

Vinyltrimethoxy Silane and Aminopropyl Triethoxysilane: Excellent Silane Coupling Agents for Cotton Fiber Functionalization

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Authors' contributions

This work was carried out in collaboration among all authors. All authors contributed equally, read and approved the final manuscript.

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ABSTRACT

Vinyltrimethoxy silane and aminopropyl triethoxysilane were used for surface functionalization to enhance the textile performance of cotton. The process was carried out in an ethanol-water medium, which accelerated the cross-linking reaction between fiber and silane monomers. The process was optimized carefully as a function of fiber weight gain. The optimized modification conditions were silane monomer concentration 600 and 300% (on weight of fiber), ethanol-water ratio 60:40 and 80:20, pH 3.5 and 5, reaction time 90 and 60 min at room temperature (30°C), for VTMS and APTES, respectively. The silane-modified cotton fiber showed improved tensile strength, water repellency, thermal stability and wrinkle recovery. These improvements are due to flexibility of the Si-O bond and the fiber matrix interfacial strength properties. Instrumental analyses, such as FTIR, TGA, DTG, SEM and EDX, were carried out successfully. FTIR was used to identify the Si-O-Si group of VTMS and the Si-O-CH₃ of APTES on modified cotton fibers, and TGA and DTG were

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used to evaluate the modification's effect on the thermal stability of the fiber. SEM was used to study the surface morphology after modification and EDX was used to measure the quantity of elemental silicon atoms on the fiber backbone. Finally, unmodified and silane-modified cotton fibers were dyed with two reactive dyes and the modified cotton fiber showed better ability to accept dye than that of the unmodified fiber.

Keywords: Cotton fiber; silane coupling agent; graft polymerization; functionalization.

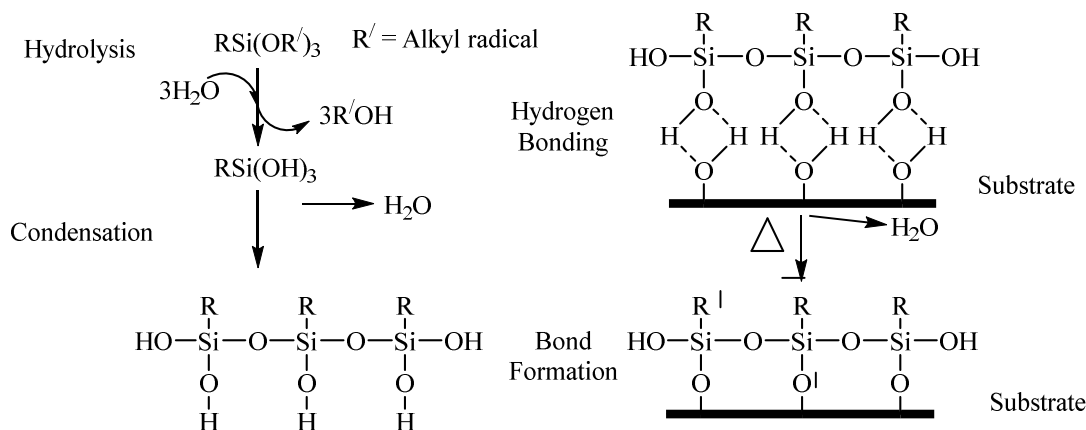
1. INTRODUCTION

Cotton is a widely used natural cellulose fiber, which provides a wide range of applications in textiles. Cotton presents the advantages of availability, low density, light weight, and, above all, it is environmentally-friendly [1,2]. Cotton contains mainly cellulose, hemicellulose, lignin and some other minor constituents [3]. Cotton with improved physical and chemical properties is in great demand in the textile industry. As modification of synthetic and natural polymers, via graft copolymerization, is a commonly-used method, there are several publications on the modification of cotton in the literature [4-7]. Therefore, chemical modification through condensation-polymerization of silane-coupling agents onto cotton fibers has been receiving considerable interest in recent years.

Physical properties of natural products can be improved by modifying them with silane monomers [8,9]. Among silane-coupling agents, vinyltrimethoxy silane (VTMS) and amino-propyltriethoxy silane (APTES) are most convenient for modification of cellulose fibers. The modification of cotton fiber by silane-

coupling agents, through a condensation-polymerization process, is regarded as a very fascinating research field, with many future industrial possibilities. The cotton fiber modified by silane-coupling agents exhibits versatile physico-chemical properties, including improved tensile strength, elasticity, better swelling properties, better wrinkle recovery properties, color-fastness and thermal stability properties, etc [8]. In this context, there has been a growing interest in chemical modification of cotton fibers by silane-coupling agents for industrial and textile applications. The interaction of silane-coupling agents with cotton fibers mainly proceeds through four steps which are (i) hydrolysis, (ii) self-condensation, (iii) adsorption, and (iv) grafting, and are shown in the following Scheme 1.

There are some studies based on graft polymerization of vinyl monomers, such as acrylic acrylate (AA), acrylamide (AAm) and acrylonitrile monomers, onto cellulose, using both chemical and radiation methods, available in the literature [4-7]. Our previous study was focused on the grafting of some silane-coupling agents, such as VTES, TSPM, GPTEs etc., on cotton fibers [8].



