

Modification of Cotton Fibre with Functionalized Silane Coupling Agents Vinyltriethoxysilane and Aminopropyltriethoxysilane

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Abstract

Cotton fibre was modified by condensation polymerization with functionalized silane coupling agents like vinyltriethoxysilane (VTES) and aminopropyltriethoxysilane (APTES) in an ethanol/water medium. The modification of cotton fibre enhanced the tensile properties, softness properties, water repellence and wrinkle recovery, due to higher flexibility of the Si-O bond between the silane coupling agents and the cotton fibre. The ability of the modified cotton fibre to swell decreased in an aqueous solution, which affects the overall chemical suitability of the fibre. The optimized condition in which to modify cotton fibre was a monomer concentration of: 500% for VTES and 600% for APTES (depending on the weight of the fibre); pH 3.5 for VTES and 5 for APTES; ethanol-water ratio 40:60 for VTES and 80:20 for APTES; reaction time of 90 minutes both for VTES and APTES at room temperature (30°C), in the fibre-liquor ratio of 1:50. Swelling of the modified cotton fibre decreased in polar solvents and increased in nonpolar solvents. Fourier transform Infrared spectroscopy was used to identify the incorporation of silicon-containing molecules. Energy Disperse X-ray analysis determined the quantities of atomic silicon which directly reflects its valence bond with organic moieties. Scanning electron microscopy and thermogravimetric analysis were used to investigate the surface morphology and thermal behavior of the modified fibre, respectively. Examination of the dyeability of washed and modified cotton fibres, dyed with Reactive Brown-10 and Reactive Orange-14, showed that the absorption of dye by modified fibre was comparatively higher than that of raw cotton fibre.

Keywords: Cotton fibre; Silane coupling agents; Surface functionalization; Swelling; Reactive dyes

Introduction

Cotton fibre is one of the most important natural fibres and it has a wide range of applications in the textile field because of its easy availability, low density, light weight, low cost, and, above all, environmentally-friendly characteristics [1-3]. It can easily be transformed into multifarious products, affecting every phase of our daily life, because of its widespread application.

In spite of a number of advantages, these natural fibres also have a few disadvantages, such as lower flexibility and lower softness properties during processing. The presence of the hydroxyl group in the celluloses makes the cotton fibre hydrophilic and sensitive to moisture present in nature. Such sensitivity can impair the properties of materials fabricated by utilizing these biofibres [4,5].

So, it is necessary to improve the quality of cotton fibre for commercial utilization. Such improvement is needed to meet total fabric requirements of the users, as well as to minimize the use of synthetic fibres. Various chemical treatments such as silane treatment, mercerization, etherification, acetylation, and graft copolymerization with various monomers have been used to improve or alter the physio-chemical performance of natural fibres [6,7]. These treatments include pre-treatment of the cotton fibre's surface with coupling agents.

Chemical treatment of the cotton fibres not only reduces their moisture absorption process but also increases surface roughness, resulting in improved textile performance of the fibre. Although a lot of work has been reported on modification of cotton fibres by vinyl monomers and other modifying agents [8-10], much less has been reported on modification of cotton fibre by silane coupling agents. It is expected that the properties of this fibre, after modification by a polymerization process, can improve to such an extent that the modified fibre can be utilized in the manufacture of high-grade products. The interaction of the silane coupling agents with the cellulose fibre and the effect of the following parameters on their anchoring capability onto the

fibre surface have been ascertained: such as silane structures, hydrolysis process of silanes, pH, the initial amount of silanes with respect to cellulose, and the adsorption contact time. The interaction of silane coupling agents with cotton fibres mainly proceeds through four steps: (i) hydrolysis, (ii) self-condensation, (iii) adsorption, and (iv) grafting [11,12] (Figures 1a and 1b).

These reactions are generally carried out under acid-catalyzed conditions as this condition helps to increase the rate of hydrolysis of silanes into silanols, when compared with the condensation process. Some other authors have applied different methods for silane treatment and they have studied the effect of silane treatment on surface morphological and hygroscopic character of the natural fibres [13,14].

