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Sol-gel Hybrid Nanomaterials Based on TiO_2/SiO_2 as Multifunctional Finishing for Cotton Fabric

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THIS work principally manages a novel multifunctional covering for giving, fire resistant, UV-protection and water repellent properties for cotton fabrics utilizing TiO_2 and TiO_2/SiO_2 nano-particles. We show the preparation and the application of TiO_2 and TiO_2/SiO_2 nanomaterials. The utilization of the nanomaterials on cottton fabric was accomplished by utilizing different polycarboxylic acids *i.e.* (Citric acid [CA], Succinic acid [SA], and 1,2,3,4-Butanetetracarboxylic acids [BTCA]) in the presence of sodium hypophosphite (SHP) which used as catalyst using traditional Pad-dry-cure technique. The impacts of carboxylic group number in the different polycarboxylic acids on the UV-protection and physical properties, of cross-linked cotton fabrics are researched. Some mechanical properties of coated cotton fabric (whiteness, roughness, and Crease recovery angle [CRA]) were analyzed. In addition, the impact of post treatment with various silane derivatives based on tetraethylorthosilicate (TEOS)/or 3-glycidyloxypropyltrimethoxysilane (GPTMS) on thermal stability of treated fabrics and additionally the water repellent property was also examined.

The expanding interest for multifunctional fabric materials requires a strong multidisciplinary approach and additionally the merging of conventional scientific disciplines [(1-3)]. The main nanofinishes application is found in use materials as nanoparticles through finishing procedures. However, these finishes don't withstand consequent washing because of low fixation of the nanoparticles on the substrate surface. Guaranteeing enhanced holding of nanoparticles to substrate surfaces increases the durability property, as well as the ecofriendly consideration of preventing release of loosely-bound nanoparticles into the environment [(4)]. The utilization of TiO_2 nano or other nano materials as catalysts have been researched for a long time to upgrade the catalytic impact on the improvement of many properties [(5-7)]. Finishing of textiles with TiO_2 nano as a catalyst to improve the crease recovery property has been one of the present topics [(8,9)]. For getting durable finished cotton, different poly-carboxylic acids were researched as zero-formaldehyde crosslinkers [(10-15)]. There are many advantages of these compounds such as ester linkages formation, free formaldehyde, abrasion strength and improved tensile [(16)]. Polycarboxylic acids, for example, 1,2,3,4-butanetetracarboxylic acid and citric acid are environment friendly crosslinker for cotton and can be good substitutes for methylols which contain crosslinkers [17-20]. Many researchers utilized different carboxylic acids, named, maleic acid (MA), butanetetracarboxylic acid (BTCA), succinic acid (SA), and citric acid (CA) as crosslinking agents for cellulosic textile treatment using TiO_2 nano as a catalyst under UV light [21]. The presence of SiO2 together with TiO2 has attracted much attention to a new class of material that has been broadly Investigated for an extensive variety of utilizations; a wide application of SiO2 chemical reaction that improved mechanical and thermal properties, with protecting catalytic properties of TiO_2[22], protective coating layer on stainless steel against chemical attack and oxidation[23], antireflective coatings for optical glasses[24] low thermal expansion coefficients glass materials[25], polymer composites fillers for photonic crystals [26]. Because of the desired utilization of the last reaction, the Si–O–Ti framework can control the components order; to form Si–O–Ti framework or TiO_2/SiO_2 composites[27].

With the wide application fields of nanotechnology, it is possible to get the superhydrophobic surfaces by different procedures, for example, the sol gel technique[28]. Sol–gel innovation gives another approach to functionalize fabrics by enhancing their physical properties [29]. In textile materials, this strategy can impart mechanical, hydrophobic or oleophobic surface properties of the substrate.

Experimental

Material

Mill bleached 100% cotton fabric (230 g/m^2) were kindly supplied by Misr Company for spinning and weaving Mehalla El-Kobra, Egypt.

Chemicals

1,2,3,4-Butane tetracarboxylic acid (BTCA), succinic acid (SA), citric acid(CA) and sodium hypophosphite(SHP) were supplied by Merck chemical company (Germany). TiO_2 (P-25) was purchased from Degussa Co. Ltd.,Germany. SiO_2 nanopowder was obtained from Sigma Aldrich. Isopropanol was of laboratory grade chemicals. 3-Glycidyloxypropyl trimethoxysilane (GPTMS) (98%) was obtained from Aldrich Chemical Company Inc., Milwaukee; USA. Tetraethoxysilane (TEOS) (98%) was supplied from Fluka Chemie GmbH, Buchs, Switzerland.

Preparation of TiO_2/ SiO_2 nanomaterials

0.5% TiO_2 and 0.5% SiO_2 nanoparticles were resuspended in ethanol alcohol, put in ultrasonic for 30 min then start to treat the fabric.

Preparation of tetraethoxysilane (TEOS) sol

The TEOS sol solution was prepared in three steps by mixing 10 ml TEOS, 15 ml ethanol, two droplets of HCl and 19 ml purified water in a three-neck round-bottomed flask and reflux condenser. In the first step, ethanol was introduced slowly into the TEOS with vigorously stirring. In the second step, HCl was added into the purified water, and in the third step, water and HCl mixture was added slowly into the TEOS-ethanol mixture with vigorously stirring. After that, the TEOS sol was stirred at 60 ^oC for two hours and then cooled to room temperature.

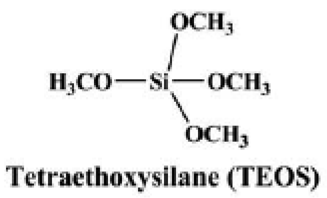


Fig. 1. Chemical structure of TEOS.

Preparation of Glycidyloxypropyltrimethoxysilan (GPTMS) sol

10 ml GPTMS (3-Glycidyloxypropyltrimethoxysilan) are dissolved in 100 ml isopropanol before hydrolysis using (1.22 ml) 0.01M hydrochloric acid. The resulting sol is stirred for at least 3h to form the basis sol (concentration of GPTMS 9,1 vol.%).