Scheme 1. Grafting mechanism of silane monomer with cotton fiber

In the present study, we aimed to investigate the capacity of VTMS and APTES to modify cotton fibers and the effect of modification to improve the physico-chemical properties of the fibers. The effects of modification parameters, such as temperature, grafting time, monomer, pH and the ethanol-water ratio on graft yield, were studied and optimum-grafting conditions were determined. The treated fibers were characterized using various experimental techniques such as Scanning electron microscopy (SEM), Thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR) and Energy disperse X-ray analysis (EDXA). The moisture absorption, swelling behavior, chemical resistance and tensile strength of the modified fibers were also studied for their physical characteristics. Besides, some instrumental and physico-chemical analysis of modified cotton fibers, such dyeing behaviour and color-fastness tests, were also performed.

2. Experimental

2.1 Materials

As a material for investigation, cotton fiber was collected from Keya Spinning Mills. Ltd. Bangladesh. Ethanol, sodium carbonate and acetic acid were purchased from Merck, Germany. The silane coupling agent vinyltrimethoxy silane (VTMS) and aminopropyltriethoxy silane (APTES) were collected from Aldrich, USA. All other reagents and solvents were commercial products of high purity. Two chlorotriazinyl derivatives of reactive dyes, Reactive Orange 14 and Reactive Brown 10, were purchased from Sigma, USA.

2.2 Washing of Cotton Fiber

Cotton fibers were at first washed thoroughly with 0.2% Na₂CO₃ solution, at 75°C, for 30 min in the ratio of 1:50 [10].

2.3 Silane Treatment

The modification procedure was performed according to the procedures reported in the published literature on the subject [9]. To determine the effect of reaction parameters on graft yield, one parameter was varied, while all other parameters were held constant. The pre-treated fibers (washed or untreated or unmodified) were dipped in an alcohol-water

mixture (60:40 v/v), containing VTMS and APTES, for 1 h. The pH of the solution was maintained in the range 3.5 to 4 using 0.2M acetic acid. The treated fibers were washed with distilled water and subsequently dried in hot air at 60°C. The swelling behavior, moisture sorption, thermal properties and chemical resistance behavior of the silane-treated fibers were studied.

2.4 Method of Dyeing

Reactive dyes were dissolved, at first, by making a paste with little hot distilled water and then by adding cold distilled water. Dyeing was carried out with reactive dyes in a dye bath containing 0-3.5 wt. % dye and 0-12.0 wt.% aluminum sulfate (Al₂(SO₄)₃), as an electrolyte, at 30-100°C, for 20-90 min in the fiber-liquor ratio of 1:30. Dyeing commenced by putting the fibrous material into the dye bath at room temperature. The temperature was increased to 70°C within 30 min and continued for 60 min. Then the dye bath was allowed to cool for 30 min. After 30 min of dyeing, the bath was rendered slightly more alkaline, using 2.0 wt.% sodium carbonate solution, for fixation of the dye on the fiber and dyeing was then continued. After dyeing, the amount of dye absorbed by the fiber from the dye bath was determined colorimetrically [11].

$$\text{Dye exhaustion, \%} = \frac{D_e \times D_o}{D_o} \times 100$$

Where, D_o and D_e are the original and exhausted dye bath concentration, respectively.

2.5 Evaluation of Physical Properties

2.5.1 Chemical resistance

The chemical resistance of the silane-treated fiber was studied as a function of percent weight loss of fiber when treated with different chemicals. A known amount of untreated and silane-treated fiber was dipped in a definite volume of hydrochloric acid and sodium hydroxide of definite strength for a time interval (12-48 h) and percent chemical resistance was calculated using the following formula [10].

$$\text{Chemical resistance, \%} = \frac{W_i \times W_f}{W_i} \times 100$$

Where, W_i and W_f indicates the initial and final weight of the fibers, respectively.

2.5.2 Wrinkle recovery angle

Wrinkle recovery of a fabric is defined as the ability of the fabric to resist the formation of wrinkles when subjected to a folding deformation. The wrinkle recovery measurement was performed by a Wrinkle Recovery Tester (Daiei Kagaku Seiki Ltd. Kyoto, Japan), in the Apparel Manufacturing Department, University of Textile Engineering, Dhaka. The test was performed by cutting the treated sample into 4.4×1.5 cm size patches. Then the patches were folded and kept under the weight of 500 gm for 5 min. Finally, the folded patches were inserted inside a template and placed in the testing machine.

2.5.3 Moisture absorption

The moisture absorption of the VTMS- and APTES-treated fibers as well as washed cotton fibers was tested at a constant humidity level. The treated and untreated samples of cotton fibers were dried at 60°C, in an oven, until a constant weight was obtained. The percent moisture absorption, was studied as a function of weight gain, and was calculated using the following formula [10].

$$\text{Moisture absorption, \%} = \frac{W_f \times W_i}{W_i} \times 100$$

Where W_i and W_f are the weight of the dried samples and the final weight of the sample taken out from the humidity chamber.

