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Study of BTCA and Nano-TiO_2 Effect on Wrinkle Force and Recovery of Cotton/Polyester Blended Fabric

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Abstract
In this study the wrinkle force and recovery, elongation, strength, Poisson's ratio and initial modulus of cotton/polyester blended fabric (65/35%) have been investigated. Different samples were considered after being treated by 1,2,3,4-butane tetra carboxylic acid (BTCA) and sodium hypophosphite (SHP) catalyst mixed with nano-titanium dioxide(TiO2) as a co-catalyst by the pad-dry curing method. The wrinkle behaviour of the samples treated was assessed by a wrinkle force tester in addition to an Italian Mesdan tensile tester and wrinkle recovery tester AATCC128, meanwhile their surfaces were evaluated by scanning electron microscopy (SEM). The results show that utilising the treatment substances led to wrinkle recovery improvement; in addition, the wrinkle force, Poisson's ratio and initial modulus were also increased. On the other hand, it is observed that tensile properties in both directions (weft and warp) of the woven fabrics were decreased. Furthermore, a different observation was induced for each direction of the woven fabrics through considering the orthotropic feature, wrinkle force and recovery.

Key words: *1,2,3,4-butane tetra carboxylic acid (BTCA), sodium hypophosphite (SHP), nano-TiO_2, cotton/polyester, wrinkle behaviour.*

Introduction

In recent years many efforts have been made in order to improve cellulose's wrinkle recovery referring to the utilisation of formaldehyde, non-formaldehyde and also nano composites. Although formaldehyde compounds have a strong impact on the wrinkle-resistant, due to the poisonous effect, it has not extensively been applied [1 - 7].

Individual methods could be utilised for finishing fabrics, among which padding is the predominant method that has usually been employed [8, 9]. Recently several polycarboxylic acids have been examined as cross-linking agents for the finishing of cotton fabrics [10 - 13]. Poly-carboxylic acids such as BTCA (1,2,3,4-butane tetra carboxylic acid) are cotton cross-linking agents and can be appropriately used to comprise anhydrides as reactive intermediate captures by hydroxyl groups of cotton fabrics [14 - 16]. The cross-linking of CA and BTCA with cellulose fabrics has been suggested to be catalysed by SHP in an acidic condition [17].

Nano-TiO_2 offers applications as a co-catalyst to improve the finishing implementation and reduce the additional and marginal effects. Moreover as a photocatalyst it may be taken into consideration for the appearance of maleic anhydride as a cross-linking agent to enhance the wrinkle recovery properties of silk fabrics [18]. In some cases nano-TiO_2 performances have been evaluated as a co-catalyst to enhance the wrinkle-resistance of cotton fabrics [19].

Despite these efficient applications, improvement of the wrinkle properties of cotton based commodities using nano-TiO_2 as a co-catalyst has rarely been considered. Concerning cotton fabric wrinkle behaviour, especially when they are blended with polyester or other viscoelastic components, more attention should be drawn to apply the reliable treatments. Due to the difficulties caused by repeated washing and ironing of cotton/polyester fabrics, permanent damage to the fabrics structure might occur. To avoid and minimise the defects, some chemical treatments have to be taken into consideration from an experimental view point. Owing to the fact that washing plays a very important role in finishing process in manufacturing and sometimes might be known as a spoiler of the durability of fabrics, recommended treatments comprising nano particles (e.g. TiO_2) which influence the physical and mechanical properties of woven fabric are likely to be dependable.

Previous researches have predominantly focused on the wrinkle properties of pure cotton woven fabric. In the current study samples were a blend of polyester-cotton woven fabric. There is no work reporting this kind of fabric. One of distinctions of this work is to utilise the tangential force according to the Wrinkle recovery AATCC128 Standard along with developing a new experimental technique already established by (Hezavehi et al) [20 - 22].

