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Development and Evaluation of Novel Multifunction Hybrid Containing Cationic Softener /TiO_2 /Herbal Oil for Cotton Based Fabrics

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CURRENT research was undertaken to develop a new finishing formulation using titanium dioxide nanoparticles (TiO_2) homogenously suspended in mixture of cationic softener and herbal oil. Its utilization as multifunctional finishing formulation to cotton based fabrics was investigated. The properties of the treated fabrics were monitored and compared with the untreated one. These properties were include the effect of cationic softener concentration, form of TiO_2 nano particles (mixture of rutile and anatase), type of cotton fabrics (bleached or pre-carboxymethylated) and presence of herbal oil on the properties of the treated cotton fabrics were monitored. Moreover, stability of TiO_2 nanoparticles in such finishing formulation was also determined. The effect of using eco-friendly herbal oil within the same finishing formulation on the antibacterial activities of the treated fabric was also evaluated.

The treated fabrics were monitored for UV-protection, self-cleaning and antibacterial properties. Morphology and structure of TiO_2 nanoparticles were characterized using XRD and TEM,. The antibacterial activities of the treated fabrics were evaluated against Staphylococcus aureus (gram-positive) strains and Escherichia coli (gram-negative), the results show significant antibacterial effect mainly against Staphylococcus aureus. In addition, the treated fabrics showed enhanced UV protection and self-cleaning properties with improving softness properties.

Keywords: Cationic softener, Titanium dioxide Nanoparticles, UV protection, Self-cleaning, Cotton Fabrics, Herbal Oil, Antimicrobial.

Introductions

Textile hand is a terminology that describes both touch sensation of textile material and also final garment quality that could be modified during industrial finishing. The textile finishing process affects both softness and stiffness of textile materials that ultimately determine tactile comfort while wearing [1]. Further modifications to textile materials appear also during wearing and post-laundering processes.

Softeners are the most important global textile finishing chemicals in terms of value and amount. It may be anionic, cationic, amphoteric and nonionic, but the cationics are the most effective [2, 3]. Softener molecules could be deposited on textile surface in a process that depends mainly on ionic nature of textile surface hydrophilicity and also softener molecules. It is reported that small softener molecules has the ability to penetrate fiber materials resulting in internal plasticization for the fiber material through formation of polymer that reduces its glass transition temperature. The physical positioning of these softener molecules on textile surfaces is crucial. It is dependent s on the hydrophilic character of the surface and ionic nature of softener molecules. Cationic softeners, for instance, are arranged so that the positively charged groups are directed towards partially negatively charges of the fiber material. This orientation offers maximum softness and lubrication properties for the fabric materials [4, 5]. Softeners have been reported to produce the

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softest possible hand to improve crease recovery, tear & abrasion resistance and they are excellent for improving sewing properties of fabrics[6].

The use of nanotechnology in the finishing of technical textiles has resulted in imparting new and more complex functions on textile substrates as well as improvements in existing functions such as durability without losing the fabric's feel and texture, with the minimum use of chemicals. [7-9].

The versatile applications for TiO_2 photocatalysis in window glass, cement and textile attracted superior attention to many researchers [10, 11].Various researchers have reported the incorporation of nano-TiO_2 in textile finishing to enhance maximal UV protection, and antimicrobial activity in addition to self-cleaning property [12-14]. They also do not affect handle or breathability of the fabric surface [15]. The ability of TiO_2 nano particles to impart such properties is attributed to its ability to produces highly active radical species such as hydroxyl and superoxide ions when expose to ultraviolet irradiation[16] . These radical species are involved in further series of oxidation reactions that lead to organic contaminants destruction through mineralization action and are also capable of damaging microorganisms through their powerful oxidizing property [17].

Because of increased global awareness of environmental pollution, herbal oil and plants extracts become value added products [18-20] in textile finishing.

Current research work aimed at development a novel multifunctional finishing formulation for cotton fabrics for the sake of imparting the fabric several permanent and desired properties in one-step treatment process. These properties include softness, UV protection, self-cleaning, and antimicrobial properties. Commercially available cationic agent will utilized to serve as cationic agent and carrier for TiO_2 nanoparticles and eco-friendly herbal oil.

Experimental

Cotton fabric

Mill desized, bleached and scoured cotton fabric, plain weaved, provided by El-Nasr Company for spinning weaving and Dyeing (El-Mahallah El-Kubra, Egypt). The fabric was washed with aqueous solution containing 1 g/L non-ionic surfactant and 20 g/L sodium carbonate at 100^{o}C for 60 min. Ultimately, the fabric was washed with boiling water several times followed by cold water prior to final drying at room temperature.

Chemicals

SAPAMINE® KL NEW, commercially cationic softener was supplied by Huntsman (Germany) GmbH, titanium dioxide, mixture of rutile and anatase was provided by Aldrich chemistry, all other chemicals are of laboratory grade.

Fabric treatment

Carboxymethylation of cotton fabric (CM-Cotton)

Partial carboxylation for cotton fabric was carried out by as previously reported [19]. CM-cotton is produced commercially in a two-stage process. The first step is a caustification treatment, in which bleached cotton fabric was impregnated in 15% sodium hydroxide solution at ambient temperature for 5 minutes then squeezed to a wet pick up of 100% and dried for 5 minutes at 60^{o}C. The second stage is the etherification treatment in which the alkali treated samples were steeped in ammonium salt of monochloroacetic acid solution (0–3 mol) at room temperature for 5 minutes. Those samples were then squeezed to 100% wet pick up, sealed and then heated at 80^{o}C for 1 hr followed by washing and drying at ambient temperature.

Treatment of cotton fabrics with TiO_2 nanoparticles/-softener formulation

Aqueous dispersion was prepared by the mixing different concentrations of cationic softener (2-5%) with 1%TiO_2 nanoparticles then sonicated in ultrasonic water bath for 5 min. The cotton fabric samples and CM- cotton samples is putting separately in the treatment bath where the treatment bath of cotton fabric contain Fixapret ECO®(50 g/L), as crosslinking agent and $MgCl2•6H2O$ (10 g/L) as a catalyst. The fabrics samples were padded twice in a laboratory-padding machine. Then samples were dried immediately at 80°C for 5 min and cured at 130°C for 3 min.