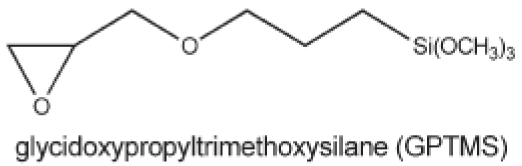


Fig. 2. Chemical structure of GPTMS

Preparation of TEOS/GPTMS sols

Firstly, GPTMS and TEOS were dissolved in proper ethanol, respectively, and then, they were mixed together and stirred vigorously for additional 12 hr to form the homogeneous solution through sol–gel process.

Treatment of fabrics with different poly carboxylic acids (crosslinking of fabrics)

Various aqueous solutions of butantetracarboxylic acid, citric acid and succinic acid (30 g/l) with sodium hypophosphite (4%) were prepared. Bleached cotton fabrics were padded in the previously prepared solutions in two dip and nip and then squeezed to a wet pick-up of 100%. Padded fabrics were dried at

80°C for 5 min and then cured at 130°C for 3 min. Treated fabrics were rinsed with hot water then with cold water and finally dried at room temperature.

Treatment of cotton fabrics with nanomaterials

Fabrics were coated with the nanoparticles and nanocomposite using pad-dry-cure method. Three suspended solutions of TiO_2 nanoparticles, SiO_2 nanoparticles and TiO_2/ SiO_2 nanomaterials were firstly prepared by dissolving of (0.5 wt %) of the nanoparticles in isopropanol for 20 min. fabrics pretreated with SA, CA or BTCA were immersed in suspended solutions of the nanoparticles or the nanomaterials for 10 min. All fabrics were padded in two dip and nip and then squeezed to a wet pick-up of 100%. Padded fabrics were dried at 80 °C for 10 min and then cured at 130 for 3 °C min. Cured fabrics were finally washed for 10 min in order to remove the nanoparticles having no bonding reaction with the fabrics.

Post treatment with TEOS/or GPTMS

TEOS/or GPTMS were used to lower the surface energy of the finished fabrics. The treated fabrics in preceeding step were immersed in TEOS/or GPTMS solutions for ten minutes before it was padded, and cured at 130 °C for 5 min.

Tests and analysis

Characterization of TiO_2/or SiO_2 nanomaterials

Transmission electron microscope (TEM): Particle shape and size were obtained with a JEOL-JEM-1200. Specimens for TEM measurements were prepared by dissolving a drop of colloid solution on a 400 mesh copper grid coated by an amorphous carbon film and evaporating the solvent in air at room temperature.

Characterization of treated fabrics

Fourier transform infrared (FTIR): Infrared spectroscopy of TiO_2 nanoparticles, SiO_2 nanoparticles and TiO_2/SiO_2 nanocomposites were carried out using Perkin Elmer FTIR spectrophotometer.

Scanning electron micrograph SEM/EDX analysis: SEM was studied using a scanning electron probe microanalyzer (JXA-840A) Japan. The specimens in the form of fabrics were mounted on the specimen stabs and coated with thin film of gold by the sputtering method. The micrographs were taken at magnification of 2000 using (KV) accelerating voltage.

UV protection factor

UV–vis spectrum was recorded on Perkin Elmer Lambda 3B UV-Vis spectrometer. Ultraviolet protection factor (UPF) was measured using UV Shimadzu 3101 PC-Spectrophotometer.

Mechanical properties of treated fabric

Fabric whiteness and yellowness: Whiteness and yellowness index were evaluated with a Color- Eye 3100 Spectrophotometer from SDL Inter.

Surface roughness: Surface roughness was monitored according to JIS 94 standard, using surface roughness measuring instrument, SE 1700α made in Japan.

Crease recovery angle: Standard method was used to measure wrinkle recovery angles WRA (AATCC test method 66-1984).

Thermal analysis (TGA)

Thermogravimetric analysis (TGA) was carried out using PerkinElmer TG-DTA analyzer, model Pyris1, operating under nitrogen atmosphere with initial sample weights of 8mg. The runs were performed over a temperature range of 50-600◦C at a heating rate of 10◦C/min under a continuous N2 flow of 100 ml/min.

Wettability measurements

The wettability test according to TEGAWA was applied[30], in this test the wettability was determined by recording the drop penetration time of the drop penetration test which was done by using an aqueous dye solution based on TEGEWA condition. Since a drop of certain volume (0.05 ml of 2% solution of the dye amino blue V-PW) from a height of 40 mm dropped on the treated sample then the penetration time was recorded as complete dipping of the drop.

Result & Discussion

The following are different carboxylic acid which used in this study, as shown in Fig. 3.

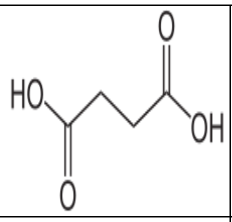
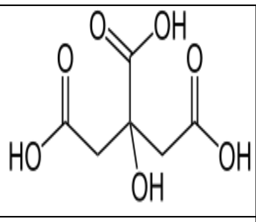
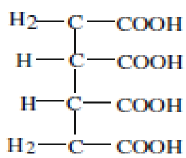
		
Succinic acid	**Citric acid**	**1,2,3,4 butanetetracarboxylic acid**

Fig. 3. polycaroxylic acids.