2.5.4 Swelling behavior

Swelling behavior of the modified and cotton fibers was observed by treating the fibers with water, methanol, and carbon tetrachloride. Known initial weights W_i of the VTMS- and APTES-treated and untreated samples were immersed in 100 ml of solvent at room temperature for 72 h. The samples were filtered and the excess solvent was removed with the help of filter paper, then the final weight W_f was measured. The percent swelling was calculated [10] as:

$$\text{Swelling, \%} = \frac{W_f \times W_i}{W_i} \times 100$$

Where, W_i and W_f are indicated the initial and final weight of the fibers, respectively.

2.6 Characterization of Washed and Surface-modified Cotton Fibers

2.6.1 Infrared spectroscopy

FTIR spectra of the silane-treated and washed cotton fibers were recorded with KBr pellets on a Shimadzu IR-8900 spectrophotometer (Shimadzu Kyoto Japan). The samples were analyzed in an attenuated total reflectance (ATR) detector over a range of 400-4000 cm^{-1} at a resolution of 4 $\text{cm}^{-1}/\text{min}$ [11].

2.6.2 Thermal analysis

The experiments were performed using a Seiko-Extar-TG/DTA-6300 (Seiko, Japan). The tests were conducted between 25-600°C in an inert atmosphere (argon). The heating rate and the air flow rate were 10°C/min and 200 mL/min, respectively [11].

2.6.3 Scanning electron microscopy and energy disperse X-ray analysis

Scanning electron microscopy (SEM) and Energy-disperse X-ray analysis (EDXA) were performed, using a scanning electron microscope (FEI Quanta Inspect, Model: S50, Kyoto Japan) to observe the micro-structure, the surface morphology and elemental analysis of the treated and untreated fibers' as well as untreated fibers. The fibers' surfaces were coated with a thin film of carbon to render them conductive [12].

3. RESULTS AND DISCUSSION

3.1 Modifications Parameters

The effect of silane monomer concentration in the modification of cotton fibers was studied by varying its concentration between 100 to 700%, with surfactant and without surfactant, at constant pH, time, temperature and fiber-liquor ratio. The experimental results were shown in Fig. 1 which represents the graft yield of cotton fibers as a percentage of weight gain.

Fig. 1 shows that the weight gain increased considerably at first, with increasing monomer concentration, reached a maximum value and then decreased. Maximum weight gain for VTMS was 40.1% at 600% silane concentration and for APTES was 46.5% at 300% silane concentration. This gradual increase in weight gain was due to cross-linking reactions between the -OH groups

of cellulose and $-OH$ groups of the silane-coupling agents at higher concentrations. Increasing rates of diffusion of monomer into the fiber structure also lead to a higher graft yield. The decrease in graft yield can be explained by the enhancement of homopolymer formation at higher monomer concentrations [9]. Similar results were also reported in chemically-initiated graft co-polymerizations of various vinyl monomers on natural fibers [5].

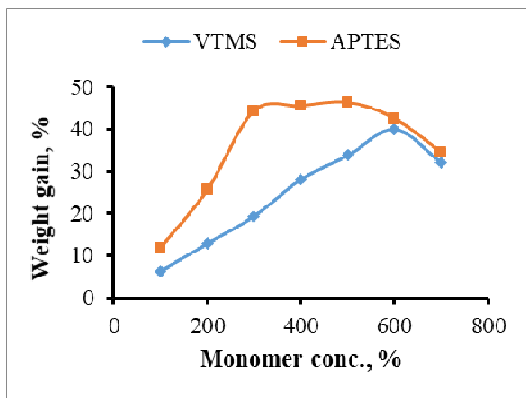


Fig. 1. Plots of silane vs. weight gain for cotton fiber

From the comparison study between the silane-coupling agents and other vinyl monomers, it can be concluded that silane-coupling agents are suitable grafting monomers for cotton fibers under the conditions established in this study [13]. The results in Fig. 1 justify this conclusion.

The effect of pH on weight gain was investigated by changing the pH from 2.5 to 6 while holding monomer concentration, temperature, time and ethanol-water ratio constant. As shown in Fig. 2, the weight-gain percentage increases with the increase of pH up to 3.5 for VTMS and 5 for APTES. At higher pH, weight gain starts to decrease.

The explanation for this rises and falls in weight gain becomes steadily more acidic is that the number of hydrolysis reactions of silane monomers gradually increased as the pH of the liquor increased, up to the stated *maxima* of 3.5 for VTMS and 5 for APTES (Fig. 2). At these maximum pH levels, the reactions between the silanol and hydroxyl groups of the fiber occur easily. Above these pH values, the hydrolysis of silane decreases gradually, yielding lower weight gain percentages of the modified cotton fiber [14].