For those samples treated with similar formulation but in presence of herbal oil, similar procedure were employed except subjecting the formulation to stirring using homogenizer for 30 min before application.

Testing and Analysis

Transmission Electron Microscopy (TEM)

Shape and size of TiO_2NPs were obtained using TEM; JEOL-JEM-1200. Specimens for TEM measurements were prepared by placing colloidal solution droplets on 400 mesh copper grid coated with amorphous carbon film and then evaporating solvent at room temperature.

Scanning electron microscopy measurements

The Philips XL30 scanning electron microscope (SEM) was used for microscopical analysis on textile samples. This apparatus is equipped with the LaB6 electron gun and the Philips-EDAX/DX4 spectroscope. The secondary electrons (SE) approach was implemented to take images at different magnification power based on image clarity. Textile samples were fixed and metalized with carbon and gold vapor deposition respectively to record images.

Fourier transform infrared (FTIR)

The ATR-FTIR instrument (JASCO, Model IR 4700 Japan) was used to measure infrared spectroscopy.

Antibacterial tests

For reproducibility purposes, antibacterial tests were performed in triplicates. Antibacterial property was tested against E. Coli and staphylococcus Aureus, (ATCC 1533) bacteria through disk diffusion method. All petri dishes and nutrient used for these tests were autoclaved efficiently. Liquidated agar was cast on petri dishes and allowed to cool in a laminar airflow. Around 105 colonies of E. Coli bacteria were inoculated on each petri dish and a piece of each fabric sample sized 292 cm2 was planted onto agar dishes. All petri-dishes were incubated at 37^oC for 24 hours and tested for inhibition zones.

Wrinkle recovery angles measurement

Dry crease recovery angles (DRA) for treated samples were evaluated in wet and warp directions; (w+f) based on the AATCC standard method [66-1998].

Roughness measurement

The Surfacoder was used to evaluate roughness based on the AATCC standard test method (1700a).

Tensile strength

The strip method as used to determine tensile strength based on the ASTM, Standard Test Methoin accordance with ASTM D5035.

UV protection factor

The Perkin Elmer Lambda (USA) 3BUV–Vis spectrometer apparatus was used to measure ultraviolet–visible spectrum. The ultraviolet Shimadzu 3101 PC-Spectrophotometer apparatus measured ultraviolet protection factor (UPF). Ultraviolet classification and protection were determined based on AS/NZS 4399:1996 [20].

Results and Discussion

The main aim for this work is to develop desired functionalities with enhancement of cotton fabric performance such as ultraviolet protection, self-cleaning and antibacterial properties using finishing formulation hybrid consists of TiO_2 nanoparticles, cationic softener and/or herbal oil.

TEM

Figure1a and 2b show the morphology of TiO_2 nanoparticles. The figure shows homogeneous morphology with uniform and spherical particles size distribution of TiO_2. The particle size is ranging from 10–14 nm. The stability of TiO_2 nanoparticles in the cationic formula are described in Fig. 1b.

X-ray diffractometry

Figure 2a and 2b show XRD patterns for cotton and carboxymethyl cotton fabric treated with cationic softener and titanium dioxide. This figure shows anatase form as major spectrum peaks (2θ= 22.4◦). Therefore, the coated fabrics with cationic softener and TiO_2 nanoparticles and CARBOXYMETHYL-cotton with cationic softener and TiO_2 have anatase crystallite phase.

FTIR Spectroscopy

Green CM-Cotton, Blue CM-Cotton, 5% Cationic softener., Red CM-Cotton +5% Cationic softener+ 1%TiO_2 nanoparticles.

Physical properties

The physical properties for treated and untreated fabrics in terms of tensile strength and roughness were evaluated and detailed in figure 4 and figure 5 respectively. The tensile strengths for treated cotton or CM-cotton in addition to coated cotton are decreased slightly. Fabric roughness has also slightly decreased, but in a tolerable degree by changing the cationic softener concentrations in presence and absence of TiO_2 nanoparticles. The physical properties of treated fabric doesn't have marginal change inspire of enhancement in the crease recovery angle which show an improvement either for cotton treated or for CM-cotton.

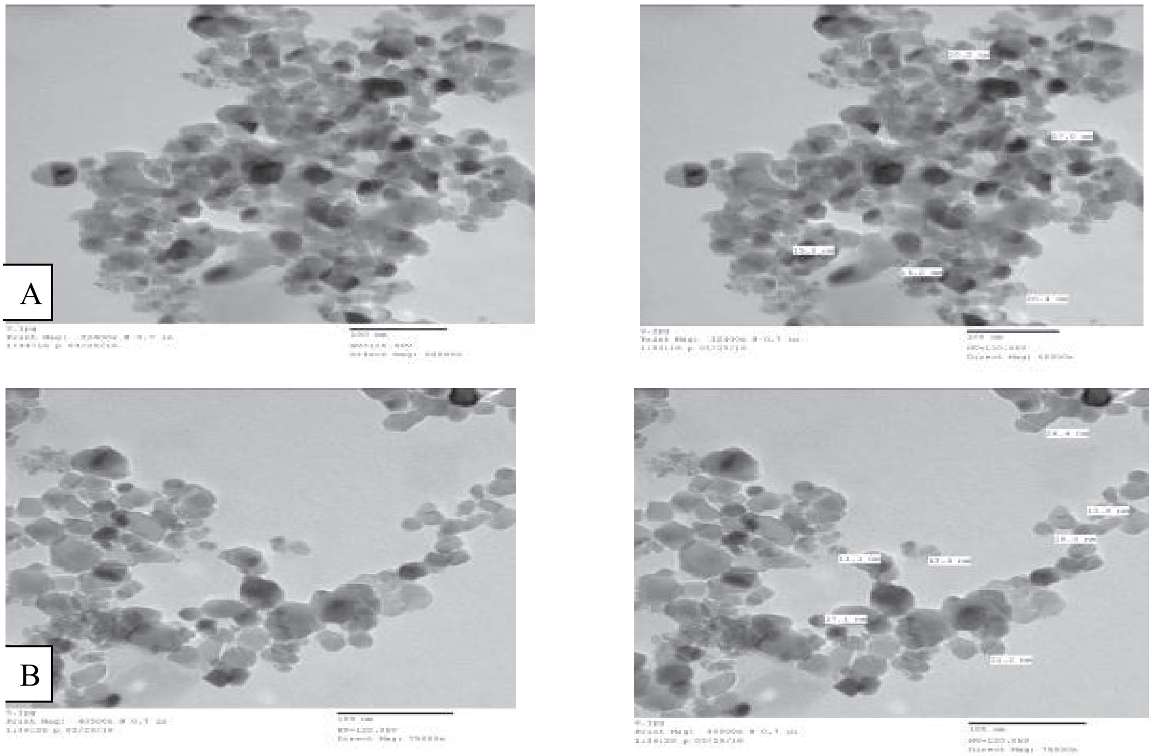


Fig. 1. TEM of TiO_2 Nanoparticles. (a) in the solution (b) in the cationic formula.