Tentative mechanism

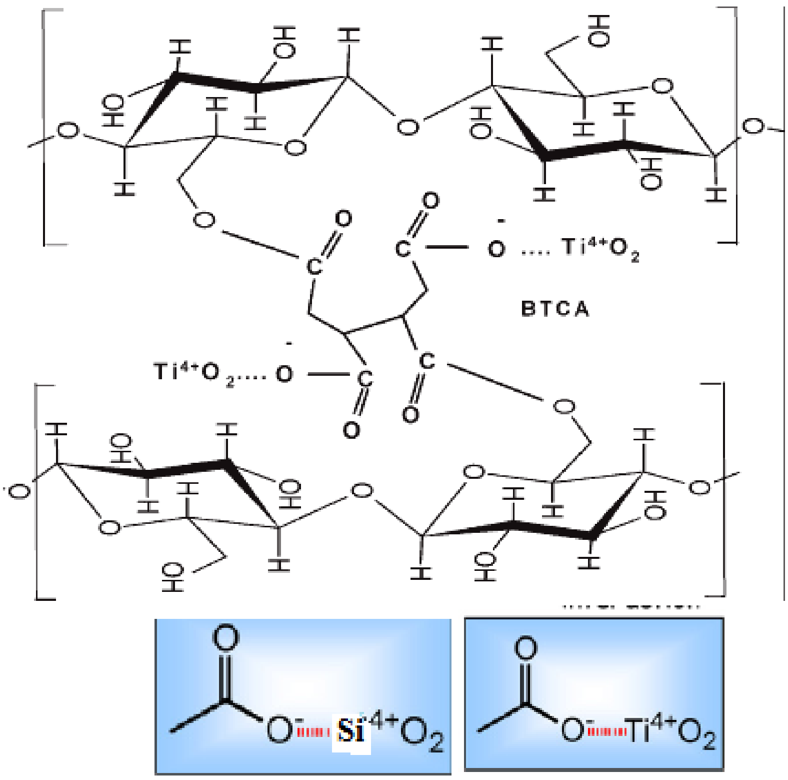


Fig. 4. Formation of ester linkages between BTCA and cellulosic chains and electrostatic linkages between BTCA and NTO.

As shown in figures 3&4, cotton fabric treated with 1,2,3,4-Butanetetracarboxylic acid (as an example for polycarboxylic acid) , are adequately advanced by nano TiO_2or nano SiO_2 and sodium hypophosphite. This is because of the ionic interaction between nanoparticle cation used and carboxylate anionic groups of the polycarboxylic acid.

Researchers reported that, there are many structures were formed in the mixture, such as precipitated titania-silica mixtures, isolated SiO^{4-}tetrahedra and exposed Ti^{4+}-terathedra [(31)].

GPTMS / TEOS sol-gel

An organic-inorganic hybrid sol was formed; since GPTMS ethoxy groups and TEOS methoxy groups are converting into hydroxyl groups through hydrolysis and condensation reactions leading to sol formation as shown in Fig.5.

The formed sol is then added to cotton fabric where hydrolysis and condensation takes place again. The Si-OH group of sol reacts with hydroxyl group of cotton fabric as well as Si-O-Si network formed through two Si-OH groups condensation reaction [(32)]. The coupling reaction between GPTMS and TEOS is mainly carried out via Si-O-Si bond [(33)].

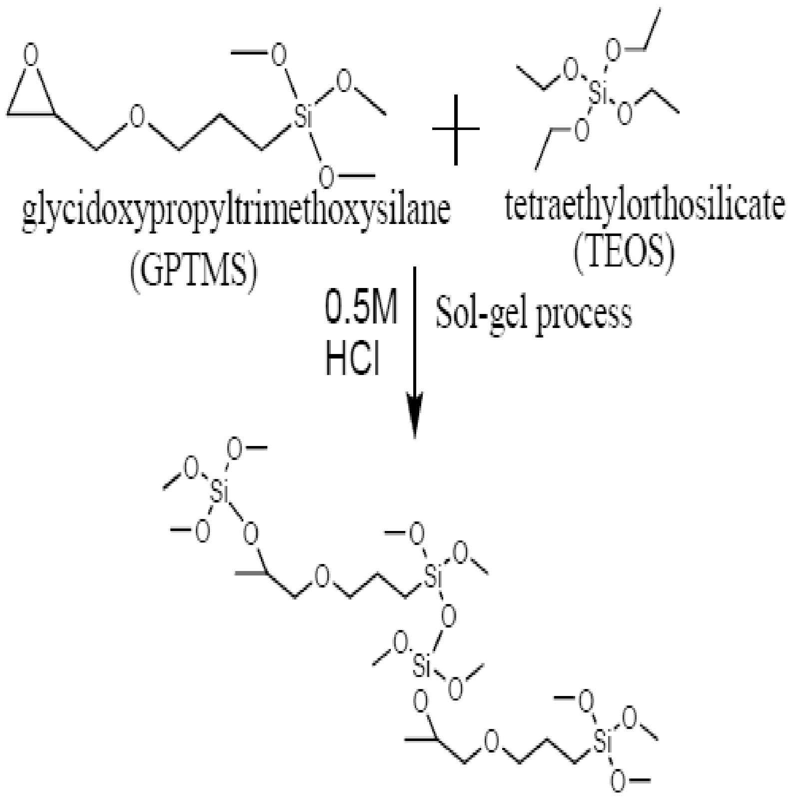
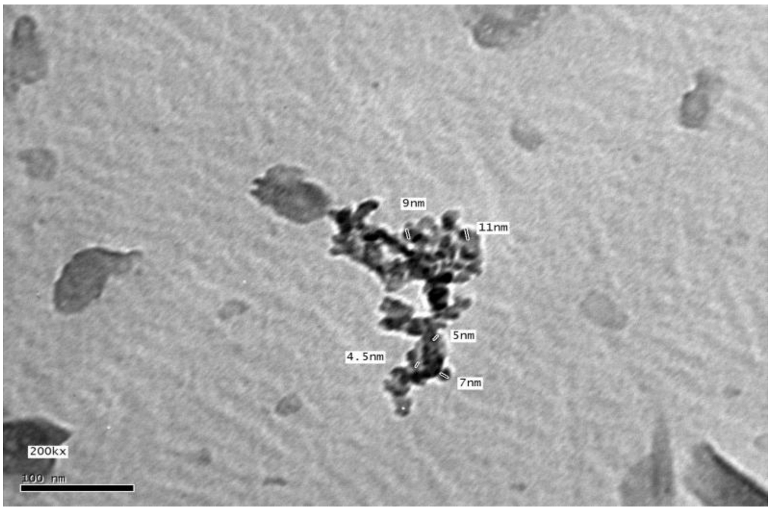