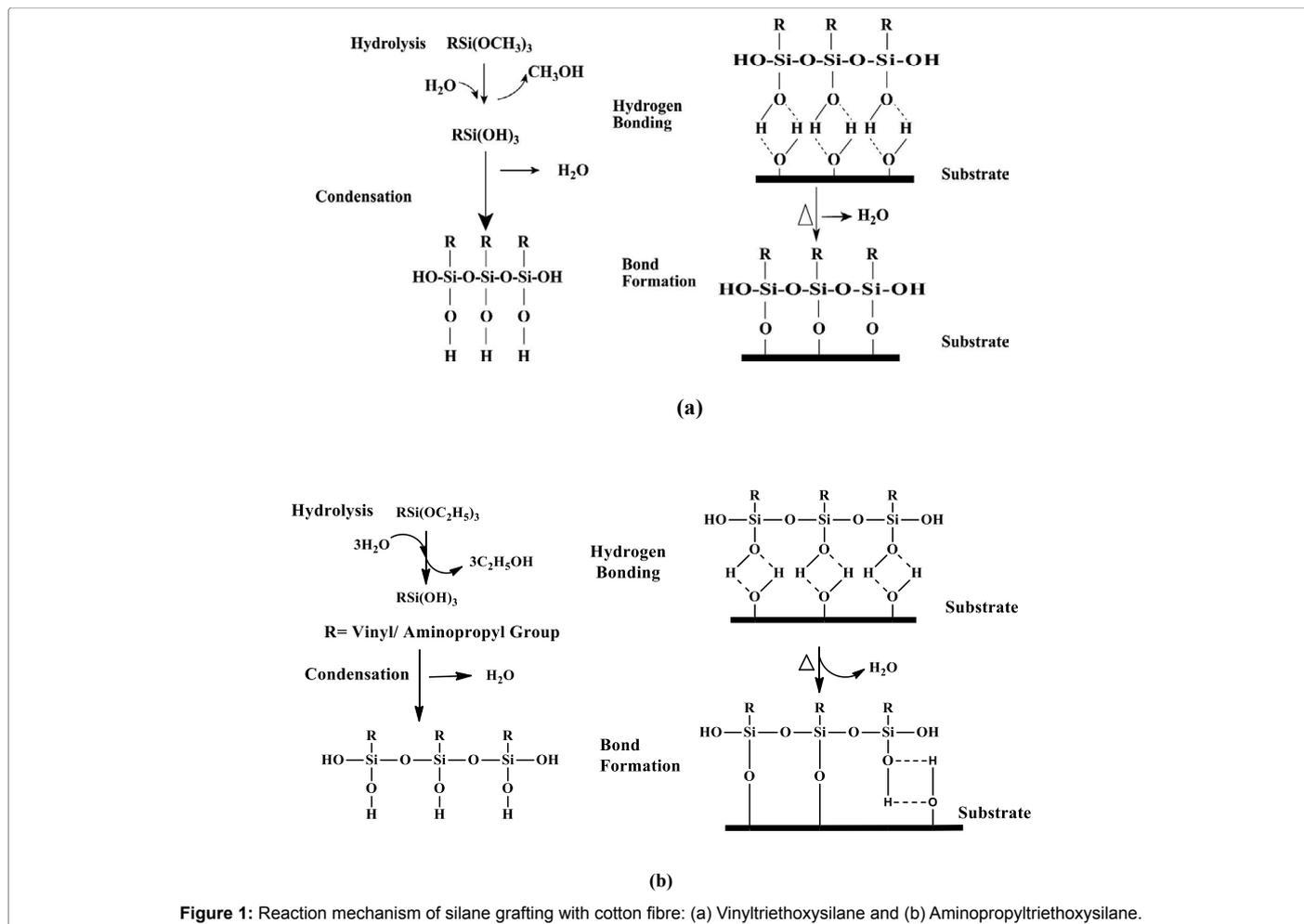
The present study, investigates the effect of silane treatment on the moisture resistance, swelling behavior, tensile strength, wrinkle recovery properties, thermal stability and surface morphology of cotton fibres. In the present paper, we report on the surface functionalization of cotton with vinyltriethoxysilane and aminopropyltriethoxysilane. The objective of this functionalization is to provide a new way to impart new and diverse properties to textiles, while retaining comfort and mechanical strength of their end-products. The modification process was optimized and grafting was determined on the basis of weight increase. The grafted fibre was characterized from FTIR, SEM, TGA

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and EDX analyses. Dyeability of raw and modified cotton fibre, dyed with Reactive Orange 14 and Reactive Brown 10, was studied.

Experiments

Material

Cotton fibre was obtained from Keya Spinning Mill, Dhaka, Bangladesh. The chemicals used for the functionalization were sodium hydroxide from Uni-chem (China), glacial acetic acid, methanol, ethanol and carbon tetrachloride from Merck, Germany, silane coupling agents from Aldrich (USA). All chemicals used were of analytical reagent grade.

Washing of cotton fibre

The cotton fibres were washed with 0.2% Na_2CO_3 solution at 75°C for 30 minutes, in a beaker, in the fibre to liquor ratio of 1:50. The fibres were then washed thoroughly with distilled water until neutralization reached and then dried in the open air. After that the fibres were dried in an oven at 60°C [15].

Silane treatment

Silane treatment of cotton fibres was carried out with vinyltriethoxysilane and aminopropyltriethoxysilane. First, the required amount (%) of silane solution was prepared, by mixing silane

coupling agents with an ethanol/water mixture, where the ratio of fibre to liquor was maintained at 1:50.

This solution was allowed to stand for 1 hour. The pH of the solution at 3-4 was maintained using 0.2 M acetic acid.

Then, the cotton fibres were dipped in this solution and stirred continuously for 1.5 h at varying temperatures. The cotton fibre was filtered from the solution. The fibres were dried in open air and then in an oven, at 60°C , to a constant weight [15].

Evaluation of Physical Properties

Measurement of tensile strength

The tensile strength of raw and silane modified cotton fibres was measured using a "Portable Electronic Single Yarn Strength Tester YG021" Fanyuan Instrument (HF) Co., Ltd., China, for quick and reliable tensile strength measurement. The maximum load range, gauge length and stretching rate used in this machine were 3000 kg, 500 mm and 500 m/min, respectively.