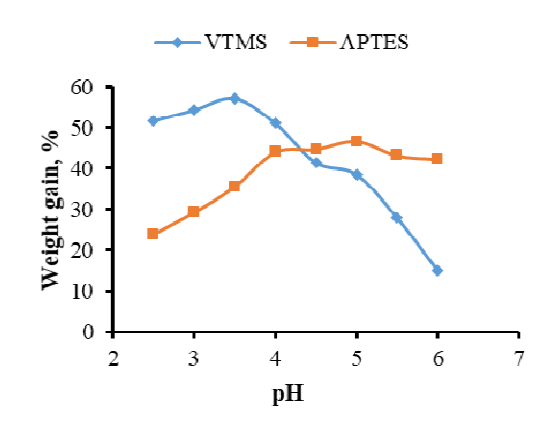


Fig. 2. Plots of pH vs. weight gain for cotton fiber

From Fig. 3 it can be seen that, at an absolute alcohol/water ratio of 60:40 for VTMS, 80:20 for APTES, the weight gain reached optimum value. At higher or lower alcohol-water ratios than the optimum values, the interaction between the water and alkoxy ($-OR$) groups during hydrolysis was less, yielding lower weight gain. This is because, at lower alcohol concentrations, the solubility of silane-coupling agents into the ethanol-water mixture is lower, but, at higher alcohol concentrations than the optimum values, the weight gain decreases because the hydrolysis of silane-coupling agents is less.

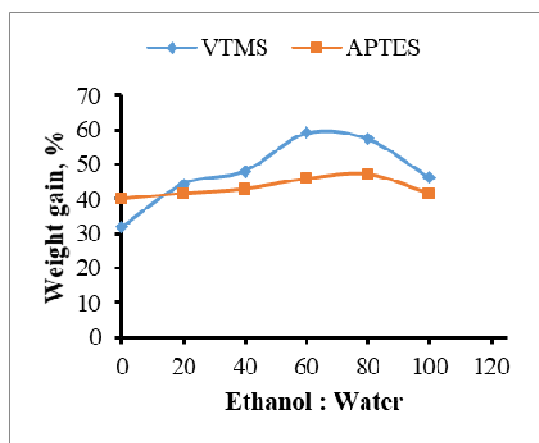


Fig. 3. Plots of ethanol water ratio vs. weight gain for cotton fiber

The effect of polymerization time on modification of cotton fiber was investigated by changing the time from 30 to 180 min at constant monomer concentration, pH and ethanol-water ratio, with

the results shown in Fig. 4. Weight gain initially increased with time and then reached a saturation grafting value, after 90 min for VTMS and 60 min for APTES. The rate of grafting increases with time, at first very sharply, due to maximum copolymerization reactions between the reactive sites of cellulose fibers and the activated monomers [15]. Beyond the optimum time for VTMS and APTES, homopolymerization reactions prevail over the copolymerization; this is possibly because of a lack of reactive sites on the jute and cotton fibers [13].

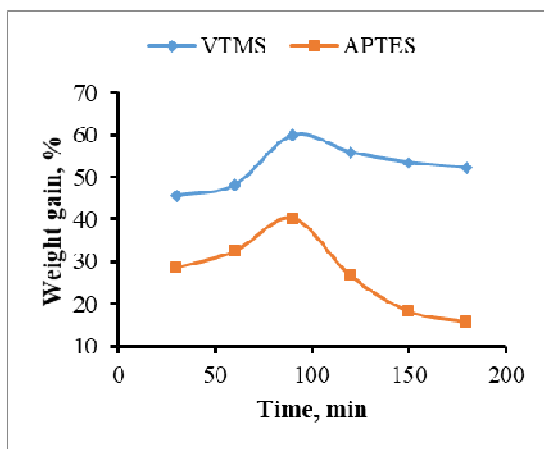


Fig. 4. Plots of time (min) vs. weight gain % for cotton fiber

3.2 FTIR Spectra of Grafted Cotton Fiber

The FTIR spectra of unmodified and silane-monomer modified cotton fibers were mostly similar, with adsorption peaks at around 1033-1060 cm^{-1} (C-O stretching), 1401 cm^{-1} (-CH₂ bending), 897-899 cm^{-1} (CH₂=CH-) and 1637 - 1637 cm^{-1} (C=C stretching) [11,16,17]. However, there are some new additional peaks at 761 cm^{-1} for Si-O-Si in the VTMS modified fibers and at 860 cm^{-1} for Si-OH, and at 1210 cm^{-1} for Si-O-CH₃ in the APTES modified cotton fiber which are shown in Figs. 5a-c, respectively. The peaks are indicated by arrows in the spectra, which confirms the incorporation of silane monomer on the cotton fiber surfaces [16-20].

3.3 Thermal Behavior

Thermal behavior of washed (unmodified) and silane modified, cotton fibers was examined by a study of their TGA thermograms, the results of

which are shown in Figs. 6a-c. The actual pyrolysis region of unmodified cotton fiber is from 321 to 369.7°C and that for the VTMS-, and APTES-modified cotton fibers is from 337 to 386.09°C, and from 308.3 to 377.9°C, respectively. The weight loss is about 60.0% for unmodified cotton fiber, and 39 and 33.6% for the VTMS-, APTES-modified fibers, respectively. Thus, the thermal stability of silane-modified fibers is increased compared to that of unmodified fiber which might have happened due to the incorporation of silane monomers with the fiber [9,20].