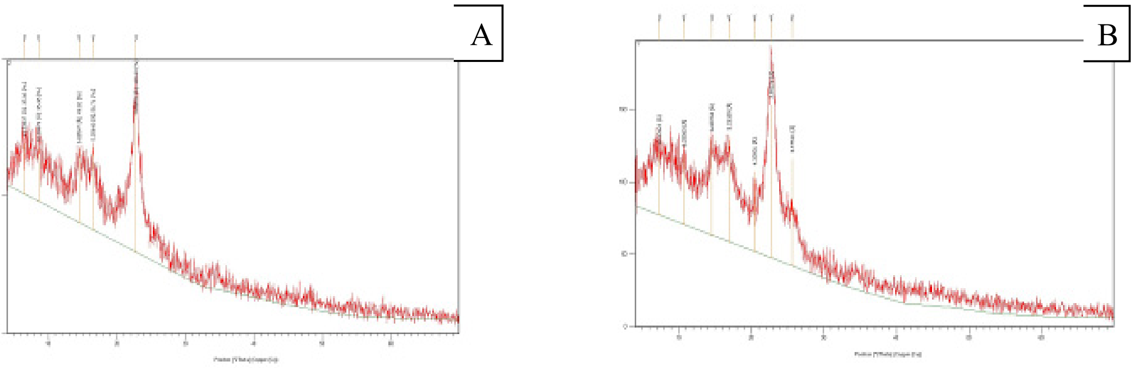


Fig. 2. X-ray diffraction patterns for treated samples. (A) blank cotton treated with cationic softener and TiO_2 nanoparticles and (B)CM-cotton with cationic softener and TiO_2..

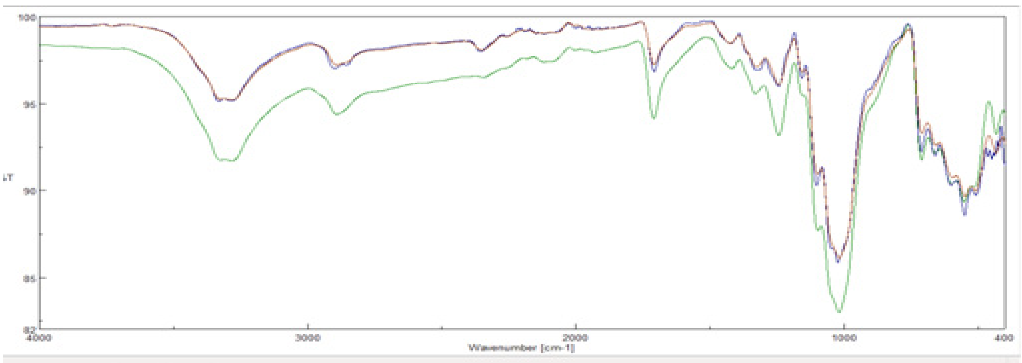


Fig. 3. FTIR for cationic softener applied on blank cotton in presence of TiO_2 nanoparticles Green CM-Cotton, Blue CM-Cotton , 5% Cationic softener., Red CM-Cotton +5% Cationic softener+ 1%TiO_2 nanoparticles.

UV protection

Ultraviolet protection factor (UPF)is one of the main indicators for fabric ability to prevent skin from sun burn. The higher the UPF value, the longer an individual can withstand the sun. Laundry test was carried out to evaluate durability of finishing treatments. The obtained data for our study is listed in Table1. The sample were characterized for the cotton fabric treated with cationic softener and TiO_2 nanoparticles in presence or absence of oil herbal the Data in table 1 shows the UPF values of the fabric samples formulation was applied on blank cotton and CM-cotton treated with the TiO_2 nanoparticles.

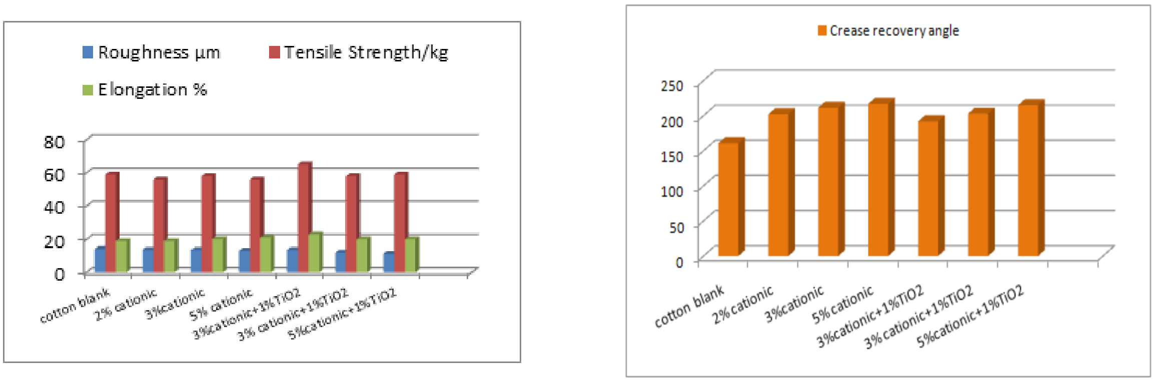


Fig. 4. Physical Properties for cationic softener cotton treated in presence and absence of TiO_2 Nanoparticles.