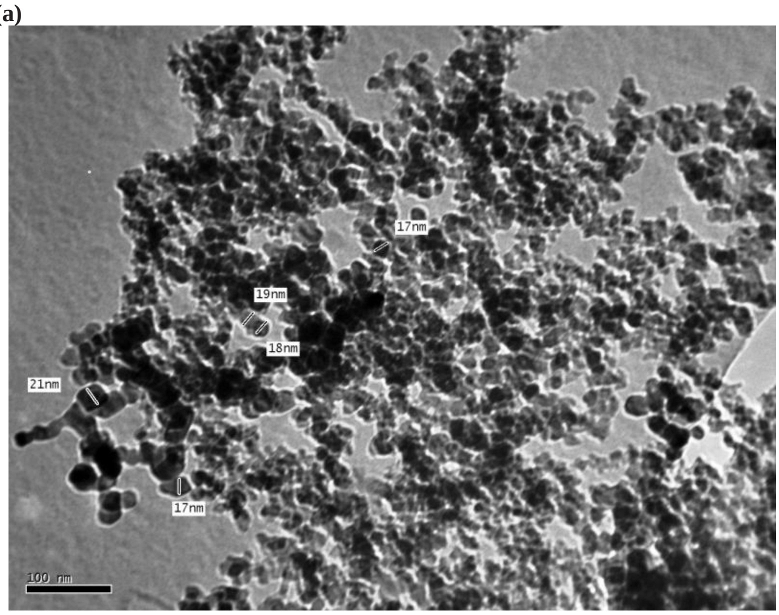
Fig. 5. Formation of GPTMS / TEOS sol.

TEM of TiO_2 nanoparticles solution and TiO_2/SiO_2 solution mixture

Figure 6 (a,b) shows the morphology of SiO_2 nanoparticles and TiO_2/SiO_2 nanomaterials separately. Figure 3a shows a homogeneous distribution of 5-11 nm particles size SiO_2. While figure 3b shows the morphology TiO_2/SiO_2 nanomaterials, it is clearly observed that TiO_2 and SiO_2 have different particles sizes. The particle size of SiO_2 is smaller than that of TiO_2 (17-27 nm), so SiO_2 nanoparticles can attach to the TiO_2 particles surface.



(a)



(b)

Fig. 6. (a) TEM image of SiO_2 nano-particles, (b) TEM image of TiO_2/SiO_2 nanomaterials.

Modified fabrics Characterization

The major task of the following part is to evaluate the treated fabrics and to establish the appropriate finishing formulations as well as treatment condition of cotton containing fabric with TiO_2 nanoparticles and its mixture with SiO_2 nanoparticles.

Easy care/ Anti UV functional finishing

To consider the durability of the finishing, laundry test were done. All cotton samples were washed at 40 °C, for 20 min, before the UV transmission was measured. Table 1 shows the changing in the absorption properties which expressed as UPF value of treated samples related to untreated fabrics.

Data in Table 1 indicate that treatment of cotton fabric with nano TiO_2 Particles and nano TiO_2/SiO_2 particles using different acids results in remarkable improvements in UV protection properties of the treated fabrics. Extent of enhancement in UPF value as reflection for the UV protection properties is affected not only by the nanoparticle used but also with the type of carboxylic acid used for fixation of nanoparticles.

TABLE 1. Effect of TiO_2/ or SiO_2 nanomaterials on UV- protection of fabrics treated by polycarboxylic acids.

Cotton samples	UPF value	UV Protection	
Untreated	10	10	moderate
Treated with TiO_2 nanoparticles, CA	23	20	good
Treated with TiO_2\SiO_2 nanomaterials, CA	27	25	good
Treated with TiO_2 nanoparticles, SA	15	15	moderate
Treated with TiO_2\SiO_2 nanomaterials, SA	23	20	good
Treated with TiO_2nanoparticles, BTCA	33	30	good
Treated with TiO_2\SiO_2 nanomaterials, BTCA	35	35	Very good

Data in Table 1 disclosed that treatment of cotton fabric by different carboxylic acid whatever was the nanoparticles used enhances the UV protection character of the treated fabric follow the order BTCA>CA>SA. This can be clarified by the higher carboxylic acid groups of BTCA which able to keep up more TiO_2 nanoparticles. We could also notice that treatment of fabrics with mixture of TiO_2/SiO_2 improve with great extent the UPF of the treated fabrics. This leads us to conclude that combination of SiO_2 with titania (TiO_2) leads to better UV protection.

Easy care (Performance) properties of treated cotton fabrics

Performance properties of the treated samples are summarized in Table 2.

Substrate	Roughness	Whiteness Index	CRA
Untreated	17.42	64.01	169
Treated with TiO_2nanoparticles, SA	18.62	53.43	194
Treated with SiO_2 nanoparticles,	18.93	50.23	183
Treated with TiO_2\SiO_2 nanomaterials, SA	19.35	51.38	189
Treated with TiO_2 nanoparticles, CA	18.37	54.63	220
Treated with SiO_2 nanoparticles, CA	18.45	54.7	206
Treated with TiO_2\SiO_2 nanomaterials, CA	19.82	52.28	216
Treated with TiO_2 nanoparticles, BTCA	20.36	54.12	255
Treated with SiO_2 nanoparticles, BTCA	21.34	54.38	235
Treated with TiO_2\SiO_2 nanomaterials, BTCA	21.98	53.52	250

Table 2 shows that, cotton samples finished with BTCA have better CRA when compared to those finished with CA and SA. This is because of the formation two cyclic anhydride intermediates at the same time through BTCA which has four carboxylic groups. Subsequently, four carboxylic groups of BTCA allowed ester bonds more readily. Furthermore, crosslinking of cotton fabrics with 1,2,3,4-butanetetracarboxylic acid (BTCA) are advanced by sodium hypophosphite and TiO_2 nanoparticles. The data also revealed that Pretreatment of samples with SA as crosslinking agent is accompanied by decrease in whiteness index than using the other two polycarboxylic acid CA and BTCA for crosslinking of nanoparticles and cotton fabric. But, treatment of cotton fabrics by nanoparticles is accompanied by increasing in roughness values and decrease in whiteness index, but using BTCA as crosslinking agent causing more increase in roughness value due to presence of more carboxylic group causing higher deposition of nanoparticles on fabric.

