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Simultaneous Cross-linking and Antibacterial Finishing of Cationized Cotton by 3-chloro2-hydroxypropyl trimethylammonium chloride (Quat -188) and nano TiO_2

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Abstract

In this research, the simultaneous cross-linking and antibacterial finishing of bleached cotton and cationized cotton by 3-chloro2-hydroxy propyl tri methyl ammonium chloride (Quat -188) with butane tetra carboxylic acid (BTCA) and nano TiO_2 was explored. Butane tetra carboxylic acid can be linked to the cellulosic chains by ionic and covalent bonds. To do this, different concentrations of nano TiO_2 and BTCA were examined to obtain the highest cross-linking and antibacterial effects Various characteristics of samples such as antibacterial against different microorganisms like two gram-positive bacteria (Bacillus cereus, Staphylococcus aureus), one gram-negative bacteria (Escherichia coli), and one fungus (Candida albicans), crease recovery angle, bending length, yellowness index, weight changes, water drop absorption time were investigated. The results showed that both the bleached cotton, and cationized cotton can be finished by the optimum concentration of nano TiO_2 and BTCA for producing a cotton fabric with anti-wrinkle and antibacterial properties.

Keywords: **c**otton, butane tetra carboxylic acid, nano TiO_2, antibacterial, anti-wrinkle**.**

1. Introduction

Due to the attention paid to the properties of cotton fabrics, such as water absorption, strength, and high absorption resistance, they are considered the most famous fibers. But the tendency to wrinkle and also a suitable environment for the growth of bacteria and fungi are among their shortcomings [1]. Therefore, cross-linking and antibacterial finish of cellulosic materials are of the utmost importance. Several studies using antibacterial

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materials, such as chitosan, ammonium quaternary salts, different derivations of benzophenone, and n-halamines have been done with one of the cross-linking agents such as Dimethylol dihydroxy ethyl urea (DMDHEU), Citric acid (CA) and Butane tetracarboxylic acid (BTCA) for the improvement of cotton crease resistance. [2-11].

Microbial polluters are considered the most current pollution agents in water and air. They consist of particles with biological bases (such as bacteria, viruses, and fungi) that can operate as distributing particles in the air. One of the toxic material purification techniques in the environment is to use nano TiO_2 with ultra violet irradiation. Many studies have investigated this method of [12-18].

When a semi-conducting catalyst such as nano-TiO_2 is placed in aquatic conditions under UV irradiation, hydroxyl free radicals are generated ($^{\bullet}OH$),(Fig.1). After irradiation absorption, pairs of electrons and hole are formed in conduction and valence bands respectively (Eq.1). Some of the reactions that happen after electron formation in the conduction band (eCB) and hole in the valence band (h+VB) with organic materials (RX) have been shown in equations. 1-8.

In Oxidation reactions, electron transfer has existed from RX (Eq.2), H_2O (Eq.3), and OH (Eq.4) on the catalyst surface. Equations 3 and 4, show the process of oxidation degradation that its reason is related to much concentration of $^{\bullet}OH$ and the absorbed water on the surface of nano TiO_2 [19].

$$TiO_2 + h\nu \rightarrow TiO_2\,(e^- + h^+) \quad (1)$$

$$TiO_2(h^+) + RX \rightarrow TiO_2 + RX^{\bullet+} \quad (2)$$

$$TiO_2(h^+) + H_2O \rightarrow TiO_2 + {}^{\bullet}OH + H^+ \quad (3)$$

$$TiO_2(h^+) + {}^-OH \rightarrow TiO_2 + {}^{\bullet}OH \quad (4)$$

Figure 1. *Nano TiO_2 oxidation reactions under UV irradiation* [19]

To receive electron existing in a photocatalyst conduction band, molecular oxygen act as an acceptor particle in an electron transfer reaction. (Eq.5), (Fig.2). The superoxide anion and its proton form result in hydrogen peroxide production (Eq.6-8). In addition, the addition of hydrogen peroxide can significantly increase the photodegradation rate (Eq.9). Free hydroxyl radicals ($^{\bullet}OH$) have much activities for materials oxidation, organic pollutions and microorganism deactivation [19].

Moreover, the addition of hydrogen peroxide can significantly increase the photodegradation rate $O_2^{\bullet-} + H^+ \rightarrow HO_2^{\bullet}$ (6)

$$O_2^{\bullet-} + HO_2^{\bullet} \rightarrow {}^{\bullet}OH + O_2 + H_2O_2 \quad (7)$$

$$2HO_2^{\bullet} \rightarrow O_2 + H_2O_2 \quad (8)$$

$$TiO_2(e^-) + H_2O_2 \rightarrow TiO_2 + OH^- + {}^{\bullet}OH \quad (9)$$

Figure2. *Nano TiO_2 reactions with electron acceptor components* [19]

In 2006, Amrita Pal and his colleagues studied the photocatalyst deactivation of two species of gram-negative bacteria (Escherichia coli and Pseudomonas Fluorescens) and four species of gram- positive bacteria (Bacillus Subtilis ،Microbacteriaceae str. ،Paenibacillus sp, and Microbacterium SP) ، under two different radiation of fluorescent (including 0.013 mw/cm^2 from UV-A) and ultraviolet (4.28 mw/cm^2 from UV-A) and different concentrations of nano TiO_2 (234-8662 mg/m^2) stabilized on the acetate cellulosic membrane (average of pores measurements 0.45 µm and thickness 47 mm). Acetate groups' existence instead of hydroxyl groups in cellulose chemical structure will prevent polymer chain destruction and degradation Via activated groups during photocatalyst reaction. The results of this study show that gram negative bacteria, E. coli, has the most photo catalyst degradation and gram positive bacteria, B. subtilis, has the least effect in photo catalyst process [20].

The study of antibacterial and crease resistance of cotton fabric has continued using non-formaldehyde compounds. In 2007, Montazer and his colleague reported simultaneous antibacterial and crosslinking properties of cotton fabrics using N-2-hydroxypropyl-3-trimethylammonium chitosan chloride (HTCC) and compounds such as glutaraldehyde (GA), butanetetracarboxylic acid (BTCA) and critical acid (CA). The results indicated that cross-linking agents such as GA, BTCA, and CA not only reinforce cotton fabric against crease but also lead to the stability of antibacterial properties of compounds like chitosan and HTCC, indicating the production of crease resistance and antibacterial properties on the cotton fabrics. [21].

The reaction mechanism of the cationic agent and butane tetra carboxylic acid with cotton cellulose is indicated in Figure 3. In stage 1, intermediate anhydride cycles were formed. In stage 2, cotton fabric was cationized via a chemical reaction. In stage 3, butane tetracarboxylic acid was bonded to cotton by a cationic agent (Quat-188) via ionic linkage. In stages 4 and 5, the crosslinking of the cotton fabric by butanetetracarboxylic acid continued through an ester bond and a permanent crosslink was formed on the cotton fabric. [22].