3.4 Surface Morphology

Figs. 7a-c shows the SEM micrograph of unmodified, VTMS-modified and APTES-modified cotton fibers respectively. The untreated cotton fiber show the presence of a large number of micro pores on their surfaces. After VTMS and APTES treatment, the cotton fiber surfaces are coated with an outer layer of silane monomer, as may be observed in Figs. 7b-c. The ruptured surface of the modified fiber indicates the excess deposit of silane in a layer on the cotton fiber.

3.5 Energy Dispersive X-ray Analysis

From the analytical point of view, the untreated cotton sample contains only carbon and oxygen, which is their elemental composition. The existence of silicon atoms on the functionalized fiber backbone represents the functional attachment of VTMS and APTES to the cotton fibers. The chemical elements as weight percentage are listed in Table 1.

From EDX analysis, the untreated cotton fiber has no Si atom, but, after modification with VTMS and APTES, the presence of Si atoms has been observed. After modification, the existence of Si-O-Si and Si-O-C bonds on the fiber surface was shown by the FTIR analysis. EDX analysis shows the presence of silicon atoms on the modified fiber which confirms the reliability of the FTIR analysis. The weight percentage of carbon and oxygen of unmodified cotton fiber are listed in Table 1. The VTMS and APTES functionalized cotton fiber contains 6.5 and 4.0 weight percent of silicon. The presence of tetravalent silicon on the functionalized cotton fiber ensures the presence of organic moiety with silicon atoms.

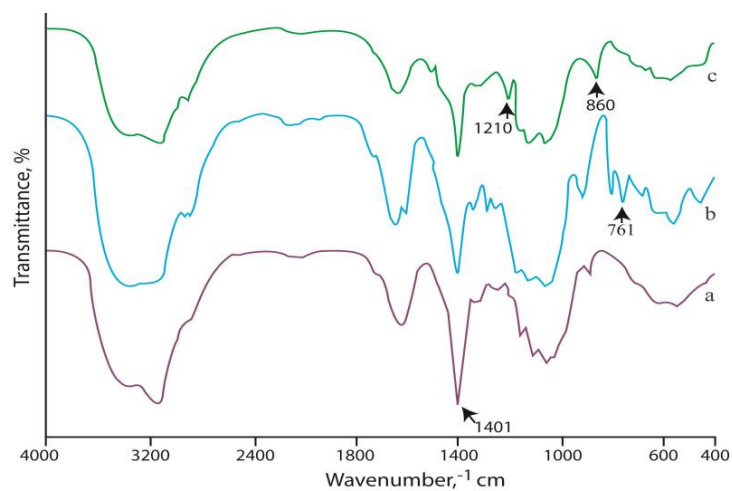


Fig. 5. FTIR spectra of (a) washed cotton fiber, (b) VTMS modified cotton fiber and (c) APTES modified cotton fiber