We can conclude from the data of UV protection properties as well as physical characteristics of the finished fabrics that, the appropriate finishing formulation to treat the cotton fabric with nanoparticles contain BTCA/SHP as ecofriendly finishing agent therefore the chemical composition of fabric treated with BTCA and different nanoparticles were investigated later.

Imparting thermal stability/hydrophobic properties to easy care finished fabric

The following two tasks deals with the effect of post treatment with different silicon sol on the thermal stability as well as hydrophobic properties of cotton treated fabrics.

Effect of post treatment with silicon compounds on thermal stability of treated fabric

Thermal stability of fabric treated with the nanoparticles as well as post treated samples, can be evaluated by TGA analysis as shown in Fig. 7-11.

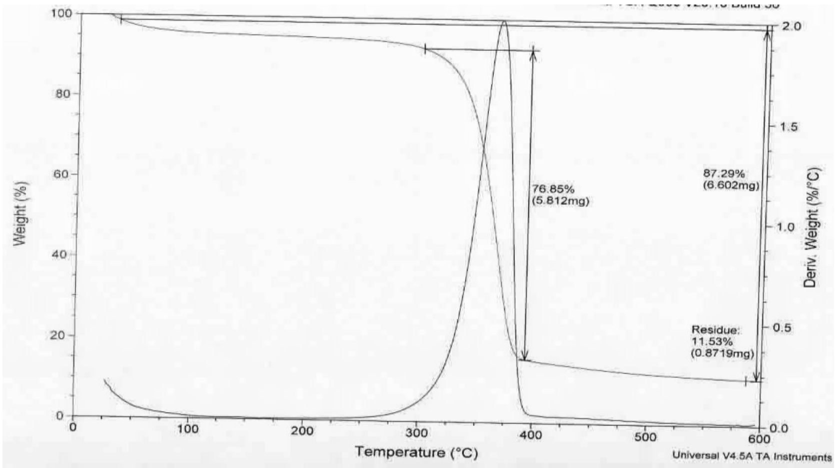


Fig.7. TGA curve of untreated cotton fabric.

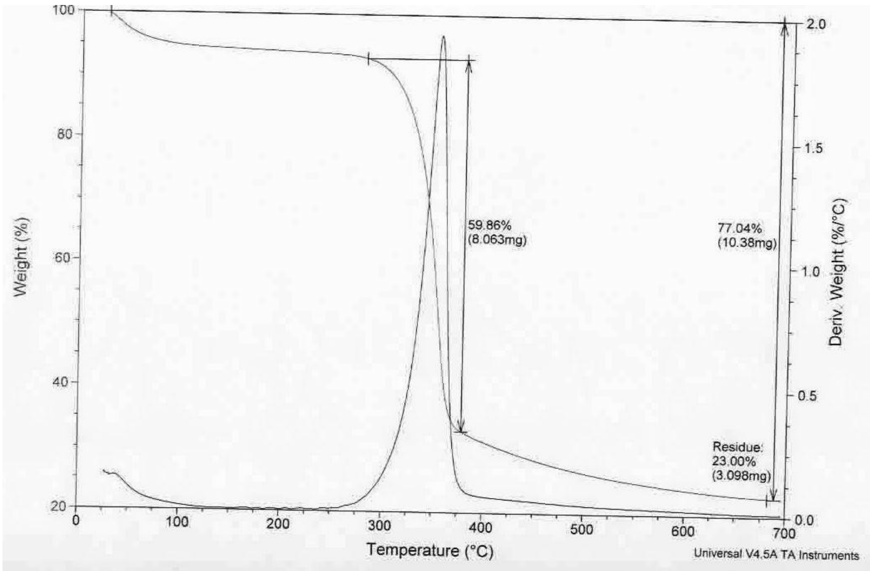


Fig. 8. TGA curve of cotton treated with SiO_2 nanoparticles post treated with TEOS.

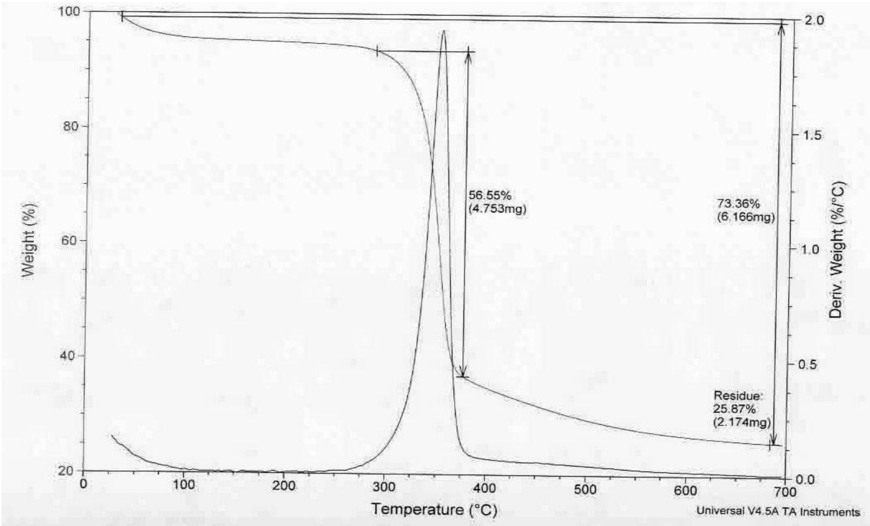


Fig. 9. TGA curve of cotton treated with SiO_2 nanoparticles post treated withT/G Where T/G TEOS/GPTMS.