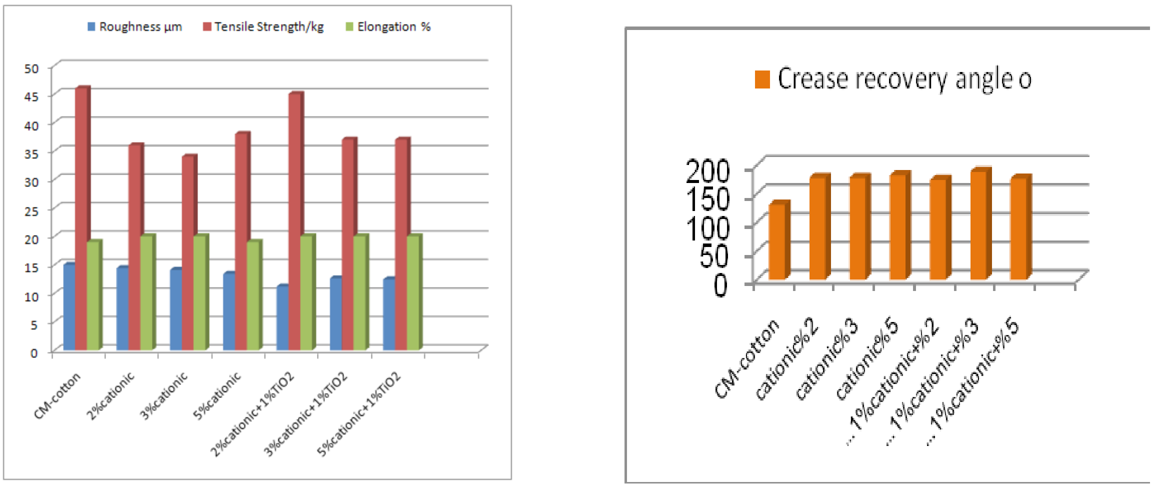


Fig. 5. Physical properties for cationic softener CM-Cotton treated in presence and absence of TiO_2 nanoparticles.

The results comprises that cationic softener with TiO_2 nanoparticles makes good UPF protection but by increasing cationic softener concentration the efficiency of UPF was decrease. These data show that UPF values for fabrics treated with cationic softener exhibited better ultra violet protection than for untreated ones.

The cationic softener on CM-cotton fabrics treated with titanium dioxide nanoparticles shows higher UPF values than for cationic softener with TiO_2 nanoparticles on cotton fabrics. Also the results indicate that the ultra violet blocking criteria for the cationic softener treated CM-cotton along with titanium dioxide nanoparticles increases significantly even at very low nanoparticle concentration.

Antibacterial activity

Antibacterial property of cotton and cotton CM-cotton fabrics treated with titanium dioxide nanoparticles in presence of different concentrations of cationic softener concentrations

TABLE 1. UPF Of treated cotton as well as treated CM-cotton fabric .

UV Protection	UPF	Substrate
moderate	11	blank cotton
Very good	37.18	cotton treated with 1% Ti nano 2% cationic softener
Very good	33.21	cotton treated with 1% Ti nano 3% cationic softener
good	27.68	cotton treated with 1% Ti nano 5% cationic softener
moderate	20.17	5 CM- cotton
excellent	47.75	6 CM- cotton treated with 1% Ti nano 2% cationic softener
excellent	43.04	7 CM-cotton treated with 1% Ti nano 3% cationic softener
Very good	41.19	8 CM- cotton treated with 1% Ti nano 5% cationic softener

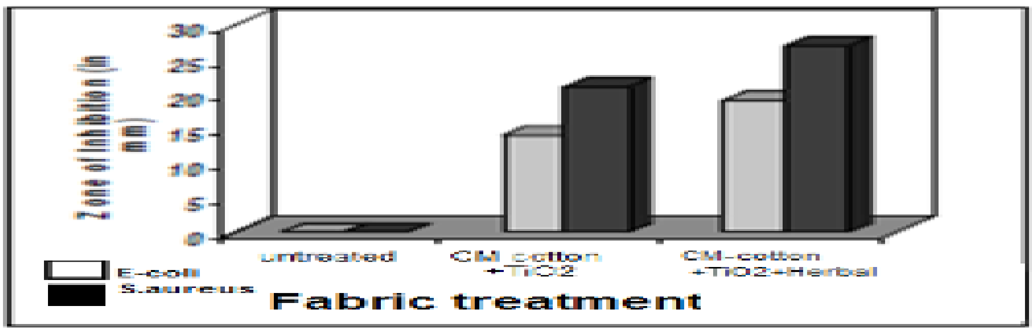


Fig. 6. Antibacterial Effect of Treated Cotton and treated CM-cotton.

(2, 3, and 5%) in presence and absence of herbal oil were evaluated. Results in figure 6 disclosed that cationic softener treated cotton fabric with TiO_2 nanoparticles and cationic softener treated CM-cotton treated with the TiO_2 nanoparticles give nearly the same results. The enhancement of the data was finding when the herbal oil involved in the formulation bath. The proposed mechanism of anti-bactericidal activity of photo catalysts may be attributed to the attack of reactive oxygen species such as superoxide anions and hydroxyl radicals upon direct contact of photo catalysts with cells. Very low concentration of titanium dioxide nanoparticles did not affect suspended cells significantly because of limited chances for contact. It was postulated that the mechanism of bacterial killing by TiO_2 photo catalyst is attributed to oxidizing reactions of the reactive oxygen species [21]. Therefore, TiO_2 nanoparticles exhibited greater antibacterial activity as they produced large amount of hydroxyl radicals. This postulation explains the obtained result in Table 2. As treatment of cationic softener formulation either on the cotton fabric or CM-cotton, did not show any significant antibacterial effect, with the presence of TiO_2 nanoparticles in presence or absence of herbal oil increasing concentration show promising enhancement in the anti-bacterial criteria for treated fabrics. As indicated in Table 2, Gram-negative bacteria are less sensitive than Gram-positive bacteria for treated cotton fabric with TiO_2.

Self cleaning cotton

Photocatalytic decomposition of methylene blue by TiO_2 nanoparticles.