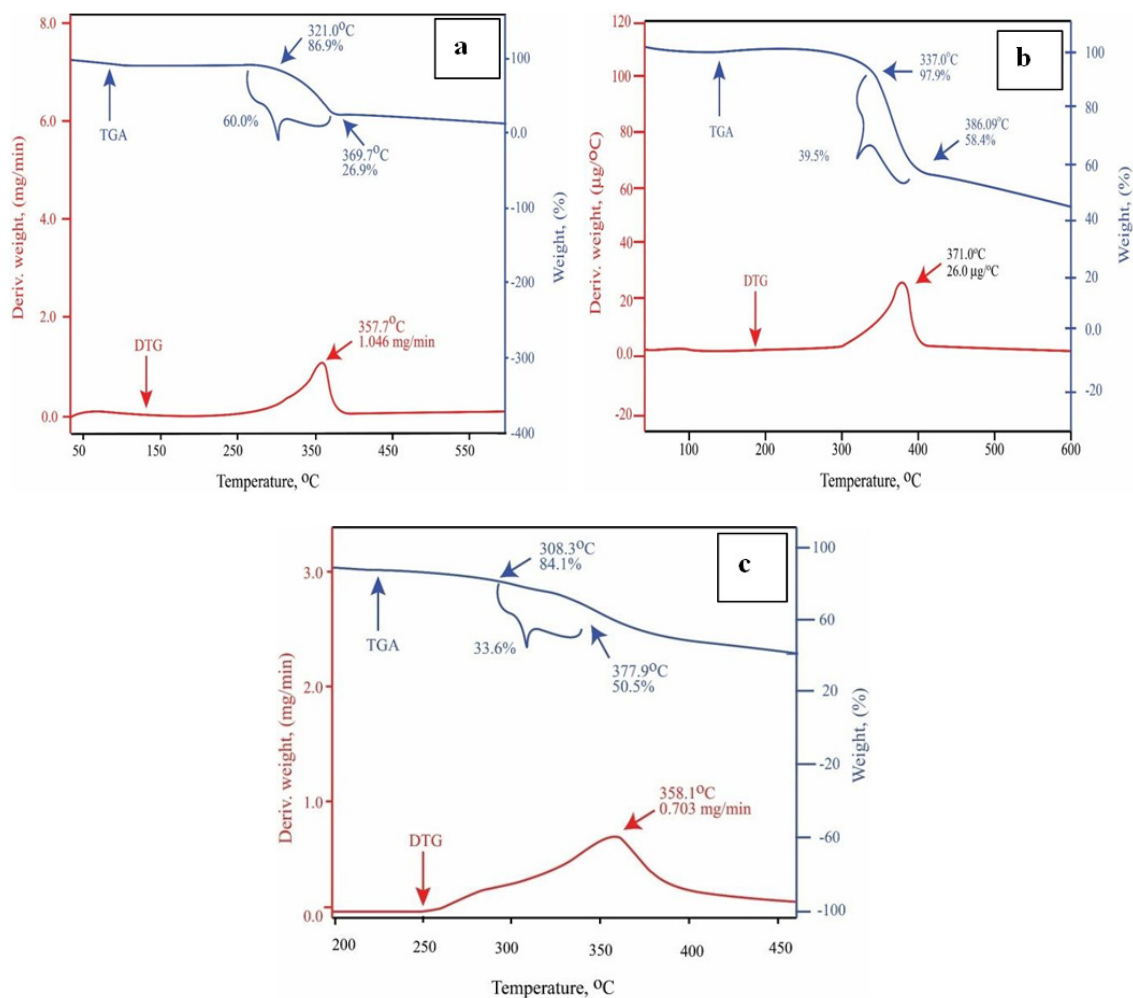


Fig. 6. TGA and DTG of (a) unmodified cotton fiber, (b) VTMS modified cotton fiber and (c) APTES modified cotton fiber

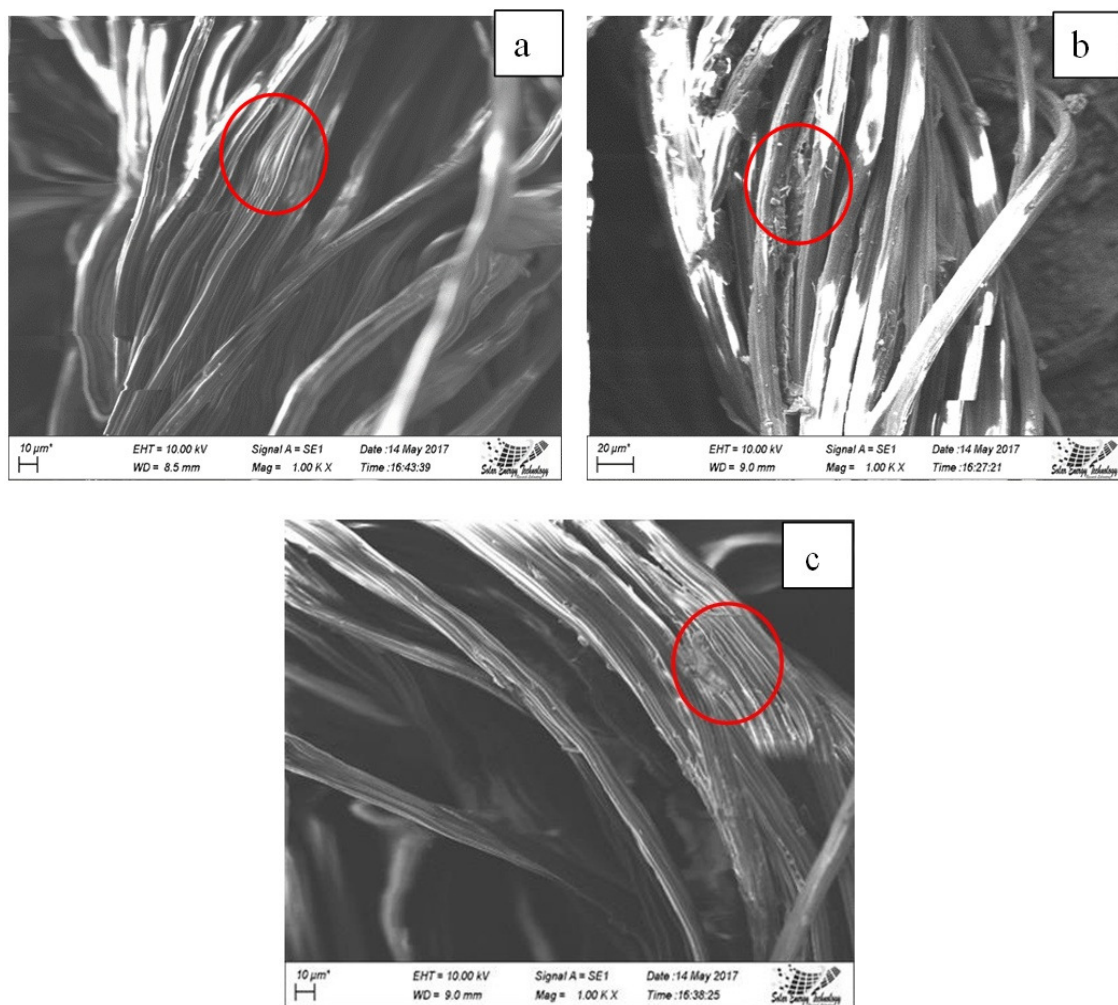


Fig. 7. SEM of (a) unmodified cotton fiber, (b) VTMS modified cotton fiber and (c) APTES modified cotton fiber