In the current study, the constituent of the finishing treatment was based on nano-

Table 1. *General specification of fabric.*

Fabric type	Yarn count, tex		End per cm	Pick per cm	Weight, g/m^2
	warp	weft			
Cotton 35%/Polyester65%	19.66	22.69	34	28	206

Table 2. *Materials consumed for each mode.*

T ype of fabrics	Code	BTCA, %	TiO_2, %	SHP, %
A	BTCA	6	----	6
B	TiO_2	----	2	----
C	TiO_2	----	1	----
D	BTCA → TiO_2	6	1	6
E	BTCA → TiO_2	6	1	6
F	BTCA + TiO_2	6	1	6
G	BTCA + TiO_2	6	2	6
H	Untreated	----	----	----

Azad A, Hezavehi E, Shahvazian M., Zolgharnein P. Study of BTCA and Nano-TiO2 Effect on the Wrinkle Force and Recovery of Cotton/Polyester Blended Fabric.
FIBRES & TEXTILES in Eastern Europe 2012; 20, 5(94): 60-65.

titanium dioxide (TiO_2) as a co-catalyst, which needs to be taken into consideration for modification of cotton/polyester fabrics properties. The modification was fundamentally expected to change the structural parameters of the fabrics, for instance the wrinkle behaviour in the weft and warp direction of the woven fabric. In addition, the mechanical properties as well as the wrinkle behaviour of cotton/polyester plain woven fabrics will be examined and discussed further.

Experimental

Materials and method

In this research a bundle of plain woven fabric was used. ***Table 1*** shows the general fabric specifications.

Chemical materials

The chemical substances applied in this study are 1,2,3,4-butane tetra carboxylic acid (BTCA) supplied by Merck Chemical Company, Germany; Sodium hypophosphite (SHP) – by the Fluka Company, Switzerland and Nano-titanium dioxide (Nano TiO_2), made by the Degussa Chemical Company, Germany.

Materials consumption amounts

The constituents of the materials are summarised as follows:
BTCA and sodium hypophosphite were prepared at a level of 6%, TiO_2 alone and with BTCA 1% and 2%, respectively, according to the sample weights. The order of treatment as well as the percentage of consumption are listed in ***Table 2***.

Due to the different proportions of treatment, 8 kinds of woven fabric were prepared.

As seen in the ***Table 2***, the preparation process for samples D and E was consecutively preformed by chemical compounds already introduced. In summary, sample D was treated first with TiO_2 as one step and then completed by BTCA as the other. An arrow is used to identify the order of the procedure. However, for sample E the process was conducted in reverse order, meaning that the samples were subjected to the BTCA compound and then to TiO_2. The other circumstance was utilised for two samples F and G. The pretreatment of TiO_2 and BTCA was prepared as a mixture to finish the samples in one step.

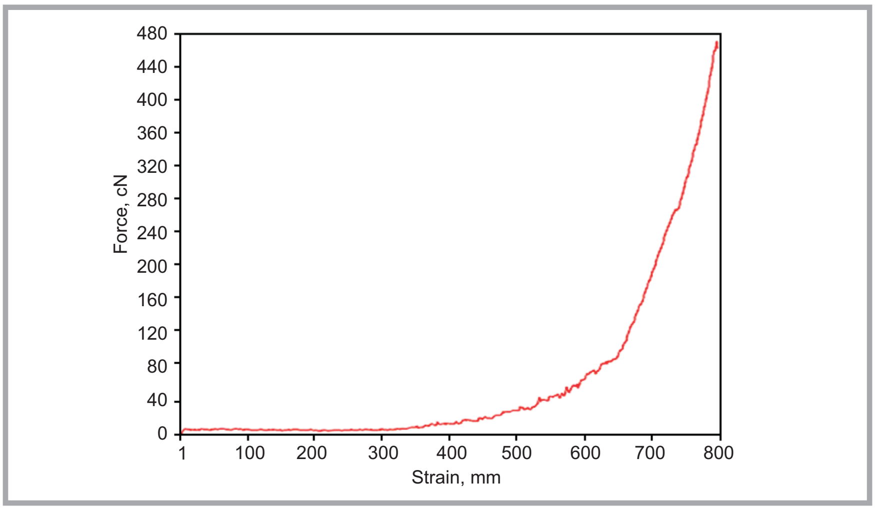

Figure 1. *Typical diagram of wrinkle force for fabric type G in the warp direction (Labview software Ver. 6).*

Method

The mixtures were finished and dispersed using an ultrasonic bath, a thermal oven for drying and the curing process, and a laboratory pad machine with the ability of balancing the roller's pressure. The padded samples were picked up at a rate of 70% and dried at 80 ºC for 5 min following the curing process at 150 ºC for 3 min.