Photocatalytic properties for TiO_2/cationic softener finishing formulation and cotton fabrics treated with TiO_2 /cationic softener were evaluated through analysis of the decrease in methylene blue colorant concentration following exposure to ultraviolet radiation. The process of photo catalysis involves many possible reactions [12]:a) Semiconductor surfaces photo excitation triggers electron–hole pair generation. b) Some of these pairs recombine again while the remaining holes accelerate oxidation reactions through creation of hydroxyl radicals. c) Oxygen accepts electrons and reduced to superoxide that ultimately leads to the generation of hydrogen peroxide which is a powerful oxidizing agent. All these generated oxidizing agent participate in the decolorization of the methylene blue dye (organic pollutants) and summarized as follow:

$$TiO_2 + h\nu \rightarrow e^- + h_{vb}$$
$$O_2 + e \rightarrow O_2^-$$
$$O_2^- + H^+ \rightarrow {}^{\cdot}OOH$$
$$2\,{}^{\cdot}OOH \rightarrow H_2O_2 + O_2$$
$$H_2O_2 + e \rightarrow OH^{\cdot} + OH^-$$
$$H_2O + h_{vb} \rightarrow H^+ + OH^{\cdot}$$

Photocatalytic activity of cotton and CM-cotton fabrics treated with TiO_2 nanoparticles in presence of different concentrations of cationic softener concentrations (2, 3, and 5%) was assessed through methylene blue fabric self cleaning. The self-cleaning process occurs on fabric surface upon UV radiation as shown in Fig. 7. UV energy activates photo catalytic discoloration of dye stains by TiO_2 particles through alteration of molecular configuration of methylene blue stains into a colorless dye. The self-cleaning criteria for treated CM-cotton with TiO_2 are higher than that for TiO_2 cotton samples as shown in Fig. 7b. This might be attributed to more accessibility for stain adsorption on photo catalyst which enhances interactions between methylene blue stain and active species produced by TiO_2. These parameters play a significant role in improving self-cleaning property on titanium dioxide treated cotton samples. TiO_2 nanoparticles, as mentioned earlier on, produce highly reactive molecules that react with dye molecules causing their discoloration.

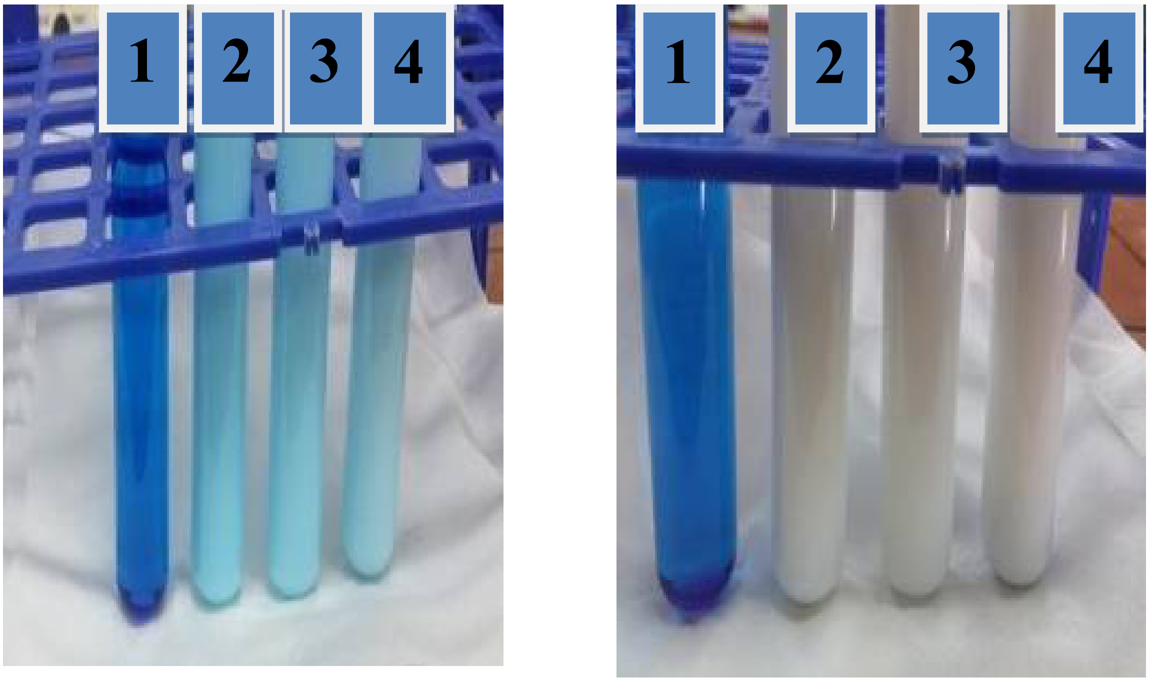


Fig. 7a. Decolorization of MB Dye in finishing formulations contain TiO_2 nanoparticles in presence of different concentrations of cationic softener concentrations where (1) 20mg/L MB (2) 10 ml of 20mg/L MB with 40 ml of (2% huntsman Cationic softener/1% Ti nano) (3) 10 ml of 20mg/L MB with 40 ml of (3% huntsman Cationic softener/1% Ti nano (4) 10 ml of 20mg/L MB with 40 ml of (5% huntsman Cationic softener/1% Ti nano).