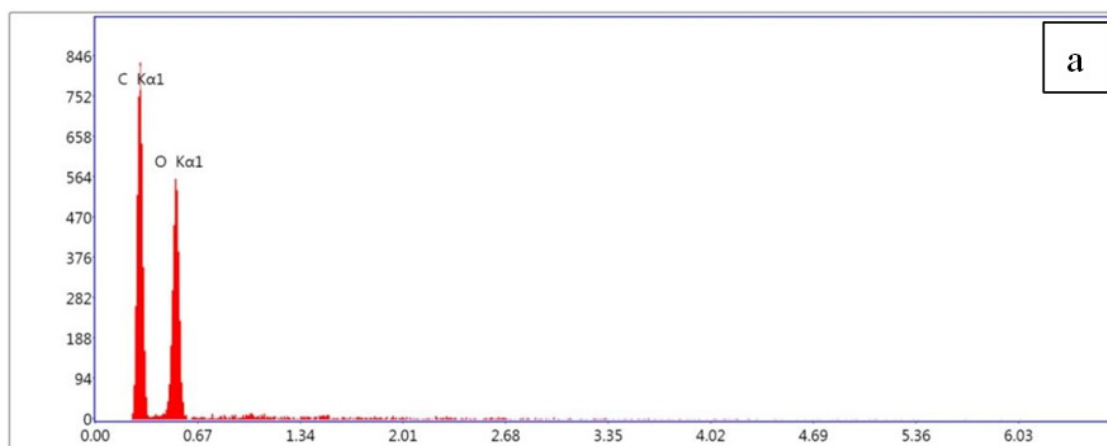
Table 1. Elemental composition of untreated and functionalized cotton fibers

Element	Untreated cotton		VTMS treated cotton		APTES modified cotton	
	Atomic, %	Weight, %	Atomic, %	Weight, %	Atomic, %	Weight, %
C	60.8	53.8	62.8	54.3	62.0	54.4
O	39.2	46.2	34.4	35.9	35.8	41.7
Si	-	-	3.2	6.5	1.9	4.0

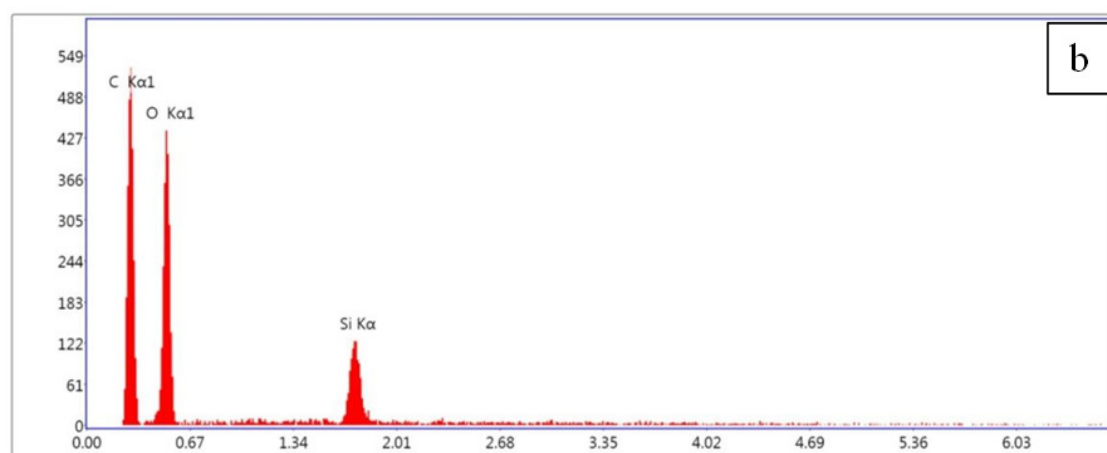
3.6 Physical Properties of Unmodified and Silane-modified Cotton Fibers

Table 2 shows the swelling behavior of unmodified cotton, as well as VTMS- and APTES- modified cotton fibers both in polar and nonpolar solvents. Swelling ability reflects the relationship between void structures in backbone polymers and the size of the solvent's molecules

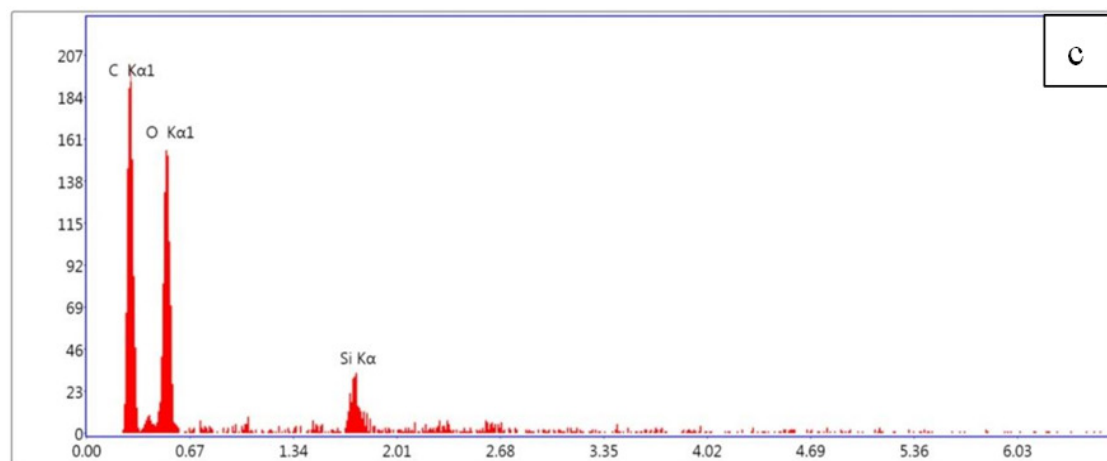
[21]. The washed, untreated cotton fibers exhibit maximum swelling with polar solvents like water and methanol, and least swelling with nonpolar solvents like CCl₄. After treatment with silane-coupling agents, there is a decrease in swelling in the polar solvents. Swelling increases in the nonpolar solvents because of decrease of the hydrophilic character of untreated cotton fiber.