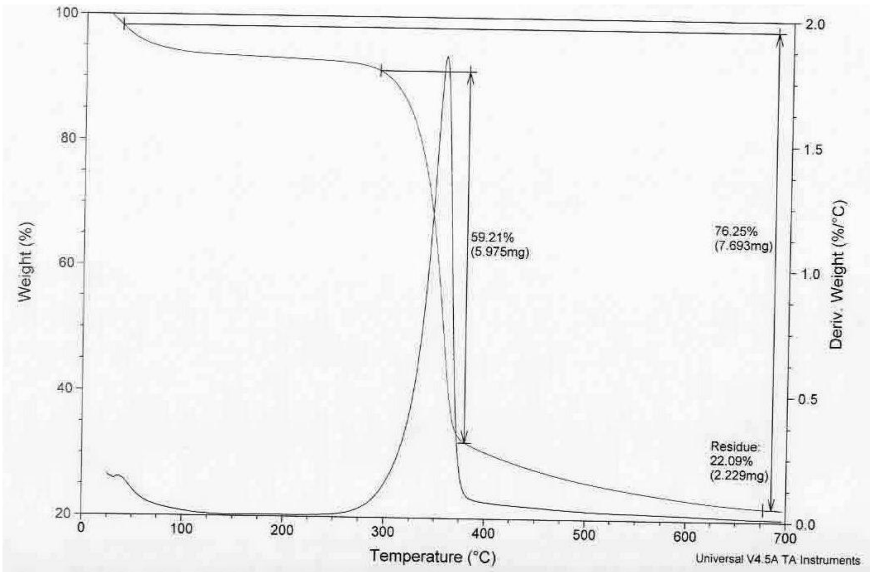


Fig. 10. TGA curve of cotton treated with TiO2/SiO2 nanoparticles post treated with T Where T TEOS.

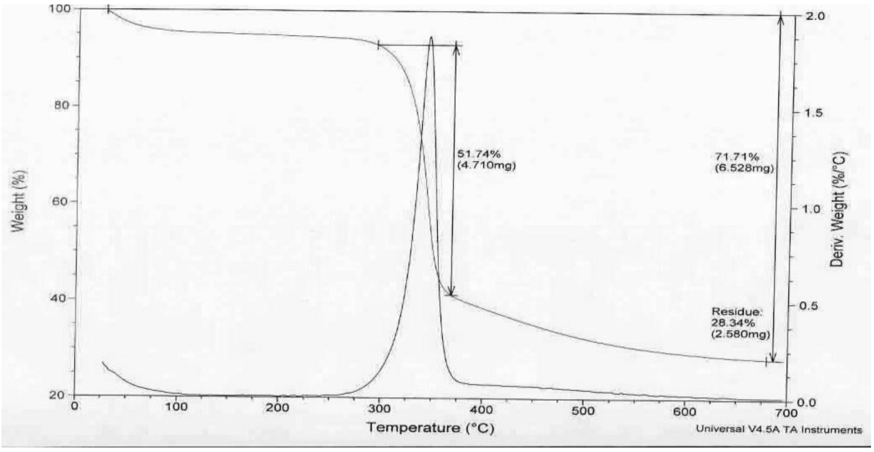


Fig. 11. TGA curve of cotton treated with TiO_2/SiO_2 nanoparticles post treated with T/G Where TEOS/GPTMS.

As we can see from data in Fig. 7-11 and Table 3, treatment of cotton fabric with SiO_2 or its mixture lower the decomposition temperature of treated samples, favour the formation of a higher char amount with respect to untreated cotton, as indicated by increase the residual content and this is highly observed in case of cotton treated with SiO_2 and TiO_2/SiO_2 mixture post treated with TEOS/GPTMS.

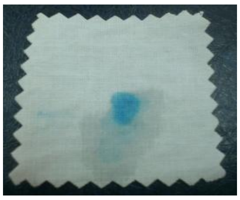
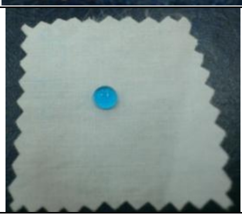
TABLE 3. Thermal decomposition of cotton treated fabric.

Samples	***Main decomposition***					***Residual content Wt%.***
	Decomposition temp.		**Remaining Weight %**		**Weight loss %**	
	T_0	T_∞	W_0	W_∞		
Cotton untreated	300	385	87.29	76.85	10.44	11.53at 600°C
Cotton treated with SiO_2/T	275	375	77.04	59.86	17.14	23 at 700°C
Cotton treated with SiO_2.T/G	275	363	73.36	56.55	16.81	25.87 at 700°C
Cotton treated with TiO_2/SiO_2/T	275	364	76.25	59.21	17.04	22.09 at 700°C
Cotton treated with TiO_2/SiO_2.T/G	275	364	71.71	51.74	19.97	28.34 at 700°C

Hydrophobic properties of post treated samples

Table 4 shows TEGEWA test which indicate the time of drop penetration for cotton treated fabrics compared with untreated samples.

TABLE 4. Drop penetration time test for cotton fabrics treated with different conditions.

Substrate	Drop penetration time [s]	Substrate after treatment
Untreated cotton	**0**	
Cotton treated with SiO_2 nanoparticles	**100s**	
Cotton treated with TiO_2/SiO_2 nanoparticles post treated with GPTMS	**>3400s**	
Cotton treated with TiO_2/ SiO_2 post treated with TEOS	**>3600s**	
Cotton treated with TiO_2/ SiO_2 post treated with TEOS/ GPTMS	**>3600s**	

Due to the hydrophilic properties of untreated cotton fabrics which attributed to the hydroxyl groups and the holes in its woven, it can be immediately wetted by the water droplet. When cotton fabric treated with silicon dioxide nanoparticles or its mixture with TiO_2 and post treated with TEOS, GPTMS and TEOS/GPTMS, this make the water droplet keep spherical and float on the cotton surface.

Increasing in hydrophobic property of the treated fabrics followed the order SiO_2 < TiO_2/SiO_2 post treated with GPTMS <TiO_2/SiO_2 post treated with TEOS< Cotton treated with /TiO_2/ SiO_2 post treated with TEOS/ GPTMS as shown in Table 4.

Surface chemical composition of treated fabric

The chemical compositions of the fabric loaded with different nanoparticles were studied by FTIR, SEM and EDX.