In order to conduct a mechanical investigation, a tensile tester made by the Italian Mesdan Company was employed to characterise the initial modulus, Poisson's ratio as well as the strength of the fabrics along the weft and warp directions. In addition, a wrinkle recovery tester AATCC-128–M272, made by the Shirley Company, was also utilised to evaluate the samples' wrinkle recovery, accompanied by a wrinkle force tester to assess the samples' wrinkle force [20, 21]. A scanning electron microscope (SEM) XL30, made by the Dutch Phillips Company, was used to estimate the fabrics' structural morphology.

Wrinkle recovery test

The wrinkle recovery properties of the specimens were measured by a wrinkle recovery tester AATCC-128 [23], which is shown in ***Table 3***. In compliance with AATCC-128 standard, the test was repeated 9 times for each one of the 8 samples in the weft and warp directions.

Wrinkle force test

A Typical diagram for the wrinkle force against torsional strain is illustrated in ***Figure 1***, which allowed woven fabric wrinkle force evaluation, presented in ***Table 3*** (see page 62) - total effective length: 110 mm, spiral angle: 32.14 degrees, rotational level: 9.1 turn/m [21]. In this case the test was also repeated 9 times in accordance with the relevant standard for each of the 8 samples in the weft and warp directions.

Tensile test

The tensile properties of the specimens including the woven fabric strength, elongation, initial modulus and Poisson's ratio [21] were measured. Numerical results of the fabric tensile test are shown in ***Table 3*** (see page 62). The test was repeated 5 times for each of the 8 samples along the weft and warp directions.

Result and discussion

A summary of the ANOVA test for wrinkle recovery, force, strength, elongation, Poisson's ratio and initial modulus at 95% percent confidence interval of 7 different states along the weft and warp directions is shown in ***Table 4*** (see page 62). The variation of physical and mechanical properties versus woven fabric types are also presented in ***Figures 2*** to ***7***.

As shown in ***Table 4***, the type of woven fabrics significantly affected the wrinkle recovery, wrinkle force, Poisson's ratio, initial modulus, strength and elongation along both weft and warp directions.

The table above shows that there is not any remarkable similarity between the means for each case for the properties,which means that the effectiveness of the chemical treatment can be deduced. To simplify the matter further, the precise

*Table 3. Experimental results of the wrinkle recovery, wrinkle force and tensile properties; * Amounts in parentheses are standard deviations.*