Moisture absorption: The moisture absorption of the silane-treated cotton fibres, as well as raw fibres, was observed at room temperature. The treated and raw samples of cotton fibres were dried at 105°C , in an oven, until a constant weight was obtained. The percentage of moisture absorption was determined as a function of weight gain and was calculated using the following formula:

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

Moisture absorption: 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, respectively.

Swelling capacity: Swelling capacity of the silane-treated and raw cotton fibres was determined by treating them with water, methanol, and carbon tetrachloride. The silane treated and raw cotton fibres were immersed in 100 mL of solvents at room temperature for 72 hours. The samples were filtered and the excess solvent was removed with the help of filter paper: then, the final weight was determined. The percentage swelling was calculated from the increase in initial weight in the following manner [16]:

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

Where W_i and W_f are the weight of the dried samples, and the final weight of the sample, respectively.

Wrinkle recovery angle: A wrinkle recovery tester, Daiei Kagaku Seiki Ltd. Kyoto, JAPAN, was used to determine wrinkle recovery angle. The machine looked like a giant round table clock and is marked angle 0 (zero) to 180. Angle 0 degree means no recovery. Higher angles mean wrinkle recovery is greater. The samples were cut into 4.4 cm × 1.5 cm pieces. Then the cut samples were folded and kept under the weight of 500 gm for 5 minutes. The folded samples were inserted inside a template and the template was placed in the testing machine. The recovery angle was observed from the dial.

Exhaustion of dye by cotton fibre: Dyeing was carried out using 0.3% reactive dye (on the weight of fibre) in a dyeing machine (DYSIN, Taiwan, China) at 65°C for 1 hour, using a plastic stoppered conical flask.

The exhaustion of dye was determined colorimetrically (Type-S104, No-221, Spectrophotometer, WPA Linton Cambridge, UK) by using the following equation:

$$\text{Exhaustion of dye, \%} = \frac{(D_0 - D_e) \times 100}{D_0}$$

Where, D_0 and D_e are the initial and exhausted dye bath concentration, respectively.

Characterization of Surface Modified Cotton Fibres

Infrared spectroscopy

FTIR spectroscopy analysis was performed by using a Perkin Elmer Spectrum 100 infrared spectrometer. The selected fibres and potassium bromide (KBr) were dried in an oven at 105°C to assure that they were moisture free. The dried cotton fibre was powdered using mortar and pestle. About one percent of the powder was mixed with dried KBr to make a pellet [8]. Then, the samples were analysed in an attenuated total reflectance (ATR) detector over a range of 400-4000 cm^{-1} at a resolution of 4 $\text{cm}^{-1}/\text{min}$.

Scanning electron microscopy analysis

Scanning electron microscopy analysis of the raw and silane-modified cotton fibres was carried out in an electron microscope (FEI Quanta Inspect, Model: S50, Kyoto, Japan) to observe the microstructure and the surface morphology. Since these materials are non-conducting, they were gold plated. The analytical steps were

performed with an acceleration voltage of 25 kV at a working distance of 11 mm, with 1000x magnification.

Thermogravimetric analysis

Thermogravimetric analysis was used to determine the thermal decomposition rate and the thermal stability of the raw and silane modified cotton fibres. The samples, approximately 10 mg each, were heated from 30 to 600°C under an inert atmosphere (of argon), at a rate 20°C min^{-1} in a Seiko-Extar-TG/DTA-6300 (Seiko-Japan).

Energy dispersive X-ray analysis

Energy Dispersive X-Ray Analysis (EDX) was performed in a solid state device (FEI Quanta Inspect, Model: S50), in which the specimens were subjected to an electron beam of accelerating voltage 15 kV of energy. Then X-rays emitted from the chemical elements present in the specimen were detected by a SUTW- SAPPHIRE detector. An EDX instrument converted these X-rays into an electrical charge. This charge became the signal which, when processed, then identified the X-ray energy and its elemental source.