Fourier transform infrared (FTIR)

Figure 12 shows the FTIR data of TiO_2/SiO_2 (50:50) on fabric using KBr background. Since FTIR is used to indicate the established bonding between the oxides in the nanomaterials. In this case FTIR was utilized to affirm the presence of Si—O—Ti and Si—O—Si bonds. At 450 cm^{-1} there is a Peak due to the presence of Si—O—Si bond [34].Another peaks related to symmetric and asymmetric stretching vibrations of Si—O—Si bonds at 1100 cm^{-1} and 800 cm^{-1} respectively [34]. A little peak corresponding to Ti—O—Si linkages appeared at 960 cm−1 indicate the bonding of Ti and Si [35]. Around 3400 cm^{-1}there is a wide OH stretching peak of hydroxyl groups of the fiber can be seen.

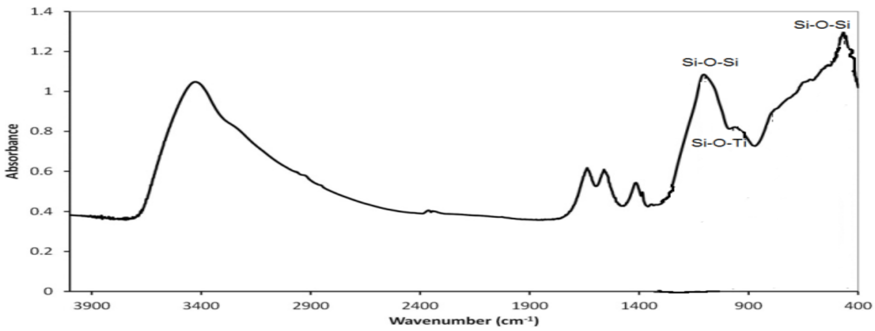


Fig. 12. FTIR spectrum of synthesized TiO_2/SiO_2 50:50 over 400–4000 cm^{-1}.

EDX/SEM investigation

SEM is used to indicate the topography changes of the treated and untreated fabric surface. Fig.13 (a) shows that the surface of the treated fabrics appears much smoother than that of untreated fabrics which is comparably rough, since the coatings process in case of treated fabrics leads to a flattening of the fabric. As a result, Fig. 13 (b) shows a uniform surface of cotton treated with TiO_2 nanoparticles. Figure 13 (C) shows the surface morphology of TiO_2/SiO_2 nano coating on cotton fabric; more deposition of nanoparticles on the surface of fiber was observed. Fig 13 (D) shows the surface morphology of TiO_2/SiO_2 nano coating on cotton fabric post treated with TOES/GPTMS.

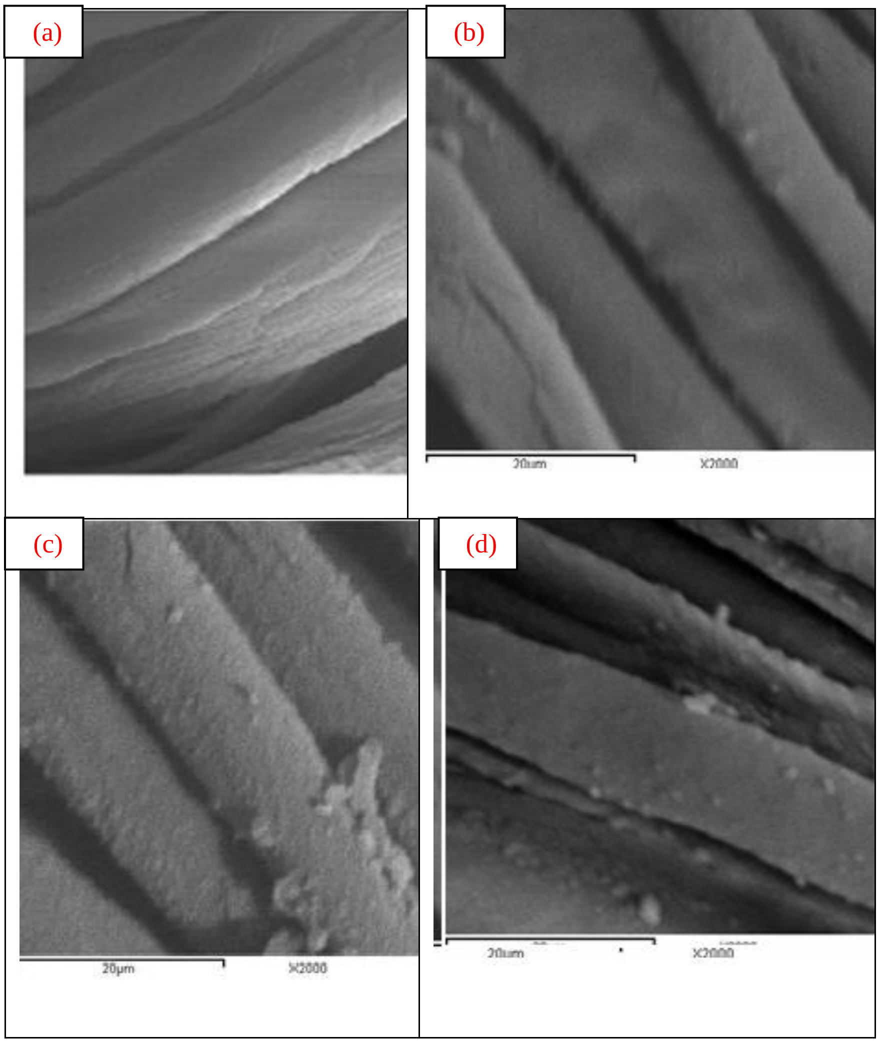
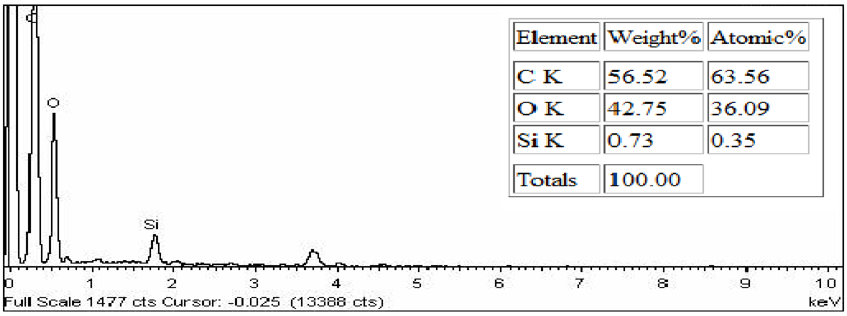
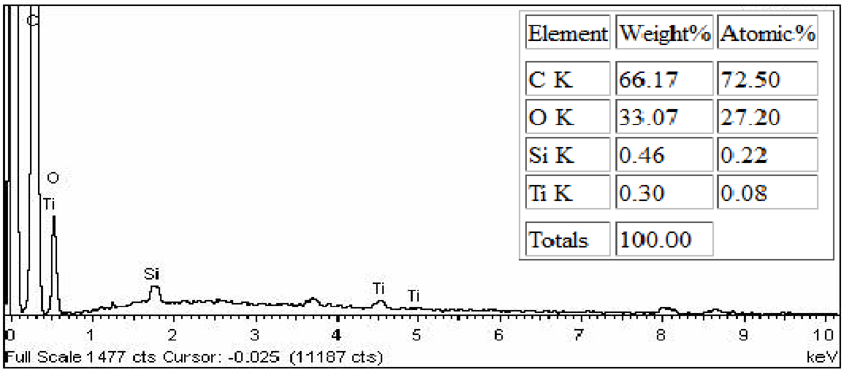