Type of fabric	Wrinkle recovery (Replica)		Wrinkle force, N		Strength, N		Elongation, mm		Initial modules, N/mm		Poisson's ratio	
	warp	weft	warp	weft	warp	weft	warp	weft	warp	weft	warp	weft
A	2.8 (0.44)*	2.8 (0.44)	10.5 (0.96)	2.3 (0.54)	509.5 (12.37)	497.8 (1.40)	29.4 (0.71)	49.1 (1.61)	5.78 (0.55)	1.28 (0.39)	0.77 (0.09)	0.75 (0.19)
B	2.2 (0.44)	2.8 (0.44)	3.5 (0.45)	1.7 (0.40)	527.1 (71.12)	478.4 (70.15)	33.6 (1.74)	44.8 (3.97)	7.05 (0.62)	1.08 (0.11)	0.78 (0.17)	0.76 (0.19)
C	2.2 (0.44)	2.8 (0.44)	3.3 (0.74)	2.0 (0.44)	455.2 (32.80)	505.6 (7.35)	28.8 (0.68)	41.3 (2.97)	5.54 (1.60)	1.58 (0.62)	0.75 (0.10)	0.72 (0.25)
D	2.8 (0.44)	4.0 (0)	3.5 (0.48)	3.4 (0.57)	439.1 (47.62)	439.5 (32.87)	32.9 (4.49)	41.7 (1.43)	2.62 (0.88)	1.88 (0.33)	0.77 (0.29)	0.76 (0.11)
E	2.8 (0.44)	3.0 (0)	8.7 (0.53)	2.5 (0.42)	410.0 (33.27)	469.7 (41.56)	44.5 (6.89)	37.6 (1.66)	3.00 (0.86)	1.66 (0.39)	0.78 (0.10)	0.75 (0.19)
F	2.8 (0.44)	2.8 (0.44)	6.0 (0.24)	1.7 (0.31)	393.6 (39.77)	281.25 (24.21)	37.7 (2.08)	40.3 (2.68)	2.93 (0.37)	1.50 (0.50)	0.78 (0.18)	0.73 (0.25)
G	2.8 (0.44)	3.8 (0.44)	5.6 (0.38)	1.8 (0.30)	357.6 (31.33)	447.7 (62.27)	28.3 (3.67)	40.7 (3.83)	8.22 (0.77)	1.94 (0.53)	0.76 (0.10)	0.75 (0.15)
H	1.2 (0.44)	1.2 (0.44)	4.6 (0.45)	1.8 (0.39)	490.2 (18.83)	490.3 (61.73)	33.1 (0.40)	47.4 (5.09)	6.47 (0.51)	1.27 (0.63)	0.78 (0.35)	0.74 (0.41)

*Table 4. A summary of ANOVA statistical analysis results; *Sig: P-value which presents significant values for amount under 0.05.*

Type of fabric	Wrinkle Recovery		Wrinkle force, N		Strength, N		Elongation, mm		Initial modules, N/mm		Poisson's ratio	
	F	Sig*	F	Sig	F	Sig	F	Sig	F	Sig	F	Sig
Warp direction	6.095	0.019	26.410	0.000	14.755	0.001	3.895	0.04	.433	0.043	4.957	0.033
Weft direction	10.286	0.003	4.460	0.043	11.775	0.002	4.857	0.035	2.332	0.042	17.185	0.000

discrepancy of the properties is illustrated as a bar chart in ***Figures 2*** to *7*.

Effect of fabric type on wrinkle recovery along the warp and weft directions

In ***Figure 2*** the wrinkle recovery properties of the finished samples in the warp and weft directions are plotted.

According to the ***Figure 2***, sample D in the weft direction (BTCA and TiO_2 order) shows the best wrinkle recovery result. In contrast, sample H presents a remarkably lower amount of wrinkle recovery in both the warp and weft directions. Furthermore, the wrinkle recovery properties for samples G (BTCA + TiO_2 2%), F (BTCA + TiO_2 1%), E (TiO_2 and BTCA) and D (BTCA and TiO_2) are significantly higher than those for the untreated sample (sample H).

The common reaction of the BTCA and cellulose hydroxyl group was spontaneous and occurred in two steps. The BTCA reacted with the cellulose molecules (35% cotton) very likely inwardly, forming cyclic anhydrides as a reactive mediator, which esterifies cotton cellulose fabric. Employing nano TiO_2 alone may increase the bipolar gravity between polyester chains, but it may not make a crosslink with the polyester since polyester does not include any free groups; as a result, blending nano particles and acids led to more efficient wrinkle recovery in comparison with the application of each one individually.

Effect of fabric type on tensile properties in the warp and weft directions

Tensile properties of the finished samples in the warp and weft directions are illustrated in ***Figure 3***, especially for the force at the break point.

The results in ***Figure 3*** show a less significant tensile strength, which is related to the blending of carboxylic acid and nano TiO_2; hence it could be effective in the wrinkle recovery process. As has been mentioned in the wrinkle recovery section, in the weft direction with BTCA and TiO_2 treated order, the most efficient wrinkle recovery, followed by BTCA + TiO_2, was seen since in these two modes lateral cross-links are abundantly formed and as a result of that there are more nano particles found. On the other hand, tensile strength is a type of strength which is imposed parallel with the axis of the sample. Because of the cross-link composition, the strength was decreased; therefore in the finished samples the tensile strength is apparently lower.