Results and Discussions

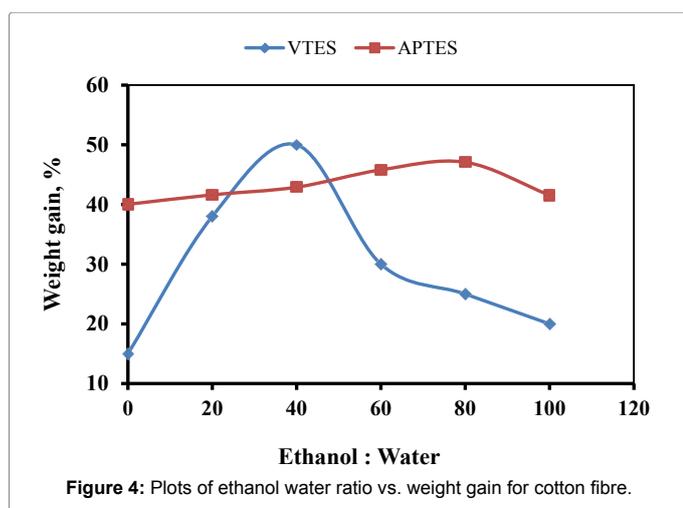
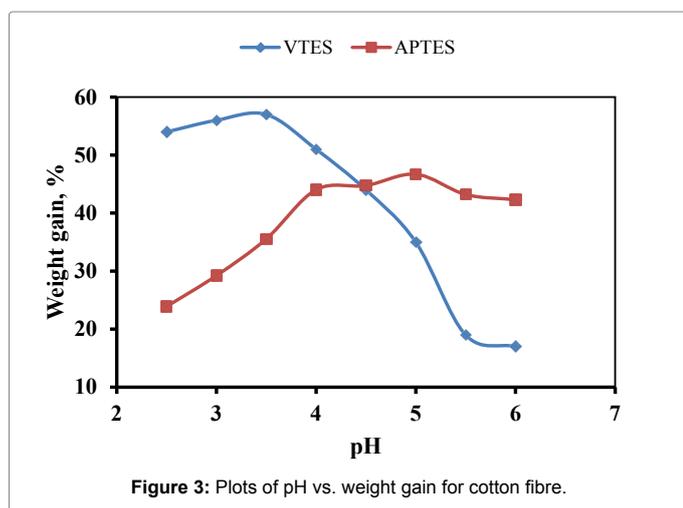
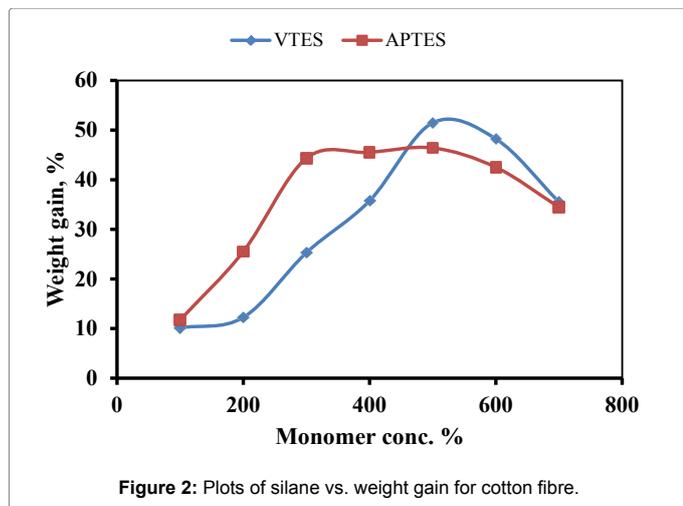
Modification of cotton fibre

The effect of silane coupling agents on modified cotton fibre is shown in Figure 1. The weight gain increased with the increase of silane concentration for VTES and APTES. After a certain value, it decreased with a further increase in the monomer concentration. This gradually increase in weight gain was due to the higher cross-linking reaction between the -OH groups of cellulose and -OH groups of the silane coupling agents at higher concentration and increasing rate of diffusion of monomer into the fibre structure also leading to a higher graft yield. The decrease in graft yield can be explained by the enhancement of homopolymer formation in higher monomer concentration instead of copolymer [17]. Condensation reaction of pre-hydrolyzed silane coupling agents reduces the availability of reactive hydroxyl groups. Slowly increasing the silane concentration definitely increases the number of reactive groups in the reaction medium.

The insertion of dilute surfactant (Sodium dodecyl sulfate) solution increases the chemical conversion, by improving the stability of aqueous silane dispersions [18,19]. The surfactant solution was used to reduce the surface tension of the reaction mixture. The maximum graft yield was obtained when the cotton fibre was treated with 500% vinyltriethoxysilane and 600% aminopropyltriethoxysilane (Figure 2).

From Figure 2, it can be observed that the weight gain percentage increases with the increase of pH value and then starts to decrease. The hydrolysis reaction of silane monomer gradually reached a higher value, when the pH of the liquor was increased to 3.5 for VTES and 5 for APTES, which appeared as a weight gain percentage. This occurred because, at this pH, the reaction between the silanol and hydroxyl groups of the fibre occurs easily. Above this pH value, the hydrolysis of silane decreases gradually, which appeared as a lower weight gain percentage of the modified cotton fibre [7] (Figure 3).

Figure 3 shows the effect of ethanol-water ratio on modification of cotton fibre. The solubility properties of the silane in the aqueous solution were triggered by using the absolute alcohol/water as a solvent for conversion of silane coupling agents into silanol and some siloxane. These agents were then deposited on the fibre surface by chemical bonding. The hydrolysis process was facilitated by homogenizing agents like absolute alcohol [7,12,13].



From Figure 3, it can be observed that, at an ethanol to water ratio of 40:60 for VTES and 80:20 for APTES, the weight gain after modification reached the optimum value. At lower alcohol concentration than optimum value, the weight gain decreased. This is due to lower solubility of silane coupling agents in low ethanol concentration. At

higher alcohol concentration than optimum value, the weight gain decreased because of decreasing the hydrolysis (Figure 4).