After about an half hour color of MB dye disappeared from the solution

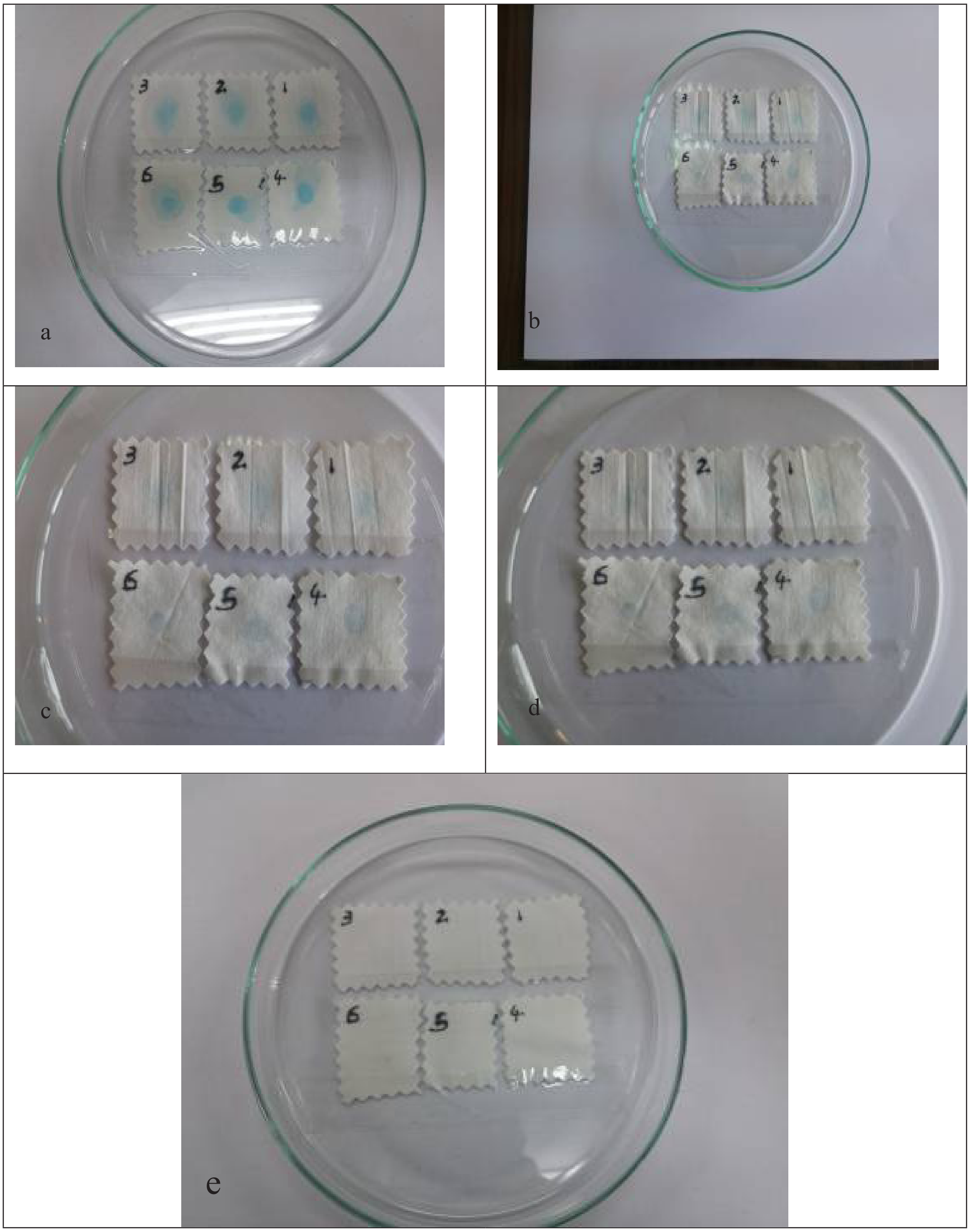


Fig. 7b . Decolorization of MB dye on cotton and cm cotton fabrics surface where .

(1)Blank cotton treated with 1% Ti nano 2% huntsman cationic softener , (2)Blank cotton treated with 1% Ti nano 3% huntsman cationic softener (3)Blank cotton treated with 1% Ti nano 5% huntsman cationic softener (4) CM-cotton treated with 1% Ti nano 2% huntsman cationic softener (5)CM- cotton treated with 1% Ti nano 3% huntsman cationic softener (6)CM- cotton treated with 1% Ti nano 5% huntsman cationic softener .

While a,b,c ,d,e indicate time of exposure to sun light 0, 1,2,3,4 hrs respectively.

SEM – EDX analysis of treated fabric

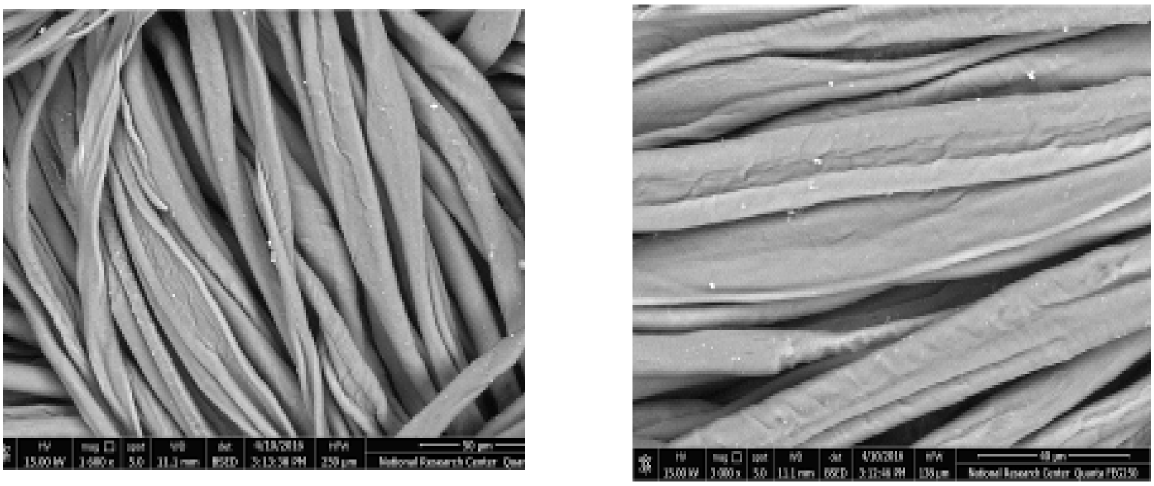


Fig. 8 A. 5% Hunstman Cationic Softener on Cotton Blank.