In both the warp and weft directions, the sample concerned indicates efficient tensile strength. The reason for the nano particle existence was that the tensile strength was not reduced, which means that the nano particles in modes B & C may not make a lateral crosslink, and as was mentioned earlier they are improved because of the cellulose cross-link made; therefore bipolar gravity between polyester chains might be increased. However, sample F in the weft direction shows the minimum tensile strength in comparison with the other samples. The elongation to rupture in both directions is indicated in ***Figure 4***.

Samples treated by BTCA only, were considered as the reference and the other samples treated by nano TiO_2 only reflected elongation, which are A, H, and G respectively. This could be due to the lack of efficiency of such materials as cross-linking agents. As a result, they underwent more stretch. The results from the comparison of the samples obtained concerning the application of BTCA and nano titanium dioxide (TiO_2) in the weft mode indicate that TiO_2 usage as a catalyst in BTCA and its composition affected the wrinkle recovery process sufficiently, although it reduced the elongation.

Figure 2. Comparison of wrinkle recovery for both warp and weft directions.

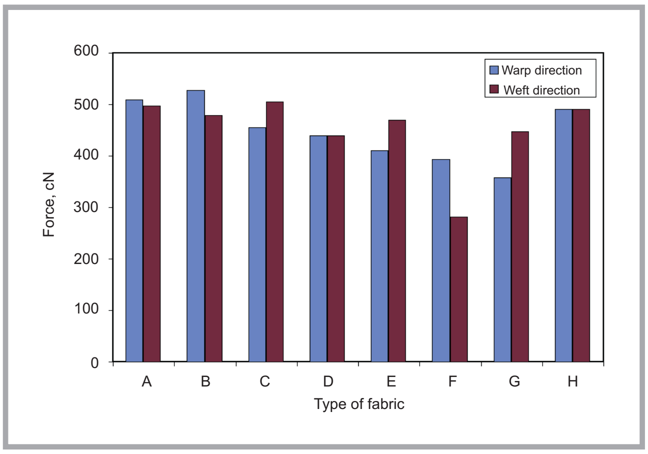

Figure 3. Comparison of the strength at the break point in both the warp and weft directions.

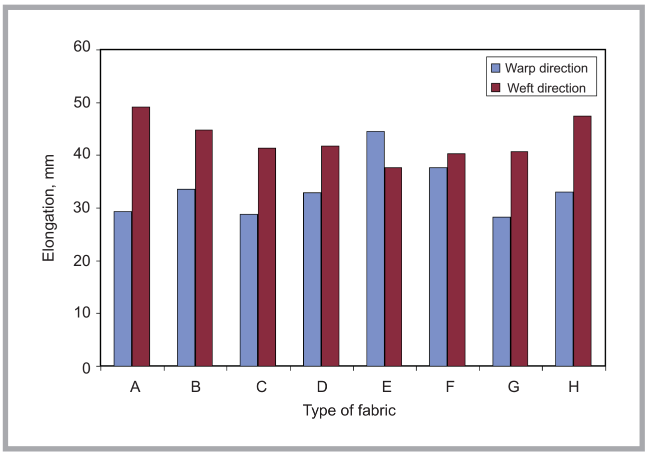

Figure 4. Comparison of elongation in both the warp and weft directions.

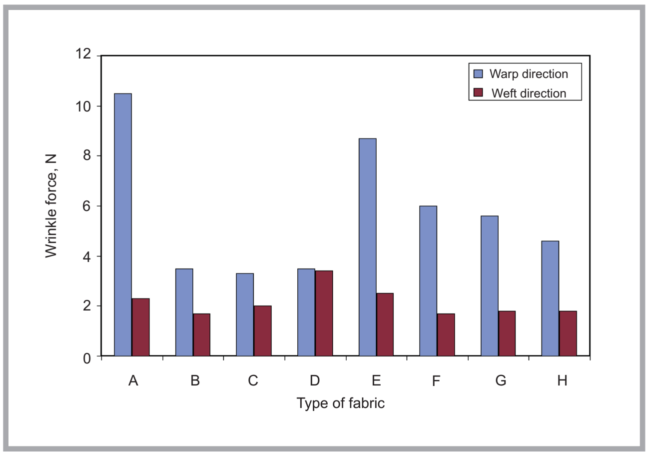

Figure 5. Comparison of the wrinkle force in both the warp and weft directions.