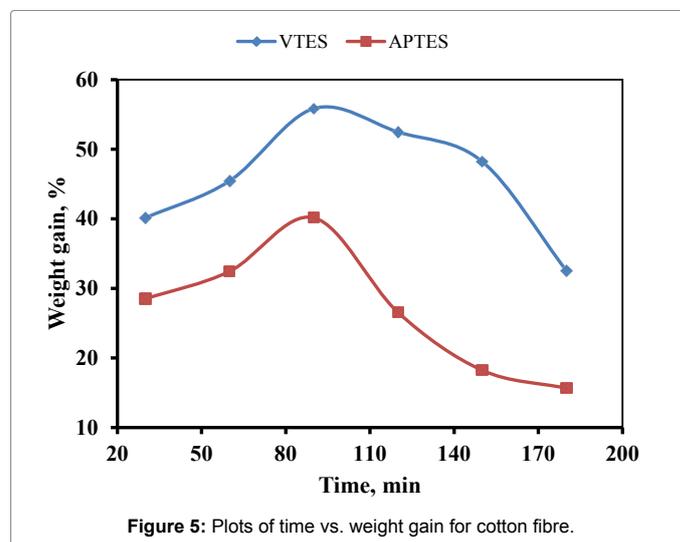
From Figure 4, we can see that weight gain increased with the increase of time, up to 90 minutes, both for VTES and APTES. After this period of time, the graft yield starts to decrease. The rate of grafting increases with time at first very sharply, due to maximum copolymerization reaction between the reactive sites of cotton fibres and the activated monomers. After 90 minutes, both for VTES and APTES, homopolymerization reactions prevail over the copolymerization reaction. Perhaps this is because the cotton fibres do not have enough reactive sites, resulting in homopolymerization (Figure 5).

Characterization

FTIR spectra of grafted jute fibre: The FTIR spectra of the raw and modified cotton fibres are shown in Figure 5. The FTIR spectra of raw and silane-modified cotton fibres are mostly similar, as the adsorption peaks were obtained in the spectra for the entire sample at around 897-899 cm^{-1} ($\text{CH}_2=\text{CH}-$), 1033-1060 cm^{-1} (C-O stretching), 1401 cm^{-1} ($-\text{CH}_2$ bending), and 1637-1638 ($\text{C}=\text{C}$ stretching) [20-23]. Except the new additional peak at 761-765 cm^{-1} in the spectra of both VTES and APTES modified and at 1280 cm^{-1} in the spectra of VTES-modified cotton fibre [24]. Figure 5 indicates the functionalization of silane monomer on the cotton fibres surfaces (Figure 6).

Thermal effects: Thermal behaviour of raw and silane-modified cotton fibres was examined by a study of their TGA thermograms, which is shown in Figure 6a-6c. The actual pyrolysis region of raw cotton fibre is 321 $^{\circ}\text{C}$ to 369.7 $^{\circ}\text{C}$, and that for the VTES and APTES-modified cotton fibres are 333.9 $^{\circ}\text{C}$ to 372.4 $^{\circ}\text{C}$ and 308.3 $^{\circ}\text{C}$ to 377.9 $^{\circ}\text{C}$, respectively. The weight loss is about 60.0% for raw cotton fibre. The weight loss is about 52.0% and 33.6%, respectively, for VTES and APTES-modified cotton fibre. So, it can be seen that the thermal stability of silane-modified fibres is greater than that of unmodified fibre, which might be due to the incorporation of silane monomers on the surface of fibre (Figures 7a-7c).

Surface morphology: Figures 7a-7c show the SEM micrograph of unmodified, VTES- modified and APTES-modified cotton fibres, respectively. The untreated cotton fibre shows the presence of a large number of micropores on its surface. After VTES and APTES treatment, the cotton fibre surfaces are coated with an outer layer of silane monomer [25] which can be observed in Figures 7b and 7c. The



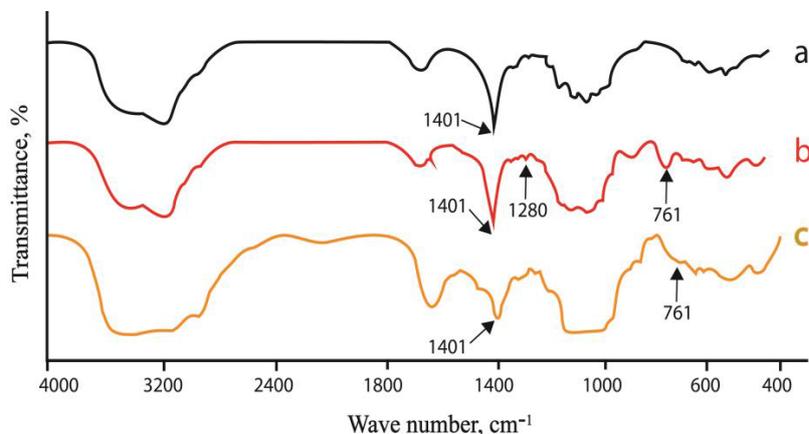


Figure 6: FTIR spectra of (a) raw cotton fibre, (b) VTES modified cotton fibre and (c) APTES modified cotton fibre.