SEM – EDX analysis of treated fabric

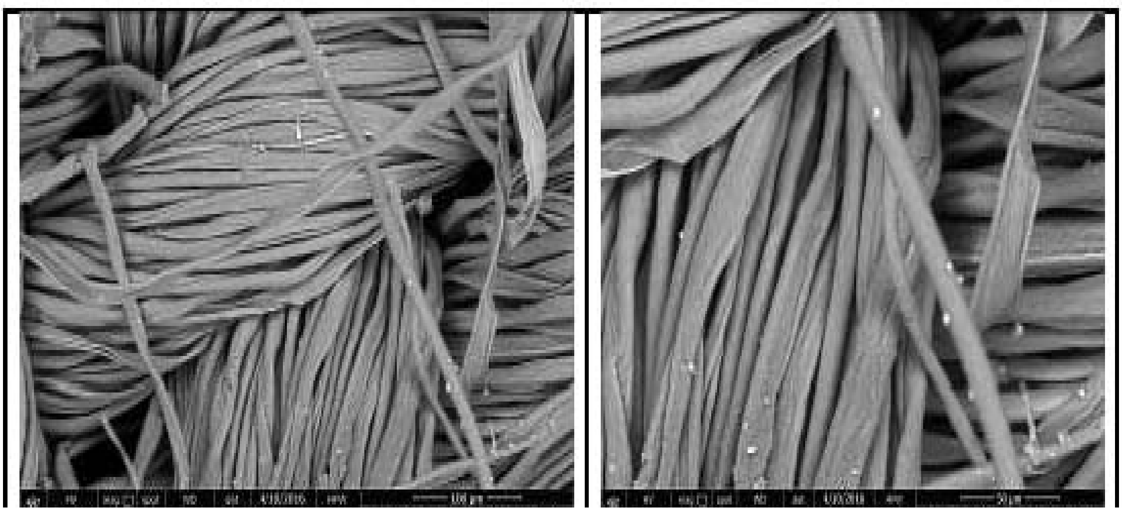
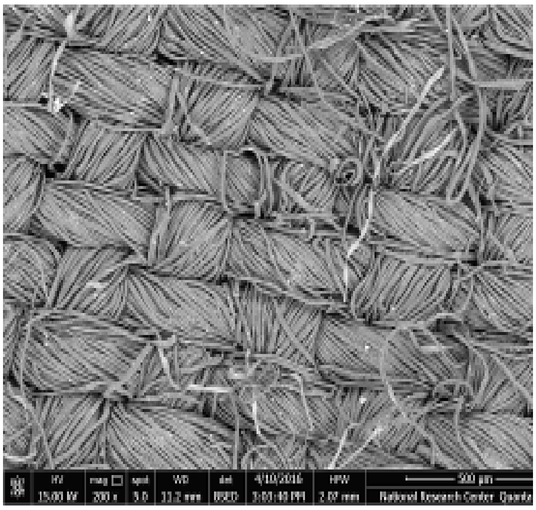
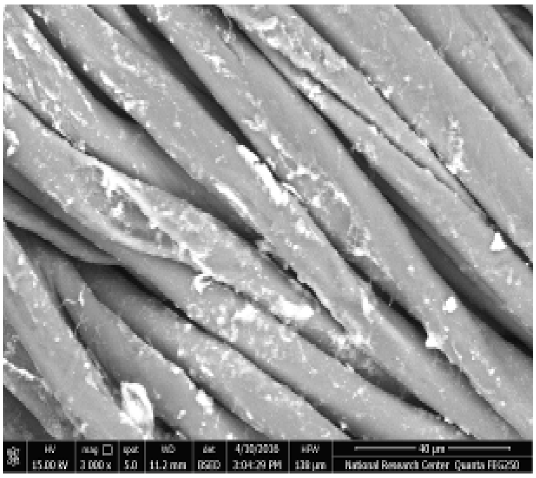
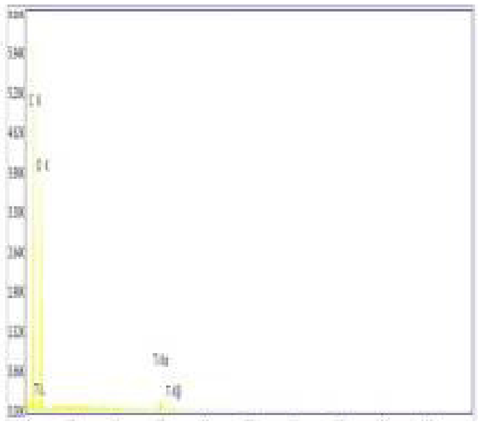


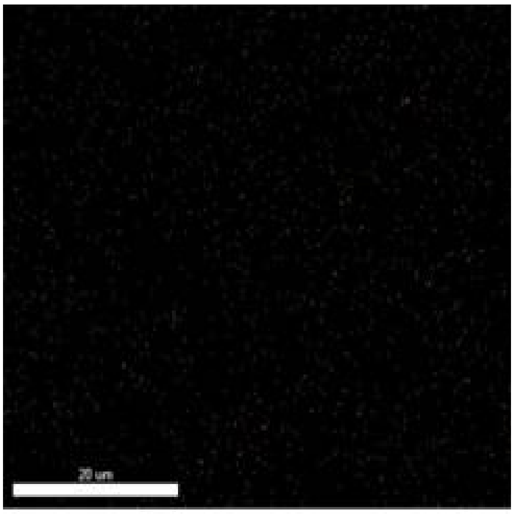
Fig. 8 B. 5% Hunstman Cationic Softener on CM-cotton.

For the characterization of cationic softener treated cotton or CM-cotton in presence of TiO_2 nanoparticles by using SEM found in Fig. 8A and B change in appearance of the cotton fiber after TiO_2 finishing; the cotton fiber surface was covered with TiO_2 particles and seemed to be somewhat rough and uneven. Therefore, we proved that the Ti particles were loaded on the cotton fiber with the pad–dry–cure process, but their distribution on the fiber surface was not even, possibly because of the aggregation of some fine TiO_2 particles.