Due to the high importance of the restriction removal of wrinkle and damage of cotton while crosslinking and finishing, and thus due to the use of environmentally friendly polycarboxylic acid compounds and nanomaterials in recent research, various cotton fabrics (bleached and cationized) were selected in this study, and their modification was investigated with BTCA and nano- TiO_2 under UV irradiation. Dry crease recovery angle (DCRA) and antibacterial properties were evaluated based on standard methods.

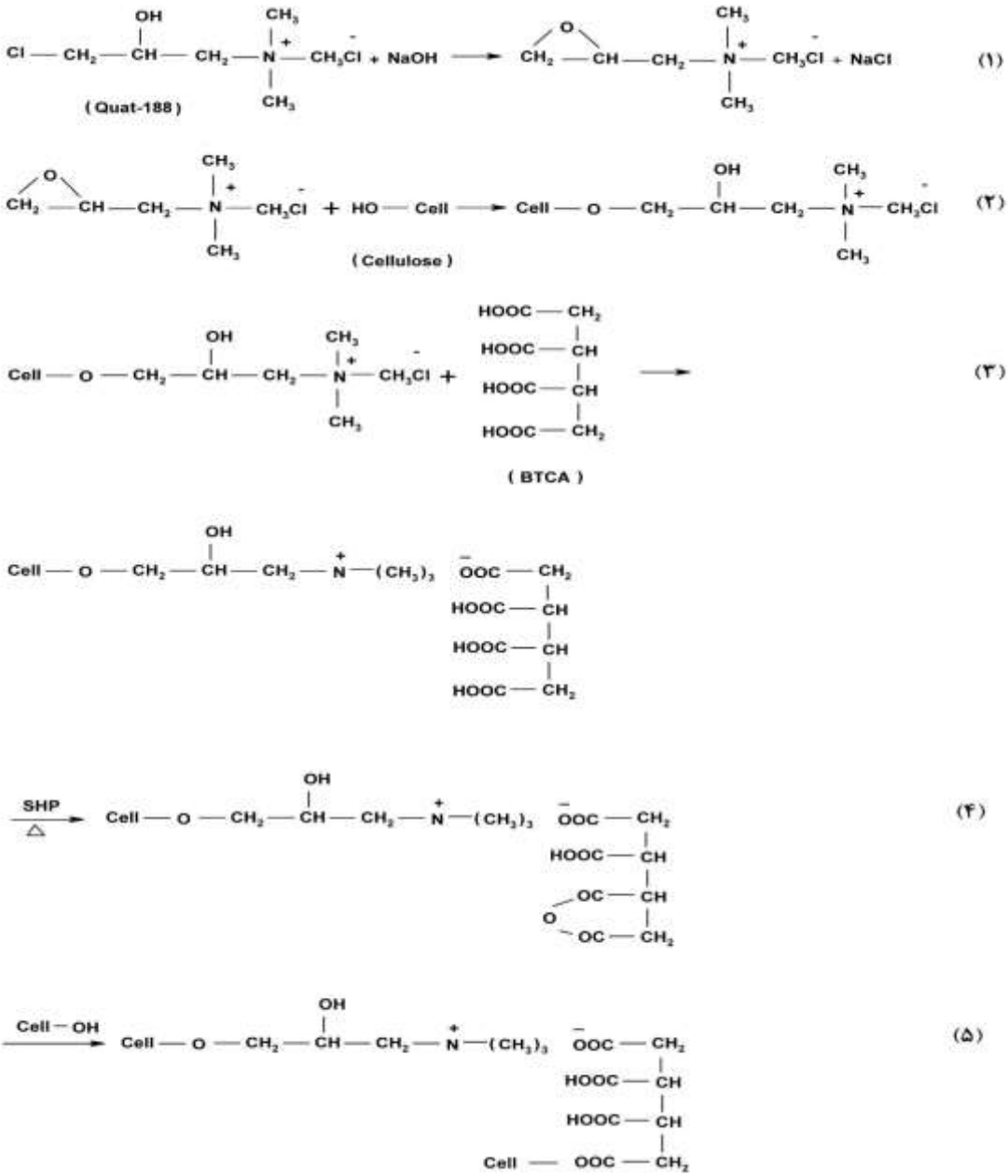

Figure 3. Mechanism of cationized and cross-linking cellulose formation with BTCA and Quat-188 [22]

2. **Experimental**

2.1. **Material**

The desized, scoured and bleached 100% cotton plain weave fabric used in the study had a wrap density of 32 yarns/cm, a weft density of 30 yarns/cm and a fabric weight of 118 g/m ^{2} and was manufactured by Yazdbaf company.

Butanetetracarboxylic acid (BTCA) and 3-chloro-2-hydroxypropyltrimethylammonium chloride (Quat-188) were produced by Fluka Company, Switzerland. Sodium hypophosphite (SHP) and fungal and bacterial growth environment (Tryptic Soy Agar) were provided by Merk company. And Nano-TiO_2 with an average particle size of 21 nm was prepared by Degussa, Germany. Two species of gram-positive bacteria, including Staphylococcus aureus (ATCC 6538) and Bacillus cereus (DTH 101), gram-negative bacteria, Escherichia coli (ATCC 11303), and a fungus, Candida albicans (ATCC 10231), were provided by Iranian industrial bacteria and fungi collection to carry out antibacterial evaluations of treated fabrics.

2.2. **Instruments**

Laboratorial pad tester adjustment capable of different Pickups, UV-C lamp (30W, Philips, Holland), Ultra sonic bath (220V, 50W, 40 KHz) were used. For crease recovery angle measurement, a thermal oven was used to dry and cure the samples. Reflectance spectra were recorded in the UV range (200-400 nm) using a UV/VIS spectrophotometer (CARRY 500 scan, Varian, Australia). Yellowness indices of treated cotton fabrics were measured using a reflectance spectrophotometer (color guide sphere, D/8-Spin, Germany). Scanning electron microscopic (SEM) observations on samples of treated fabrics was made using a LEO 440i electron microscope (UK). An X-ray diffractometer type 3003 PTS, SEIFERT, Germany (λ= 1.54060 A, at 40 kV and 30 mA) with Cu K irradiation was used to determine the crystalline phase and also the crystal size according to the Scherrer method [23]. The angle domain was 070(2). The Scherrer equation is as follows:

$$D = \frac{K\,\lambda}{B\,\cos\theta} \qquad (10)$$

where K was taken as 0.9 and B is the full width of the diffraction line at half the maximum intensity. The autoclave was used to steam sterilize solutions in antibacterial analysis and incubators to prepare a suitable environment for bacterial and fungal growth.