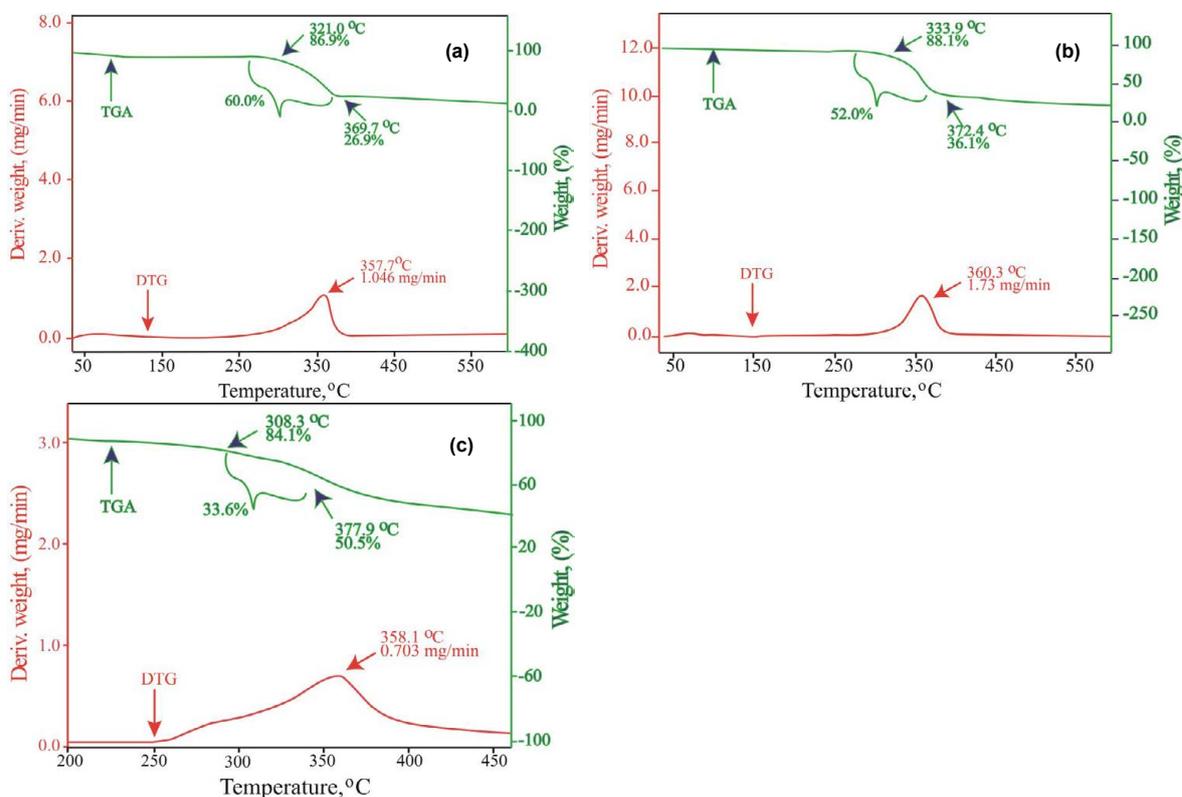


Figure 7: TGA and DTG of (a) raw cotton fibre, (b) VTES modified cotton fibre and (c) APTES modified cotton fibre.

modified fibre has a smoother surface than that of raw fibre, due to the homogeneous deposition of the silane layer on the fibre backbone (Figures 8a to 8c).

Energy-dispersive X-ray analysis: From Figure 8, it can be seen that the raw cotton sample contains carbon and oxygen, which are its elemental composition. The existence of a silicon atom on the functionalized fibre backbone represents the attachment of VTES and APTES on modified cotton fibres. The chemical elements, by weight percentage, are listed in Table 1. From EDX analysis, the raw cotton fibre has no Si atom (Figure 8a) but, after modification with VTES and APTES, the presence of a Si atom is observed in Figure 8b and 8c. After modification, the existence of Si-O-Si and Si-O-C bonds on the fibre

surface were identified by the FTIR analysis. The VTES- and APTES-functionalized cotton fibre contains 1.9 and 4.2 weight percent of silicon. The presence of tetravalent silicon in the modified fibre sample ensures the modification as well as functionalization of cotton fibres (Figures 9a to 9c and Table 1).

Physical properties of raw and silane-modified cotton fibres: Table 2 shows the swelling of raw cotton as well as VTES- and APTES-modified cotton fibres, both for polar and nonpolar solvents. Swelling ability reflects the relationship between void structures in backbone polymer and the size of the solvents molecule [4,5]. The raw cotton fibres exhibit maximum swelling with polar solvents like water and methanol and least swelling with nonpolar solvents like CCl₄. After