Fig. 13. SEM (a) untreated cotton fabric (b) cotton fabric treated with TiO_2 nanoparticles(c) cotton fabric treated with TiO_2/SiO_2 nanoparticles (d) superhydrophobic cotton fabric treated with TiO_2/SiO_2 sol post treated with TOES/GPTMS.

Chemical composition of the surface of cotton treated fabric treated with TiO_2 sol and TiO_2/SiO_2 sol are given in figure 14. It is clear that in addition to oxygen and carbon signals, Si elements signal is detected as shown in figure 14(a), while figure14 (b) shows signals for Ti as well as Si elements indicate the presence of these elements on the surface of treated fabrics.



(a)



(b)

Fig. 14. EDX images of (a) the SiO_2-loaded substrate, (b) TiO_2/SiO_2 -loaded substrate.

Conclusion

CA, SA and BTCA are environment-friendly compounds used for the finishing of cotton fabrics. In this work, the impact of different three polycarboxylic acid agents together with SHP as catalyst and TiO_2 nanoparticles, SiO_2 nanoparticles and TiO_2/SiO_2 nanomaterials as a novel multifunctional finish for cotton fabrics was investigated. In addition, the cotton fabrics were coated with sol-gel solution containing TEOS or GPTMS and mixture of them as precursors. The results demonstrate that, the treatment with BTCA improves the durable press performance, whereas the application with a TEOS/GPTMS– solution remarkably forms better nonflammable char residue and increment char formation after heating, and also improve hydrophobic functional properties.

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صول-جل المواد النانومترية الهجين المعتمدعلى جسيمات SiO_2/ TiO_2 النانومترية كتجهيز متعدد الاغراض للاقمشة القطنية

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يهدف هذا البحث إلي إستخدام النانو تكنولوجى كإتجاه حديث فى إضافة خصائص ووظائف متعددة للأقمشة القطنية مثل خصائص مقاومة الحرق والحماية ضد الاشعة فوق البنفسجية والتنظيف الذاتى ومقاومة الاقمشة للابتلال وكذلك مقاومة الأقمشة للبكتريا بإستخدام مواد نانومترية معتمدة على جسيمات TiO_2 أكسيد التيتانيوم او TiO_2/SiO_2 النانومترية. كما يهدف هذا البحث الى استخدام بعض الاحماض الكربوكسيلية لتثبيت جسيمات أكسيد التيتانيوم النانومترية $nanoTiO_2$ وخليط من جسيمات أكسيد التيتانيوم TiO_2 وأكسيد سيليكون SiO_2 على سطح الاقمشة بطريقة الغمر ثم العصر والتخزين على البارد.

- تم تثبيت جسيمات اكسيد التيتانيوم TiO_2 النانومترية وكذلك الخليط من جسيمات $TiO_2\backslash SiO_2$اكسيد التيتانيوم واكسيد السيليكون النانومترية على الاقمشة القطنية بإستخدام مادة رابطة صديقة للبيئة وهى حمض البيوتان تتراكربوكسيلك BTCAوحمض السكسينك SA فى وجود مادة مساعدة وهى صوديوم هيبوفوسفيت SHP وذلك بطريقة الغمر والتجفيف ثم التحميص.

- تم دراسة خصائص متعددة للاقمشة القطنية المعالجة بكلا المعالجة بالمواد النانومترية ومقارنتها بمثيلتها الغير معالجة، ودراسة تأثير إستخدام الأحماض المحتوية على اعداد مختلفة من مجموعة الكربوكسيل -COO.

- تضمنت الدراسة أيضا خاصية مقاومة الاقمشة القطنية للإبتلال ومقاومتها للحريق وذلك فى معالجة لاحقة للمعالجة بجسيمات النانو بإستخدام بعض البوليمرات الهجينة وهى TEOS و على GPTMS على حدة او خليط منهما معا.

- أثبتت الدراسة أن المعالجة الأولية للأقمشة القطنية بالمواد النانومترية تؤدى الى تحسن كبير فى الخواص التى تضفيها المواد النانومترية،حيث ان استخدام خليط من $TiO_2\backslash SiO_2$ يؤدى الى تحسن ملحوظ يفوق حالة استخدام جزيئات TiO_2 النانومترية . و أثبتت الدراسة ان حمض البيوتان تتراكربوكسيلكBTCA كان الافضل فى الاستخدام المتعدد الوظائف. وأظهرت النتائج ان استخدام خليط من TEOS و GPTMS ادى الى تحستن ملحوظ فى مقاومة القماش للحريق اضافة الى تحسن خاصية الحماية الذاتية .