2.3. **Methods**

Samples were prepared in 15×6cm^2 swatches. These samples were from bleached (B) and bleached cationized (C) fabrics to conduct experiments. Fabric type (C) has undergone a cationic treatment. The cationic process was performed based on the pad-batch method using 20 g/l 3-chloro-2-hydroxypropyltrimethylammonium chloride (Quat-188) and 8 g/l sodium hydroxide with 100% wet pick-up. The samples were placed in sealed pouches for 24 hours at ambient temperature.

Then, they were rinsed several times with distilled water at 40°C. Finally, samples were put into the bath, further added 0.5 g/L acetic acid for neutralization, and then dried at 70°C temperature for 5 minutes. Specific baths were prepared for samples based on bath weight (Table 1) using different amounts of nano-TiO_2 with

butanetetracarboxylic acid (BTCA) and sodium hypophosphite (SHP) in an ultrasonic bath for 30 minutes in the dispersion form. Next, cotton fabrics (C, B) were placed in the mentioned baths for 2 minutes and after impregnation they were padded with 90% wet pickup by freshly prepared aqueous solution. Then the samples were dried at 60°C for 3 min and cured at 180°C for 2 minutes. Finally, finished fabrics were washed with 1 g/l non-ionized detergent and 1 g/l sodium carbonate at 70°C for 30 minutes.

Table1. Material and amounts in each bath to finish cotton Fabrics (o. w. b)

column	Bleached samples	Cationized samples	NanoTiO$_2$(%)	SHP (g/L)	BTCA (g/L)
Control	B	C	0	0	0
1	1- B	1 - C	2.50	0	0
2	2 -B	2 -C	0	45	75
3	3 -B	3 -C	0.12	45	75
4	4 -B	4-C	2.50	45	75
5	5 -B	5 -C	5.00	45	75

o. w. b: based on weight of bath

2.4. Yellowness *Index*

The yellowness index was measured based on the ASTM D1925 standard using a reflectance spectrophotometer, color guide sphere, D/8 rotation.

2.5. Add-*On*

First, untreated and washed samples were weighed. Upon completion, the samples were washed and reweighed. The addition level of the treated samples was calculated based on Equation 11.

$$\Delta w = \frac{w_2 - w_1}{w_1} \times 100 \qquad (11)$$

In this equation, w_1 and w_2 are the washed fabric weight before and after finishing, respectively. Δw is percentage of add-on.

2.6. Water-*drop absorption time of cotton fabrics*

For this purpose, each finished and control fabric was placed on the flat surface and then one drop was dropped vertically onto the surface using a burette (50 ml) and a 1 cm jig. The water drop absorption time was measured on the fabrics. This experiment was evaluated based on standard test method AATCC 79-2000.

2.7. **Bending *length***

To evaluate the rigidity properties of the fabric, samples were made in 62.5 cm 2 patterns based on warp direction. The bend length was evaluated based on the ASTMD 1388-96 (2002) standard and according to Equation 12

$$C = \frac{L}{2} \qquad (12)$$

In this equation, C is the bend length (cm) and L is the fabric length (cm).

2.8. **Crease *resistance evaluation***

The warp (w) plus fill (f) dry crease recovery angle (DCRA) of the treated cotton fabrics was evaluated using AATCC Test Method 66-2003.Samples were prepared in 4015mm samples and 5005g of weight was loaded on the folded samples for 5min ±5 s. The recorded vertical angle guidelines were aligned and the recovery angles were measured.

2.9. **Antibacterial *activity***

Experiments of resistance measurement and reduction of microbial agents were performed based on quantities method of AATCC 100-2004, using two species of gram-positive bacteria (Bacillus cereus, Staphylococcus aurous), one species of gram to the pour plate method and incubated for 24 hours (bacteria) and 48 hours (fungi). The mentioned conditions prepare a suitable environment for their growth. Finally, colony forming units (CFU) in each petri dish were counted in 0.1 ml. Percent microbial reduction was determined according to Equation 13. negative bacteria (Escherichia coli) and one fungi (Candida albicans). In this procedure, 1.5×1.5cm2 samples were placed next to a bacterial suspension prepared on the basis of 0.5 Mac Farland. Then the nano-TiO_2 treated samples were exposed to (UV-C) radiation for 30 minutes.

Thereafter, they were incubated for 24 hours. Next, to evaluate the amounts of bacteria and fungi, 0.1 ml of the suspension was added to the culture environment according

$$R(\%) = \frac{C_0 - C_1}{C_0} \times 100 \qquad (13)$$

In this equation, R (%) is the reduction amount, C_0 is the amount of colonies existed in samples without microbial suspension and C_1 is the amount of colonies existed in treated samples with microbial suspension.

Table2. Compositions utilized in tryptic soy agar culture environment

	Typical compositions	g/L
1-	Peptone from casein	15.0
2-	Peptone from soy meal	5.0
3-	Sodium chloride	5.0
4-	Agar-agar	15.0

2.10. Ultraviolet *irradiation time*

In order to evaluate the ultraviolet exposure (UV-C) separately from the microbial agent, each bacterial and fungal suspension was prepared based on the 0.5 Mac Farland standard and for different times of exposure (0, 15, 30 and 45 min). Then the percentage reduction of the microbial agent was determined

3. Result and discussion

Add-on percentage in Figure 4 shows that the largest weight increase percentage is associated with samples 5 having the highest amount of nano- TiO_2 and butanetetracarboxylic acid. This means that the finishing amount in samples 5 is higher than in other samples. The reason for this may be related to the large amount of Nano-TiO_2 present in the finishing bath and consequently its strong maintenance by butanetetracarboxylic acid crosslinking agents [24]. The least amount of weight increase percentage is related to the utilization of nano TiO_2 alone. In this case, due to the lack of BTCA stable agent in Sample 1, there is the lower percentage of weight increase on the cotton fabrics.

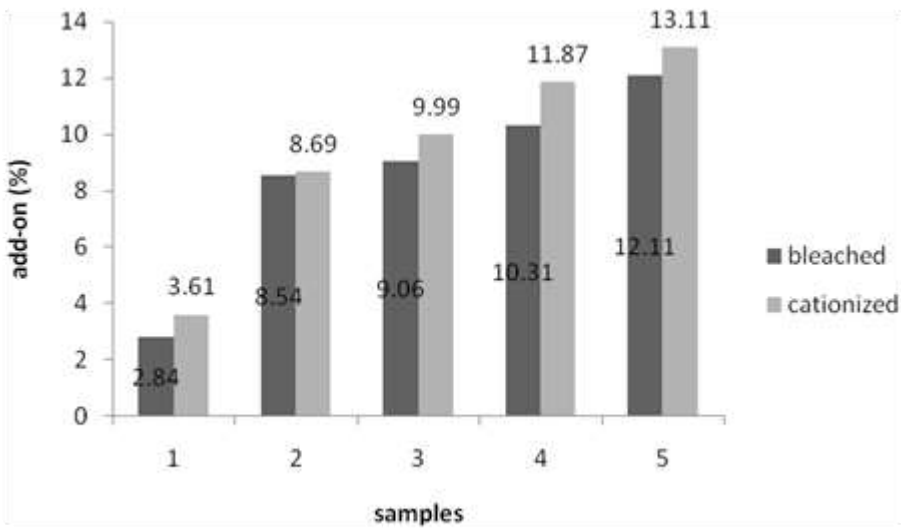

Figure 4. Weight increase percentage of treated samples

In Figure 5, the yellowness indices of treated fabrics show that the amount of yellowing of all treated samples is low, while the highest amount of yellowing is associated with fabrics treated with Nano TiO_2 without the presence of BTCA. The reason is simply the agglomeration of nanoparticles on the cotton fabric surface. In addition, samples treated with only BTCA have the lowest yellowness index as control samples.