Lsec: 50.00 Cnts 0.000 keV Det: Octane Prime Det



Lsec: 50.00 Cnts 0.000 keV Det: Octane Prime Det



Lsec: 50.00 Cnts 0.000 keV Det: Octane Prime Det

Fig. 8. EDX of (a) unmodified cotton fiber, (b) VTES modified cotton fiber and (c) APTES modified cotton fiber

The tensile strength of modified cotton fiber was greater than that of untreated cotton fiber. The increased tensile strength is due to the modification of cotton fiber with VTMS and APTES [22]. The wrinkle recovery angle of modified cotton fabric was higher than that of unmodified cotton fabric in both warp and weft directions. The presence of Si-O bonds in the functionalized fabric shows high flexibility, which easily recovers from wrinkles produced on the fabric surface by loading [23]. The moisture absorption sites are blocked after incorporation of silane chains, through surface modification, thus the modified fabric shows less affinity for moisture than did the original untreated fiber.

3.7 Dyeing Behavior of the Modified Cotton Fiber

It can be observed from the Table 3 that silane-modified cotton fiber absorbed more dye than untreated cotton fibers. Here, the reactive system of these reactive dyes enables them to react with the hydroxyl groups in cotton fiber by nucleophilic addition or substitution reaction. The reactive dyes are anionic and cotton fibers gain an anionic surface charge in water. For this reason, the charge repulsion adversely affects the dye bath exhaustion. Large quantities of electrolyte

(NaCl) are added to overcome this problem [24]. But, in the case of silane-modified cotton fiber the dye forms cross links with silane-coupling agents resulting in production of positive dye sites on the fiber surface. As a result, anionic dyes (reactive dyes) can easily be absorbed by electrostatic attraction, due to the created cationic nature of the fiber surface [25].

The exhaustion of dye by modified cotton fibers is greater than that of unmodified cotton fiber which may be caused by addition of more hydroxyl groups in cotton fibers due to the cross linking of silane-coupling agents with fiber polymers.

The exhaustion of reactive dyes by unmodified and silane-modified cotton fibers is shown in Table 3. The dye exhaustion of the silane-modified cotton fiber was higher than that of the unmodified cotton fiber and the dye exhaustion increased with an increase in the percent graft yield. The modification of cotton fiber has enhanced the dyesites in the cellulose macromolecules of cotton fibers. As a result, the modified fiber absorbed more dye than did the unmodified sample. This greater rate of absorption has increased the exhaustion percentage of dye in the modified cotton fibers.

Table 2. Swelling behavior, tensile strength, wrinkle recovery angle and moisture absorption properties of washed and modified cotton fibers

Fiber type	Swelling behavior, %			Tensile strength		Wrinkle recovery angle, degree		Moisture absorption, %
	H2O	CH3OH	CCl4	Breaking load, Kg/yarn	Elongation, %	For warp	for weft	
Washed cotton	273.2	142.3	78.5	2.4	2	28	50	6.66
VTMS modified cotton	121.95	97.62	25.0	2.7	2.5	60	52	4.12
APTES modified cotton	114.5	75.85	78.85	4.00	3.21	58	55	4.32

Table 3. Effect of dye absorption on dyeing of unmodified and modified cotton fibers

Type of cotton sample	Dye exhaustion, %	
	Reactive Orange 14	Reactive Brown 10
Untreated cotton fiber	69.10	65.20
VTMS modified fiber	74.25	67.20
APTES modified fiber	80.10	75.50

4. CONCLUSION

The results of the authors' study of the effects of chemical modification of cotton fibers with silane-coupling agents has been presented in this paper. Maximum weight gain percentage is obtained at optimum values of the reaction parameters such as silane concentration, pH, ethanol-water ratio and temperature. The chemical attachment between silanol and hydroxyl groups on cotton fibers was evaluated by FTIR, SEM, TGA, DTG and EDX analysis. The modified fibers show improved physico-chemical properties such as tensile properties, moisture absorption, elongation, wrinkle recovery and thermal stability properties when compared to that of the unmodified cotton fibers. This new type of cotton was obtained through modification with silane-coupling agents, which enhance its usefulness in the production of garments, textiles etc.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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