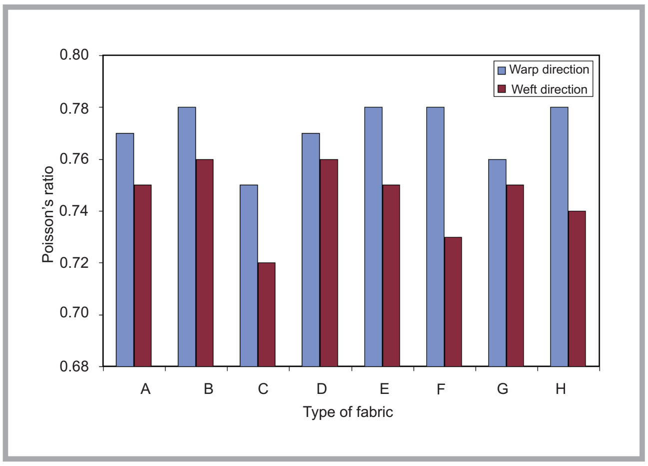

Figure 6. Comparison of Poisson's ratio in both the warp and weft directions.

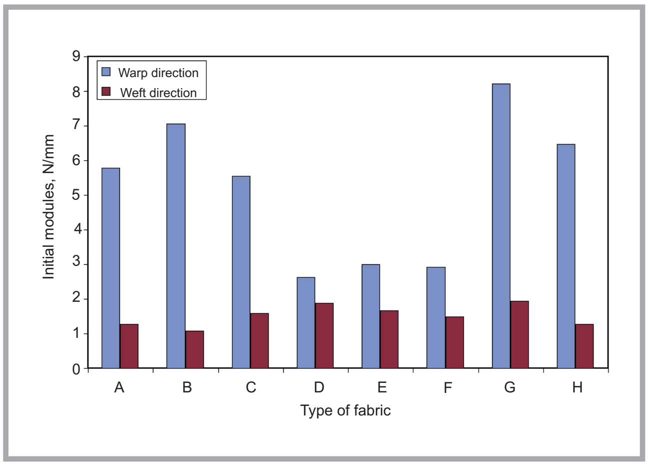

Figure 7. Comparison of the initial modulus in both the warp and weft directions.

Effect of fabric type on the wrinkle force in the warp and weft directions

The wrinkle force in the warp and weft directions is presented in ***Figure 5***.

According to the results from ***Figure 5***, it can be seen that the wrinkle force of those samples prepared by BTCA show a higher increase than the other samples. It should be mentioned that the lateral bond caused by carboxylic acid increased the force. Cross-linking among cellulosic chains causes any bond replacement or suppressing position change while imposing a force on chains in such a way as to raise the fabric resistance. Concerning nano TiO_2, it could be implied that TiO_2 particles located between chains and the cellulose construction may increase friction among chains and stop them from sliding; such a movement may increase