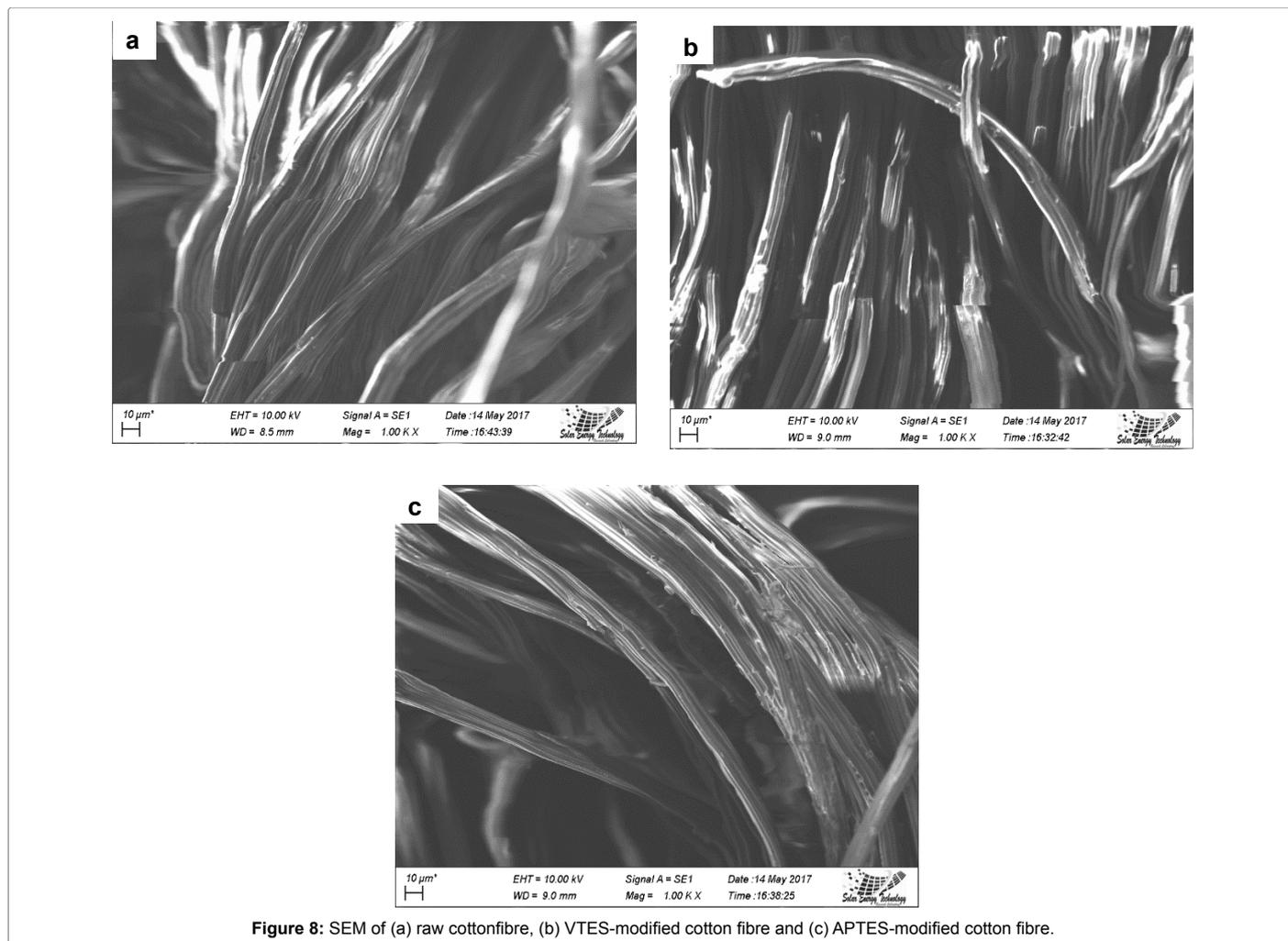


Figure 8: SEM of (a) raw cottonfibre, (b) VTES-modified cotton fibre and (c) APTES-modified cotton fibre.

Element	Untreated cotton		VTES treated cotton		APTES modified cotton	
	Atomic, %	Weight, %	Atomic, %	Weight, %	Atomic, %	Weight, %
C	60.8	53.8	62.1	54.7	61.7	53.7
O	39.2	46.2	37.0	43.5	36.3	42.1
Si	-	-	0.9	1.9	2.1	4.2

Table 1: Elemental composition of raw and functionalized cotton fibres.

Fibre type	Swelling, %			Tensile strength		Wrinkle recovery angle, degree		Moisture absorption %
	H ₂ O	CH ₃ OH	CCl ₄	Breaking load, Kg/yarn	Elongation, %	For warp	For weft	
Raw cotton	273.2	142.3	78.5	2.4	2	28 degree	50 degree	6.7
VTES modified cotton	140.3	92.6	35.4	3.10	2.80	60 degree	52 degree	3.2
APTES modified cotton	114.5	75.8	28.3	4.00	3.21	58 degree	55 degree	4.3

Table 2: Swelling, tensile strength and wrinkle recovery angle and moisture absorption properties of raw and modified cotton fibres.

treating with silane coupling agents, there is a decrease of the swelling in the polar solvents but an increase in the nonpolar solvent. This is because of hydrophilic character of raw cotton fibre is less than that of the modified fibre. The tensile strength of modified cotton fibre was higher than that of raw cotton fibre, due to the modification of cotton fibre with VTES and APTES [26].

The wrinkle recovery angle of modified cotton fabric was higher than that of unmodified cotton fabric for both warp and weft directions.

The presence of an Si-O bond in the functionalized fabric shows high flexibility, which recovers the wrinkle produced in the fabric's surface by loading [13]. The moisture absorption sites are blocked after incorporation of silane, through surface modification, reducing the affinity for moisture (Table 2).