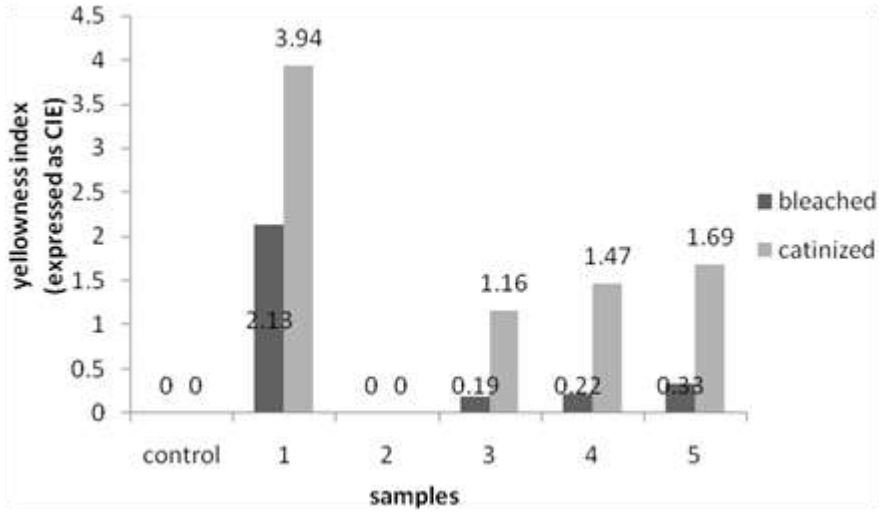

Figure 5. Yellowness indexes of control and treated samples

Figure 6 determines the water droplet absorption time of samples in seconds. It is observed that when nano TiO_2 is used alone, the highest delay in water droplet absorption time occurs on the fabric surface, which can be attributed to the formed layers on the fabric surface and no accessibility of water molecules to the fabric surface pores. However, cationized samples have a shorter water drop absorption time which can be attributed to better access to hydrophilic groups of the cationic agent in the fiber surface.

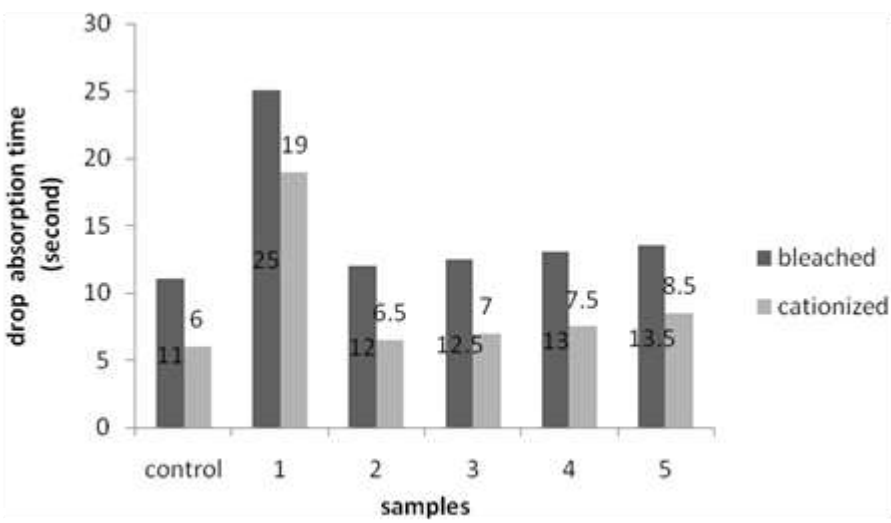

Figure 6. Water drop-absorption time on the samples surface

Bending length evaluation is considered a criterion of the finished fabric handle. As the results show in Figure 7, the difference among samples is not so perceptible. However, only nano TiO_2 has slightly more bending length compared to only BTCA and its catalyst. This may be due to more nano- TiO_2 deposition on the fabric surface and the formation of a less flexible surface. Additionally, the chemical combination of BTCA with hydroxyl groups of polymer chains within cellulose causes only a slight improvement in flexibility in the acidic state. A small increase in bending length of cationized samples compared to bleached samples may be due to ionic bonding between active groups of the cationic agent (Quat-188) and BTCA.

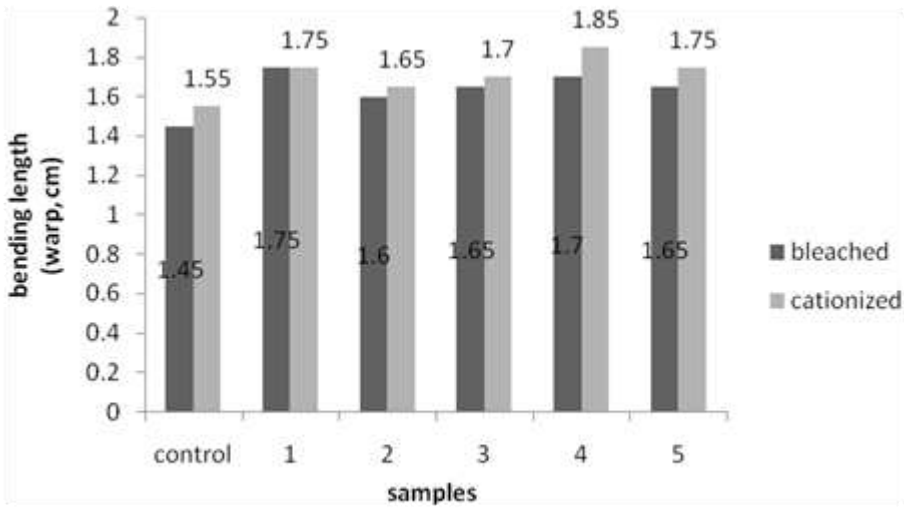

Figure7. Bending length of treated samples on warp direction

As shown in Figure 8, cationized treated fabrics exhibit the higher Dry Crease Recovery Angle (DCRA) compared to bleached samples which can be attributed to the ionic linkage among cationic centers of Quat-188 agent and anionic centers of BTCA, in addition to formed ester linkage between BTCA and cellulose polymer chains. These ester linkages are the only way of bond in bleached cotton with BTCA. The increase of nano TiO_2 concentration leads to high DCRA. The reason is related to the more cross-linking ability of the cotton-cellulose surface, but higher nano-TiO_2 concentration causes a decrease of DCRA. It can be attributed to the nano- TiO_2 agglomeration, which will lead to their preventive effect in forming cross-linkages via BTCA with cellulosic chains.