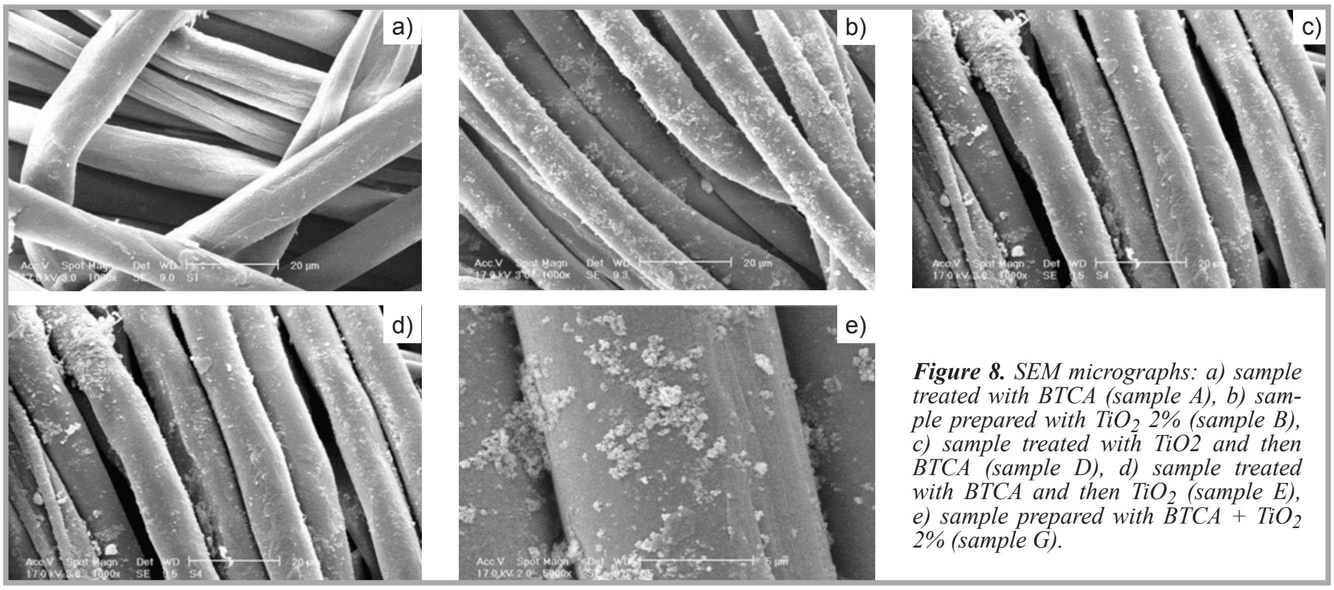

Figure 8. *SEM micrographs: a) sample treated with BTCA (sample A), b) sample prepared with TiO_2 2% (sample B), c) sample treated with TiO2 and then BTCA (sample D), d) sample treated with BTCA and then TiO_2 (sample E), e) sample prepared with BTCA + TiO_2 2% (sample G).*

the wrinkle force. As might be observed, the wrinkle force is more in the warp direction than in the weft. The most abundant elongation in the warp is observed in fabrics E and A, while the least is shown by fabrics B and C . The highest wrinkle force in the weft direction is observed for sample D, while the lowest is for sample F. In the weft direction, sample D shows the best wrinkle recovery, whereas sample F, containing a composition of BTCA + TiO_2 nano, presents the worst since, according to the microscopic evaluation by SEM, there may not be enough density of BTCA and TiO_2 to make cross-links or lateral bonds; hence, the possibility to have a cross-link is reduced.

In the warp direction, sample A, which was prepared by BTCA, reflects adequate wrinkle force, because of the bipolar gravities related to polyester chains as well as the ester bonds made between four acid groups (OH) with cellulose. Furthermore sample E indicates a desirable wrinkle force and wrinkle recovery in the warp direction for the same reason. It may make an efficient lateral bond, increasing cellulosed cross-link conformation; thus it may present more wrinkle force. Furthermore sample E reflects appropriate wrinkle recovery. Therefore there is a suitable relation between the wrinkle force and wrinkle recovery.

