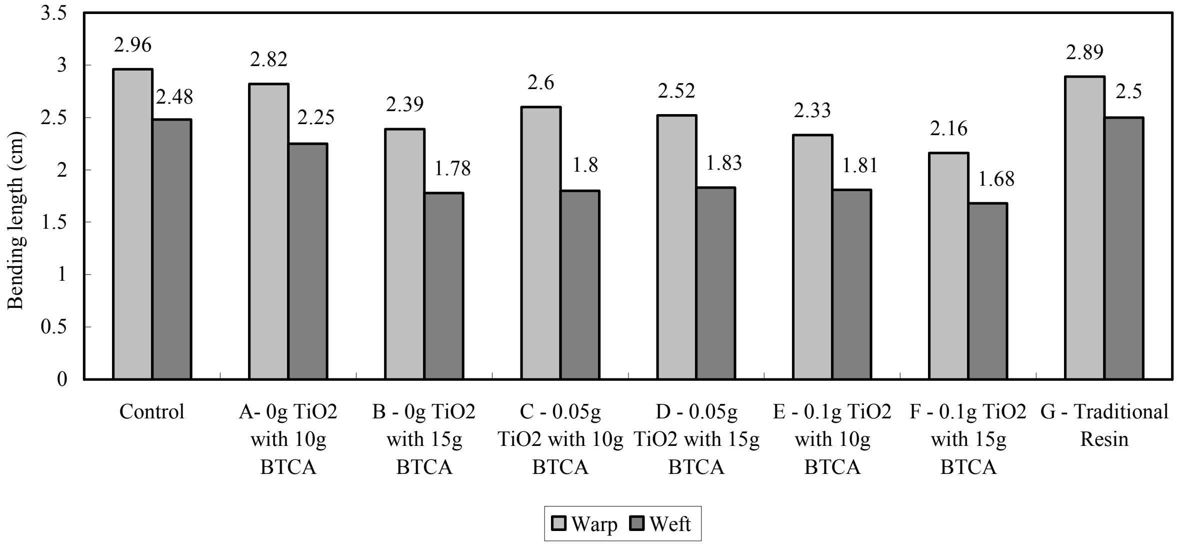

Fig. 5. Comparison of the bending length of BTCA and traditional resin treated fabrics

4. Conclusions

The study proposed a new way to employ nano TiO_2 as a co-catalyst in the BTCA treatment aiming to enhance the wrinkle recovery performance of cotton fabrics. It was found that the addition of nano TiO_2 in the BTCA treatment could further enhanced the wrinkle-resistance of cotton fabrics through its photocatalytic property. The addition of nano TiO_2 in the BTCA treatment also imparted stiffer and softer hand feel to the treated fabric. However, there was a slight decrease in tearing and tensile strength of cotton fibres as a result of the addition of nano TiO_2.

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