Dyeing behaviour of the modified cotton fibre: The exhaustion of reactive dyes by raw and silane-modified cotton fibres has been set out in Table 3. The dye exhaustion of the silane-modified cotton fibre

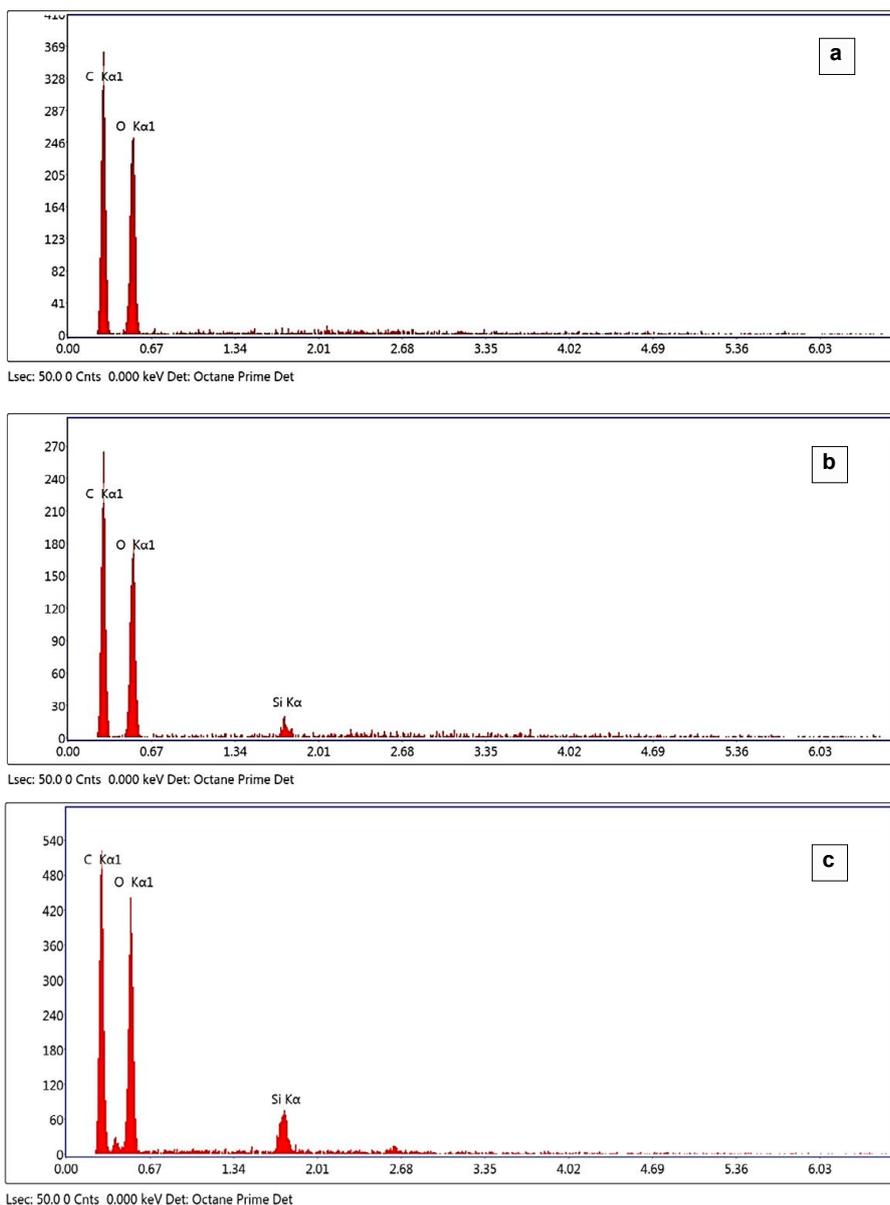


Figure 9: EDX of (a) raw cotton fibre, (b) VTES modified cotton fibre and (c) APTES modified cotton fibre.

Type of cotton sample	Dye exhaustion, %	
	Reactive Orange 14	Reactive Brown 10
Raw cotton fibre	69.10	65.20
VTES modified fibre	74.85	69.30
APTES modified fibre	80.10	75.50

Table 3: Effect of Dye Absorption on Dyeing of Raw and Modified Cotton Fibres.

was higher than that of the raw cotton fibre and the dye exhaustion increased with an increase in the percent graft yield. The modification of cotton fibre has enhanced the dye sites in the cellulose macromolecule of cotton fibres. In the modification process, cotton fibres form cross linking with silane coupling agents resulting positive dye sites on the fibre surface and anionic dyes (reactive dyes) can easily be absorbed by electrostatic attraction due to the created cationic nature of the fibre surface [27]. As a result, the modified fibre absorbed more dye than the raw sample and this absorption increased the exhaustion percentage of dye in the modified cotton fibres.

Conclusion

In this work, we have presented the results of the chemical modification of cotton fibres with silane coupling agents. Maximum weight gain percent is obtained at the optimum value of the reaction parameters such as silane concentration, pH, ethanol-water ratio and time. The chemical attachment between silanol and the hydroxyl group of cotton fibres was evaluated by FTIR and EDX analysis. The modified fibres showed improved physicochemical properties such as tensile properties, moisture absorption, elongation, wrinkle recovery and thermal stability properties compared to that of the unmodified cotton fibres. This new type of cotton was obtained through modification with silane-coupling agents, which suggest the possibility to apply this method in the production of garment products, textiles etc.

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