Effect of fabric type on Poisson's ratio in the warp and weft direction

Poisson ratio in the warp and weft direction is illustrated in ***Figure 6*** (see page 63), which shows that sample F (BTCA + TiO_2 (1%)) reflected a much higher Poisson ratio when sample C (TiO_2 (1%)) presented the minimum amount in the warp direction. In contrast, the Poisson ratio for the weft direction of sample D (BTCA + TiO_2) is reported as a maximum when the minimum amount occurs for sample C (TiO_2 (1%)). As one may observe, for sample C the lowest Poisson's ratio is achieved in both the warp and weft directions. In the weft, since the weft density is less than the warp, TiO_2 particles gather sufficiently and make a powerful electro-static bond with BTCA, while BTCA itself makes a powerful crosslink with cellulose, which is why sample D reflects the highest Poisson's ratio in the weft direction. As a result of this, the fabric's dimensional consistency may become much more efficient, and consequently it would be better with respect to wrinkle recovery and wrinkle force compared with other modes. But in the warp direction, where one may observe more warp density, in sample F the compound of (BTCA + TiO_2 (1%)) is sufficiently scattered through the fabric, as is observed in the SEM microscopical evaluation, making an efficient link with the fabric. In such a compound mode, it seems that polar gravity gets more involved in polyester chains; this is wholly observable when the warp density increases.

Effect of fabric type on the initial modulus along the warp and weft directions

The initial modulus along the warp and weft directions is shown in ***Figure 7*** (see page 63).

From the result obtained for sample G (BTCA + TiO_2 1%) and sample D (BTCA & TiO_2), the highest initial modulus may be attained in the weft direction; as evaluated earlier, both these samples present the best wrinkle recovery in the weft direction. However, sample B (TiO_2 2%) illustrates the lowest initial modulus, while in the warp direction the highest initial modulus was from sample G (BTCA + TiO_2 (1%)) and sample B (TiO_2 2%). In contrast to the weft mode, sample D (BTCA & TiO_2) shows the minimum initial modulus in the warp direction. This asserts that wherever the fabric behaves regarding Hook law and the initial modulus is abundantly in linear area, then the fabric will indicate an appropriate wrinkle recovery.

Microscopic evaluation

As can be seen in the SEM micrographs of sample D (BTCA & TiO_2) and sample G (BTCA + TiO_2 2%), there appears to be more nano particle gathering than for the other samples, which may suppress movement among lateral cross-links by BTCA in cellulosic chains. Thus sample D indicates a higher wrinkle recovery, Poisson's ratio and wrinkle force, while its tensile strength and special strength was reduced; on the other hand, elongation and tear resistance do not significantly present higher reduction.

Figure 8 shows an SEM electron microscope illustration for samples A (BTCA), B (TiO_2 2%), D (BTCA & TiO_2), E (BTCA & TiO_2) and G (BTCA + TiO_2 2%).

The SEM micrograph of sample B demonstrates that TiO_2 could not be sufficiently scattered on the fabric surface since TiO_2 would make powerful electrostatic bonds with cellulose sections and can increase the polarity of polyester. As a result of this, there would be better wrinkle recovery results comparing with sample A. Evaluating the graphs for sample E, one may realise that the sample treated by BTCA first and then TiO_2 shows better wrinkle recovery behavior compared to untreated samples, especially samples A or B. However, in comparison with the samples that were prepared with BTCA and nano TiO_2, the same results were not observed. This may be because of the fact that, despite TiO_2 increasing the possibility of shaping into a cross-link, when the sample is initially treated with BTCA spatial obstruction could prevent nano TiO_2 particles from sufficiently scattering in the sample. Therefore wrinkle recovery behaviour may not be as good as for sample D.

Conclusion

This study attempted to assess wrinkle recovery specifications of blending cotton/polyester fabric by applying BTCA with a SHP catalyst and nano titanium dioxide (nano TiO_2) individually in order and in compound modes through the pad-dry method. According to the results, conclusion could be made that nano TiO_2 and BTCA application in both kinds of ordered and compound modes would have better results compared to individual application. Furthermore, it could be asserted that TiO_2 and BTCA show the best wrinkle recovery outcome since carboxylic acids would make cross-linking cellulose, and also TiO_2 improves the process - increasing bipolar gravity among polyester chains. It could also give better mechanical properties as a reduction rate when samples were finished with BTCA. Finally, from the wrinkle recovery results, there were some differences between the parameters in the warp and weft directions which may be attributed to the orthotropic behaviour of the woven fabrics.

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Received 30.08.2011 Reviewed 23.02.2012