Figure 8. Dry crease recovery angle of control and treated samples

3.1. Antibacterial properties

Evaluation of the antibacterial properties of various bleached and cationized cotton samples was performed based on a standard quantitative method. As shown in Figure 9, it is clear that just UV irradiation is capable of removing microbes. But in the short time from 0 to 45 min, this amount is small and the highest decrease is 6.63% for C. Albicans, 8.1% for B. Cereus, 10.61% for Staph. Aureus and 12.54% for E. Coli.

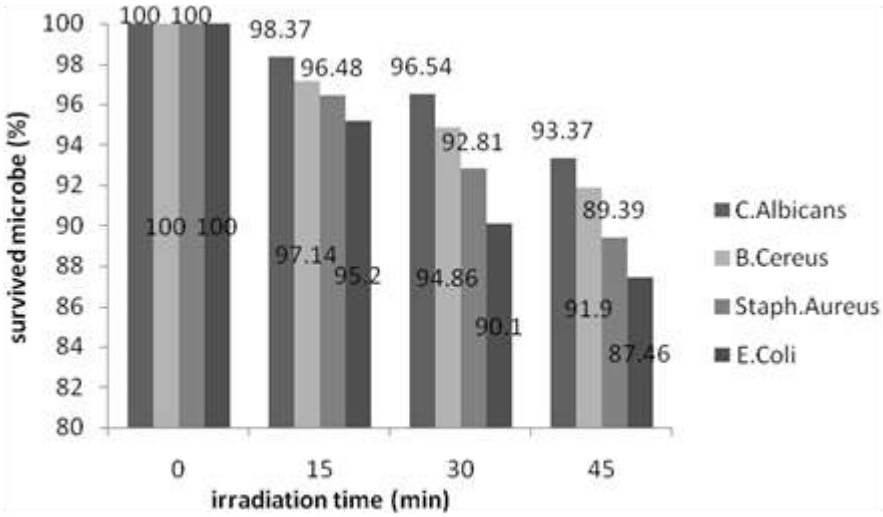

Figure 9. Effect of UV-C irradiation times on the percentage of survived microbes

Samples finished with various agents (Fig. 10 and 11) have antibacterial properties. Cationised fabrics with the cationic agent 3-chloro-2-hydroxypropyltrimethylammonium chloride have a stronger antibacterial effect compared to bleached samples. It can be attributed to ammonium quaternary groups connected to cellulose fiber and their reaction with membrane negative charge and microorganism cytoplasm and their degradation. In this study, BTCA was considered as a cross-linking agent and used as a nano- TiO_2 protector, in addition to its function as a potent antibacterial agent. It can be a result of ionic reactions between BTCA and microbial ingredients and the degradation of their activity. Nano TiO2, with UV irradiation, shows microbes' deactivation properties that reason is hydroxyl active radicals production via system (Nano TiO_2 –UV) and microorganism degradation. Increasing the nano- TiO_2 concentration leads to an increase in antibacterial properties. At higher concentrations, however, the antibacterial effect decreases. The reason is the agglomeration of nano- TiO_2 particles and the decrease in their performance, Figure. 12.

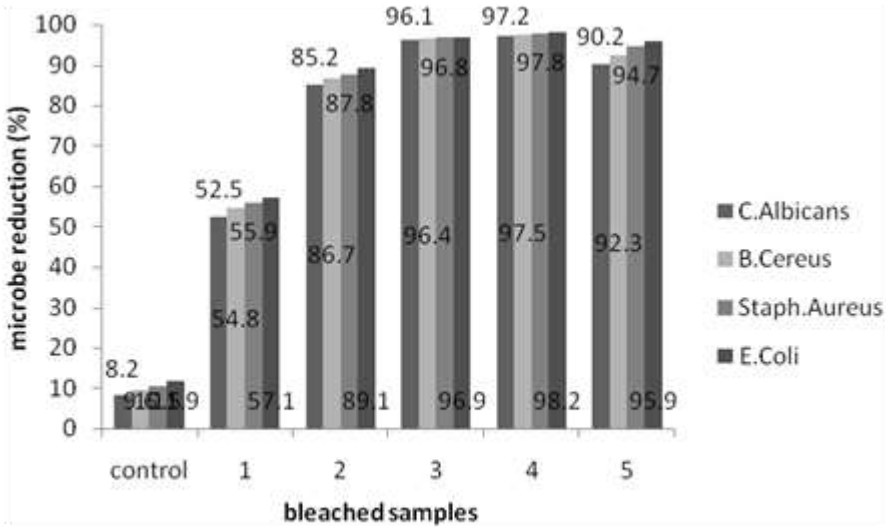

Figure10. Reduction of microbe's percentage with bleached samples

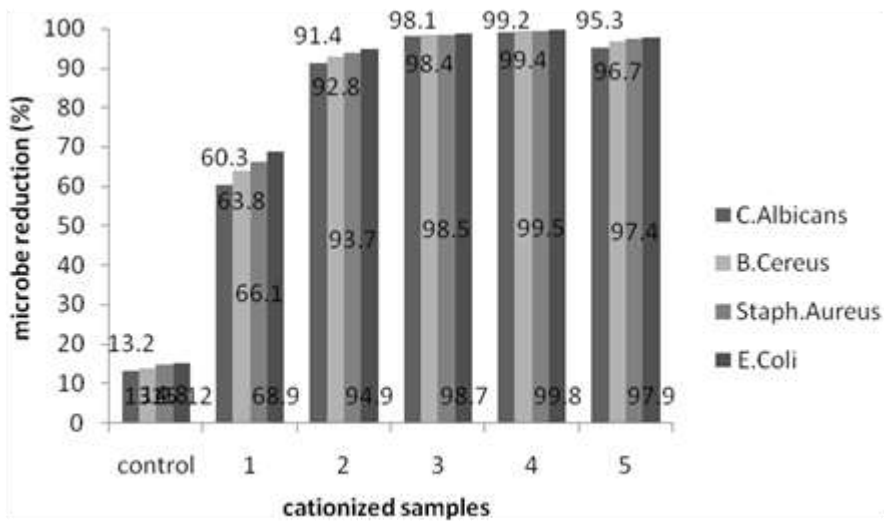

Figure11. Reduction of microbes' percentage with cationized samples

(a) (b)