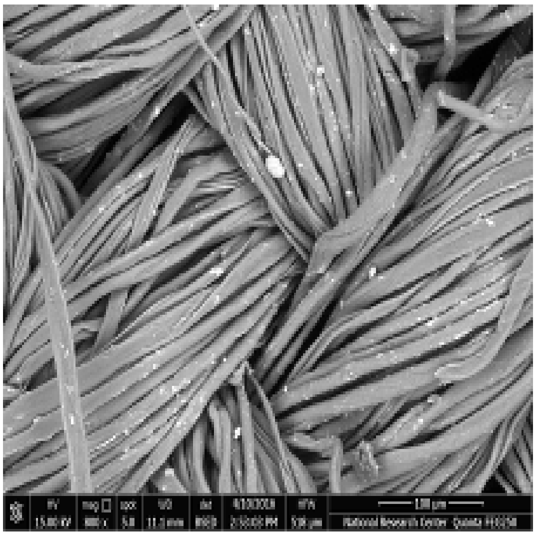


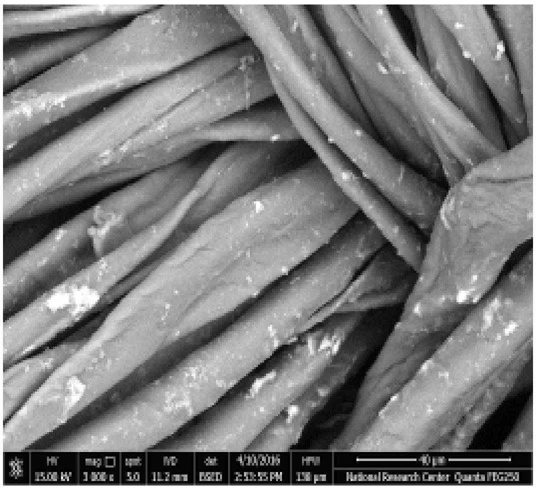


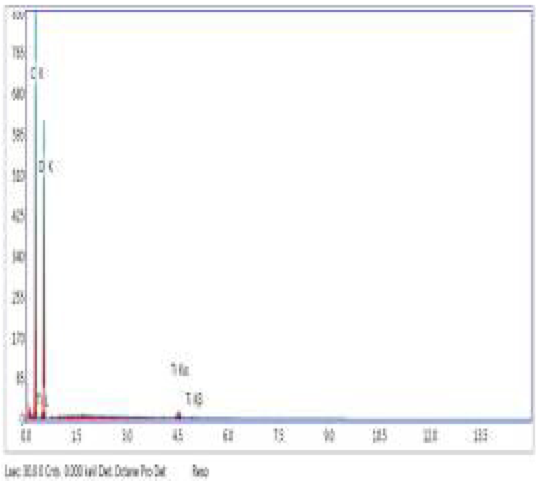


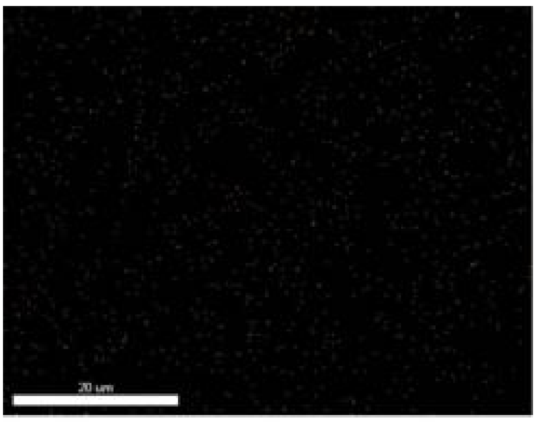
Element	Weight %	Atomic%	Net Int.	Error %
C K	50.27	57.69	130.12	6.66
O K	48.8	42.04	94.56	10.98
TiK	0.93	0.27	6.24	21.45

Fig. 9 A. 1%TiO_2 & 5%Hunstman cationic softener applied on cotton Blank.









Element	weight %	Atomic %	Net Int. Int.	Error %
C K	49.49	57.01	90.7	0.01
O K	49.31	42.64	68.6	0.01
TiK	1.2	0.35	5.8	0.07

Fig.9 B. 1%TiO_2 & 5%Hunstman cationic softener applied on CM-cotton.

Conclusions

The following conclusions can be drawn based on the study carried out on the cationic softener treated cotton and CM-cotton fabrics in presence of TiO_2 nanoparticles in presence and absence of herbal oil. The physical properties of the treated fabric were not threatened by this formulation in addition to the functionality of the treated fabric like improvement of UV protection and self cleaning properties in-addition to antibacterial properties. Beside this discussion the study of stability of nano particles in the multifunctional hybrid formulation was done and improved by the TEM analysis in addition to antibacterial.

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تطوير و تقييم هجين تجهيزى جديد متعدد الوظائف محتوى على(مادة تنعيم كاتيونية -ثانى اكسيد التيتانيوم-زيت عطري) واستخدامه لتجهيز الأقمشة القطنية

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يهدف هذا البحث الى تخليق مواصفه تجهيزيه جديدة باستخدام ثانى اكسيد التيتانيوم النانومترى و استخدام هذه المواصفة فى التجهيز المتعدد للاقشمة القطنية.تم دراسة خواص الاقمشة القطنية المعالجة بالمواصفة الجديدة. حيث تم دراسة تأثير تركيز مادة التنعيم الكتيونية , نوع الأقمشة القطنية المستخدمه (قطن مبيض-الكربوكسى ميثيل قطن), نوع ثان اكسيد التيتانيوم النانومترى(خليط من نوعى الروتيل و الانتاز) –و كذلك تأثير وجود الصبار كزيت عطري على خواص الأقمشة القطنية المعالجة.و كان ثبات ثانى اكسيد التيتانيوم فى الصورة النانومترية فى هجين التجهيز احد عناصر الدراسة و من ناحيه اخري تم دراسة تأثير وجود مواد صديقة للبيئه مثل الزيت العطري (الصبار) علي مقاومة الأقمشة المعالجة للبكتريا. تم التحكم فى الأقمشة المعالجة بدراسة الخواص المكتسبة عن طريقة المعالجة الجديدة و هى خاصية الحمايه ضد اشعة الشمس التنظيف الذاتى و مقاومة البكتيريا. اهتم البحث ايضا بتوصيف اكسيد التيتانيوم النانومترى باستخدام جهازى لTEM و XRD.

تم تقييم خاصية مقاومة البكتريا للأقمشة المعالجة ضد نوعين من البكتريا السالبة و الموجبة

حيث اظهرت النتائج تأثير ملحوظة مقاوم للبكتريا للأقمشة المعالجة ضد

Staphylococcus aureus (gram-positive) strains and Escherichia coli (gram-negative)

و قد اظهرت المعالجة تحسن فى نعومة الملمس للأقمشة المعالجة بالاضافة الى الخواص الاخرى المتعددة مثل الحماية ضد اشعه الشمس و التنظيف الذاتى و مقاومة البكتريا.