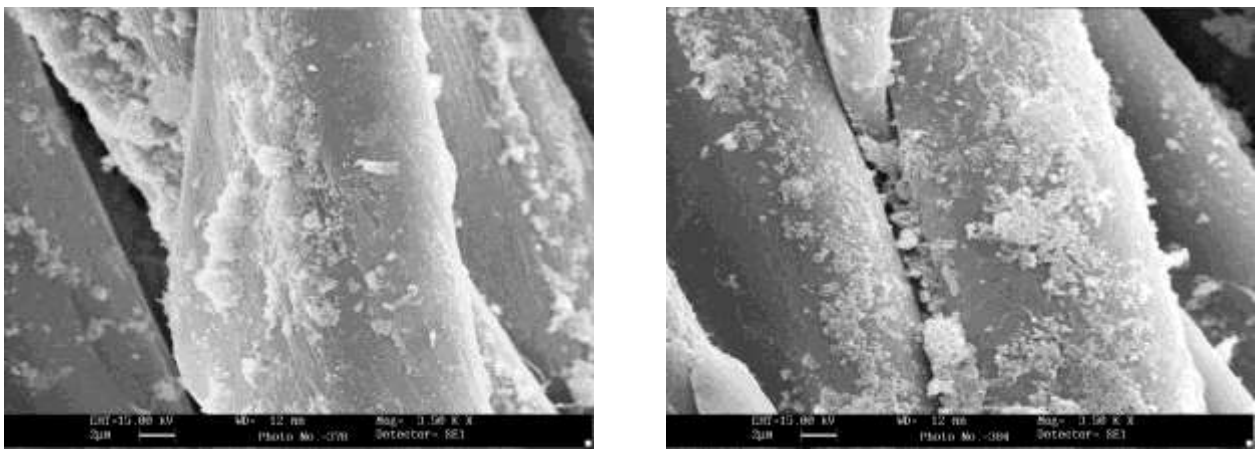

Figure12. SEM images of cotton fabric: (a) bleached sample of B-5 (b) cationized sample of C-5

In this study, the reduction of two current gram-positive bacteria, Staphylococcus aureus (source of infection for eyes, skin, bones and joints) and Bacillus cereus (source of foodborne illness, blood and digestive infection), gram-negative bacteria, Escherichia coli (source of urine, hospital and blood infection) and fungi, Candida albicans (source of aphtha in infant's mouth) were studied. In general, the high performance of the antibacterial properties of the treated samples compared to control samples in reducing Staphylococcus aureus (Fig. 13), Escherichia coli (Fig. 14), Bacillus cereus (Fig. 15) and Candida albicans (Fig. 16). Fungi Candida albicans show more resistance as compared to other bacteria. This is because Fungi Candida albicans have been listed as a mono-cell fungus (fermented). Because existing of cellulose, hemi cellulose, and chitin are the most important, the ferment wall shows more resistance than bacteria. Since many properties in funguses, such as life mechanism, cell envelope, and metabolism, are different from bacteria. Gram-positive bacteria, Bacillus Cereus with spore, are another resistant microorganism after C.albicans. Spore is the bacteria resistant to undesirable environmental conditions like high or low temperatures, acidic pH, and the lack of nutrient materials. Therefore, in antibacterial evaluation, this bacteria's resistance is more than other bacteria. Gram-positive bacteria staphylococcus aureus is another type of bacteria that shows more resistance as compared with Escherichia coli. The main difference between these bacteria is their cell wall and the amount of peptidoglycan (P.G). P.G thickness of gram-positive is more than gram-negative bacteria. Therefore, they show more resistance against antibacterial agents. Bacteria degradation by antibacterial material in different cationized and bleached cotton samples can be because of bacteria cytoplasmic membrane degradation, spatial destruction and degradation of bacteria enzyme, chromosome damage, and bacteria wall destruction [25].

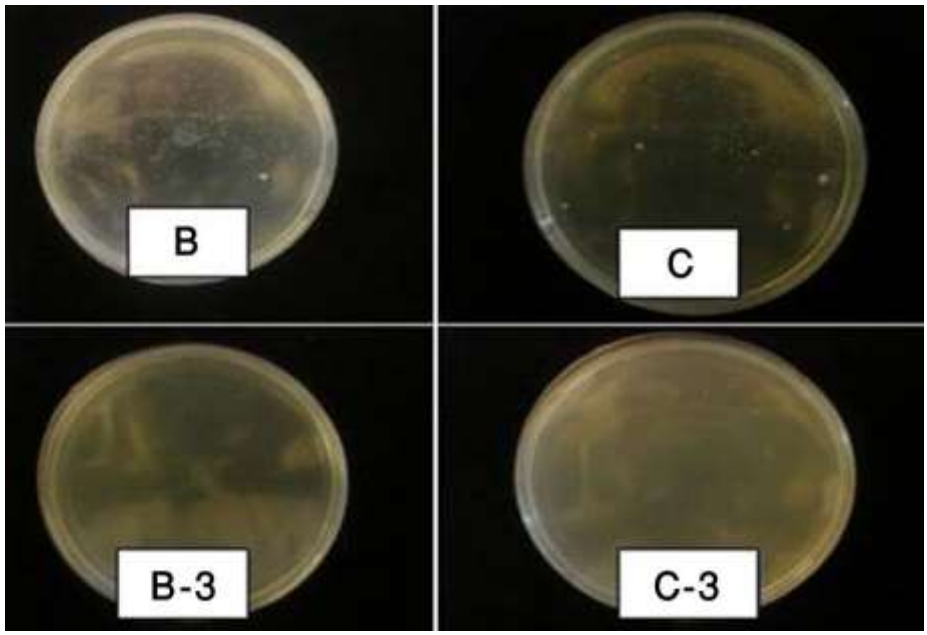

Figure13. The comparison between antibacterial properties of treated and control samples against staphylococcus aureus

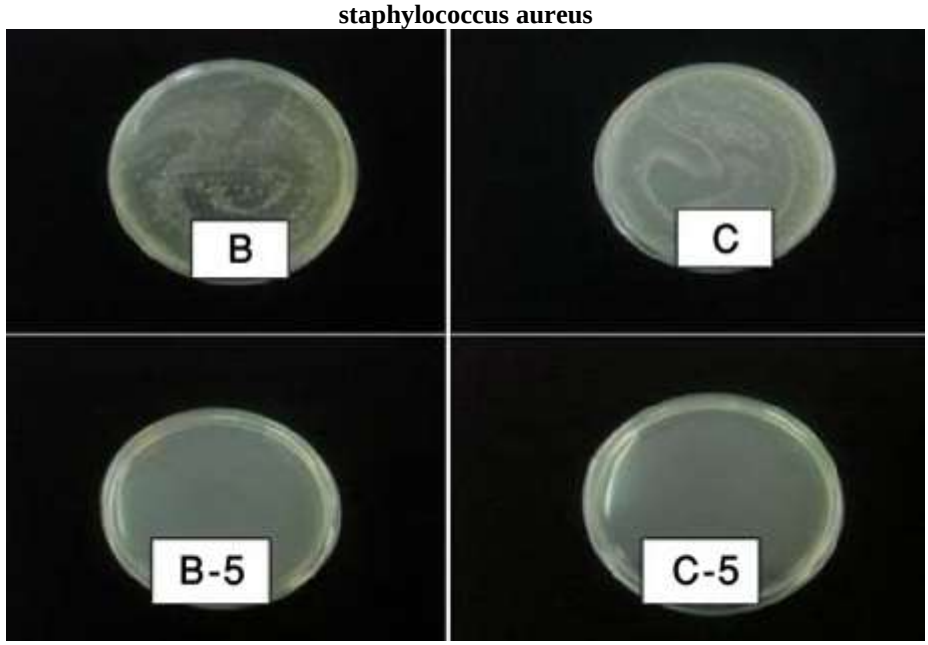

Figure14. The comparison between antibacterial properties of treated and control samples against Escherichia coli

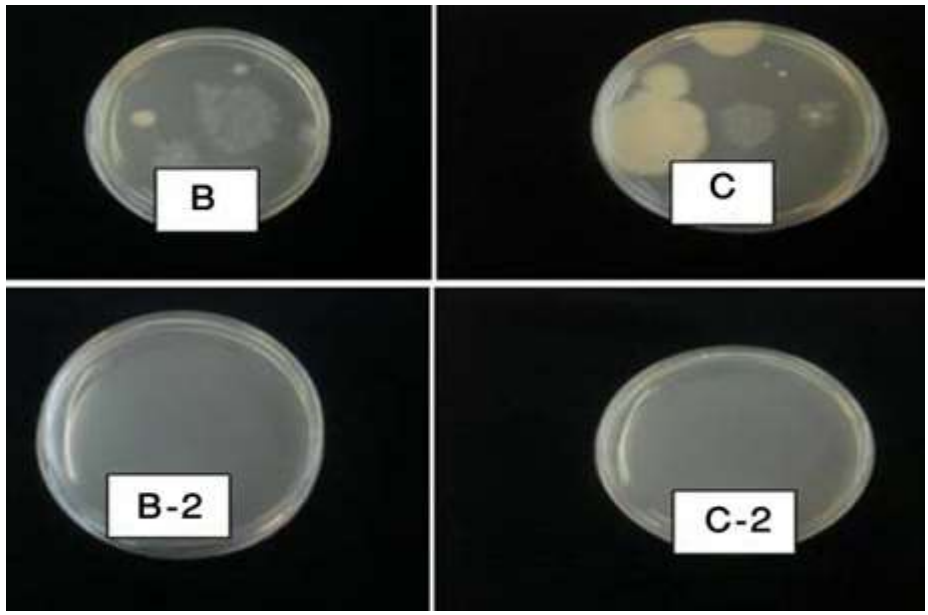

Figure15. The comparison between antibacterial properties of treated and control samples against bacillus cereus

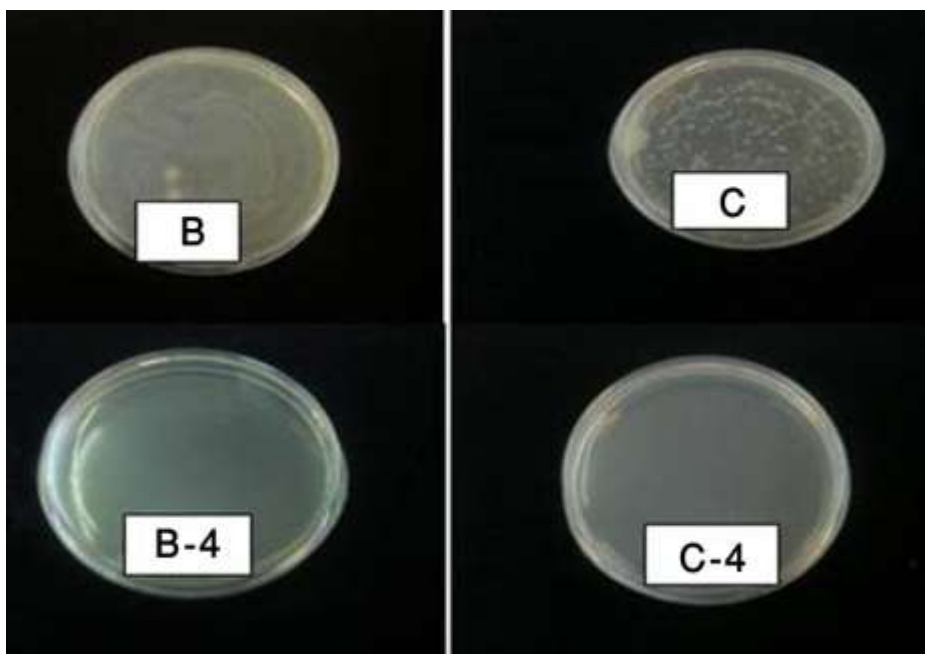

Figure16. The comparison between anti-fungi properties of treated and control samples against Candida albicans

3.2. Statistical analysis

Pearson correlation coefficient was used to study the significant effect of nano TiO_2 amounts on antibacterial and crease recovery angle characteristics, considering that independent and dependent variables were interval scales. In this analysis, if Sig. < 0.05, variation is significant, otherwise, it is insignificant.

Results show that nano TiO_2 amounts significantly affect the antibacterial and crease recovery angle characteristics of bleached and cationized samples. Also, to study the significant difference between bending length amounts of different samples, the means were compared, and an independent sample t-test, with a confidence level of 95%. Results reveal that there is not a significant difference between bending length amounts of bleached and cationized samples. Reflectance Spectrums of selected bleached and cationized Cotton Fabrics.

Figure 17 shows the reflectance curves of the treated and control samples in the ultra-violet region (200-400nm). Samples B and C are related to the control fabrics. In other samples, the reflectance values decreased, confirming the presence of nano TiO_2 particles. These particles are able to absorb wavelengths less than 358nmIn samples B-1 and C-1 that contains nano TiO_2 alone, compared with other samples the least reflectance is observed in 200-385 nm. It is because of the higher possibility of contact between UV wavelength and nano TiO_2 particles in the absence of BTCA molecules.

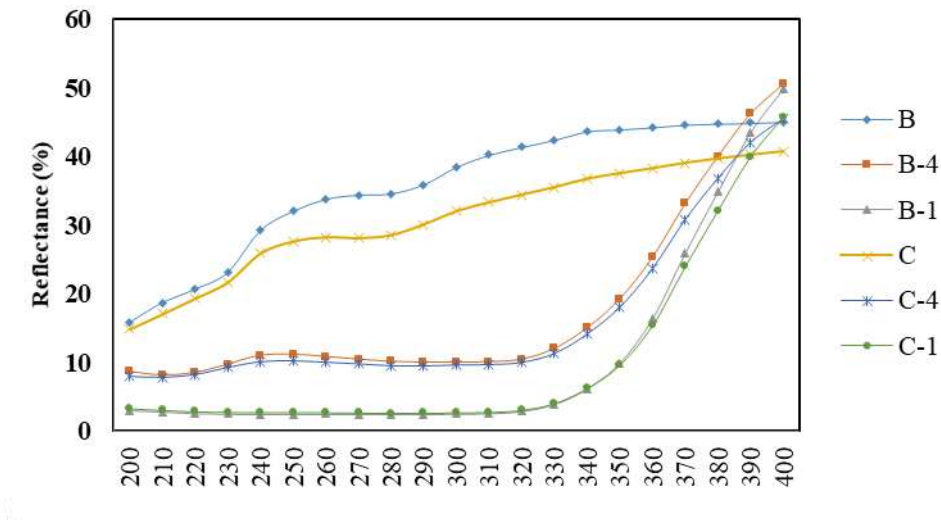

Figure17. Reflectance versus wavelength for different treated samples

(B, B-4, B-1, C, C-4, C-1)

3.3. X-ray Diffraction (XRD) Analysis

The XRD patterns of pure nano TiO_2 (Fig. 19), sample C-4 (Fig. 20) are reported. It can be observed that the highest peak of both spectrums is anatase (2θ=25.2°), whereas the peak related to rutile phase (2θ=27.5°) cannot be observed in XRD spectrums [26]. Therefore, the coated fabrics with nano TiO_2 particles have an anatase crystallite phase and can effectively operate under UV irradiation. From full width at half maximum (FWHM)

of the peak at 25.2° (0.3971) and using Scherrer's equation, the average crystal sizes of about 3.6 nm, can be calculated.

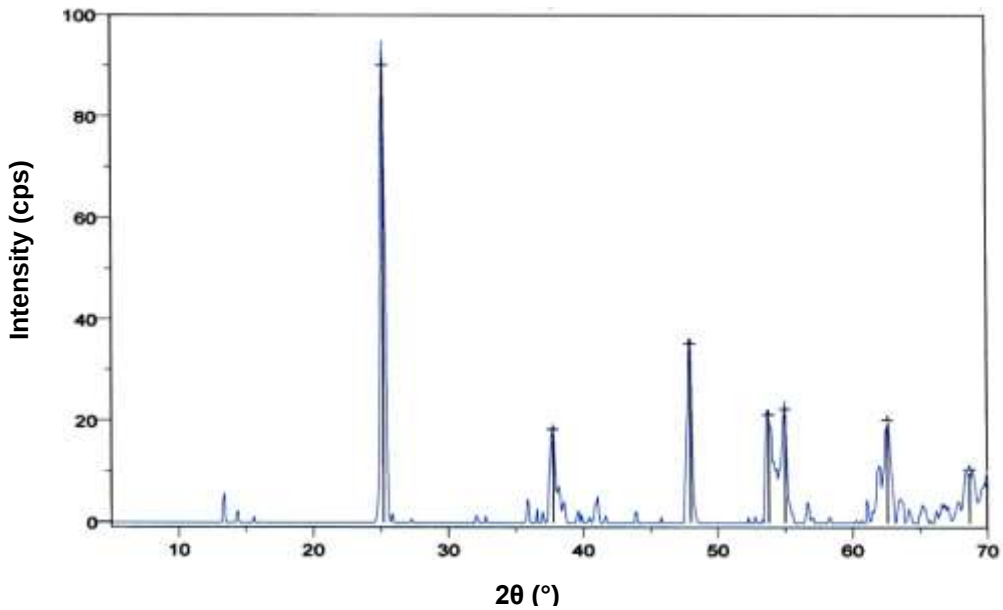

Figure18. XRD pattern of pure nano TiO_2 powders

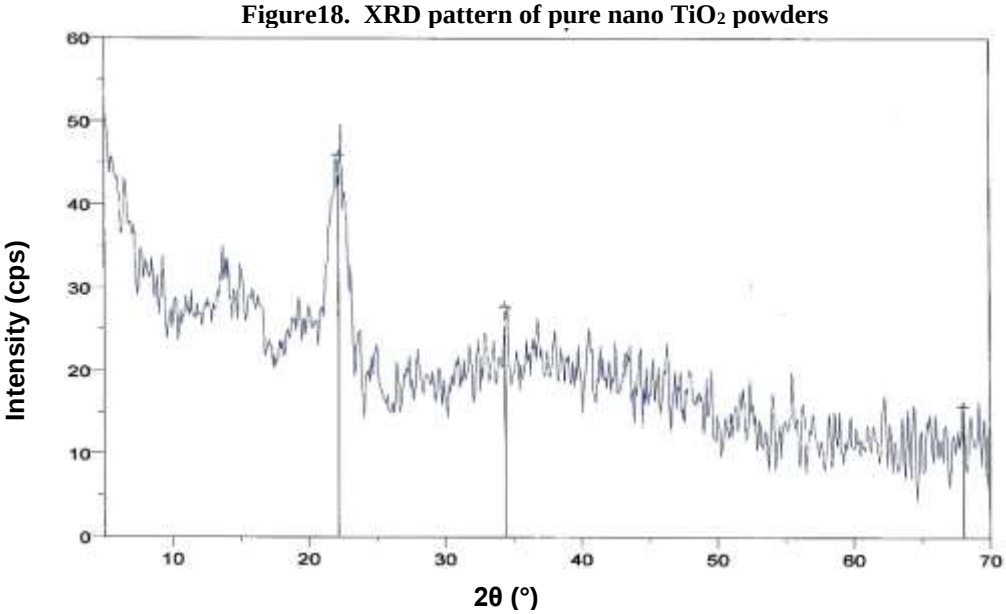

Figure19. XRD pattern of sample C-4

4. **Conclusion**

In this study, bleached and cationized fabrics treated with BTCA (non-formaldehyde and environmentally friendly material) and nano TiO_2 were considered appropriate compositions for concurrent antibacterial and cross-linking finishing. BTCA was used as a cross-linking agent. It leads to the durability of nano TiO2. Also, BTCA was presented as an antibacterial agent. So, finished fabrics with BTCA and nano TiO_2 have antibacterial and crease-resistance properties. Among the used amounts of the mentioned compound, sample 4, including 75 g/L of BTCA, 45 g/L of SHP, and 2.50% of nano TiO_2 was optimized. Bleached and cationized samples 4 show more than 97% and 99% reduction respectively of two species of gram-positive bacteria (Bacillus Cereus, Staphylococcus aureus) and one species of gram-negative bacteria (Escherichia Coli) and fungi (Candida Albicans).

Also, they show the highest DCRA. Other treated fabrics with these two compounds display high performance of these two properties. Results show that BTCA with nano TiO_2 has complementary effects and the fabrics produced have multifunctional properties. Also, they have desirable yellowness indexes and water drop absorption time on the fabric surface and have no noticeable difference from control samples.

5. References

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