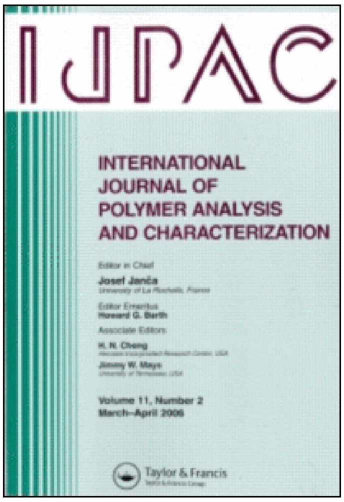

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Surface-Modified *Hibiscus sabdariffa* Fibers: Physicochemical, Thermal, and Morphological Properties Evaluation

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Abstract: Recently, interest in effective utilization of natural fibers has grown because of the advantages these fibers offer over synthetic fibers. Chemical treatment of natural fibers helps in incorporating new functionalities into them. *Hibiscus sabdariffa* fibers for use in various applications were mercerized and modified by silane treatment. Morphological and structural changes of the fibers were investigated by using scanning electron microscopy (SEM), Fourier transform infrared spectrophotometry (FT-IR), and thermogravimetric/differential thermal analysis along with DTG studies. The physicochemical (swelling in different solvents, moisture absorbance, water uptake, and chemical resistance) properties of these fibers were also measured.

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Keywords: Morphological and physicochemical properties; Silane treatment

INTRODUCTION

Polymers, especially from renewable resources, have attracted an increasing amount of attention over the past two decades and have replaced various conventional materials in a diversity of applications.[1,2] This has been possible because of the advantages polymers offer over other conventional materials. The most important advantages of using polymers in various fields are ease of production and processing and decrease in cost of the materials.[3,4] Increasing environmental awareness throughout the globe has forced researchers to produce new green materials that improve the environmental quality of products. Generally, in most of their applications, the properties of polymers are modified using natural fillers and natural fibers to suit the high-strength/high-modulus requirements.[5–7] Keeping in view the eco-friendly advantages of green materials, a large number of industries are initiating the design and engineering of these products. Natural fibers are renewable assets from various resources, especially from plants, agricultural, or forestry crops, that can be used in various industrial products. These fibers have applications in textiles, special pulps, geotextiles and ropes, insulating materials, and nonwoven materials, and increasingly as reinforcement in green or eco-friendly composites.[8–10] Natural fibers, depending upon the source, are classified into three types, namely: seed hair (cotton), bast fibers (*Hibiscus sabdariffa*, jute, and flax), and leaf fibers (sisal, pine needles, and abaca). Natural fibrous materials, because of specific eco-friendly advantages, seem to be an alternative material to synthetic fibers in various technical applications.[11,12]

With increasing environmental awareness from the global community, there are active research interests in the studies of natural fiber-reinforced polymer matrix-based composites.[12–14] For some years, new lignocellulosic fibers have attracted considerable interest for the reinforcement of polymers matrices.[15–17] Among organic fillers, cellulose fibers offer a number of benefits as reinforcement for synthetic polymers since they have high specific strength and stiffness, noncorrosive nature, low density, low cost, reduced wear, high toughness, good specific properties, low hardness, which minimizes abrasion of the equipment during processing, and biodegradability.[18–20] Along with these properties, lignocellulosic fibers also have a few disadvantages. These lignocellulosic natural fibers are susceptible to moisture content present in the environment, which affects the properties of materials composed of these fibers. However, the main disadvantage of natural fibers is their hydrophilic nature, which lowers the compatibility with hydrophobic polymeric matrices during composite fabrication.[21–23]

In an effort to improve the existing properties of natural fibers, various surface chemical modifications such as silane treatment, mercerization, etherification acetylation, and graft copolymerization have been used most frequently.[25–27] These surface modification techniques may activate hydroxyl groups or introduce new moieties that can effectively interlock with the cellulose and induce required properties in the polymer. As a result, considerable efforts are currently being directed toward improving the quality of the interfacial bonding between the polymer and fibers by surface modification of lignocellulosic fibers.[28–30] Himachal Pradesh is one of the hilliest states in India and is blessed with vast biofibrous material. Although this Himalayan region is full of natural biomass, this precious wealth of nature has not yet been exploited for better end products. Among various types of biofibrous material, *Hibiscus sabdariffa* fibers have high potential as a renewable fabric in various applications, especially in polymer composites. Even though a very large quantity of work has been published on various natural fibers and their composites, an effort has been made in the present work to introduce a novel lignocellulosic natural fiber, i.e., *Hibiscus sabdariffa*, as one of the bast fibers and its use as reinforcement in the development of new composites for lightweight structures. Although the *Hibiscus sabdariffa* fibers are available abundantly in nature and are renewable, this potential source, economical compared to other natural fibers, is still underutilized.

The work reported in the present communication is a part of comprehensive research work initiated in our laboratory to effectively use eco-friendly biofibers in a number of applications. The overall objective of this work is to investigate the effect of silane treatment on this lignocellulosic biofibrous material.

EXPERIMENTAL

Materials and Methods

Hibiscus sabdariffa fibers were collected from local resources of the Himalayan region. Reagent grade chemicals, namely, sodium hydroxide (NaOH), ethanol, acetone, and aminopropyl triethoxy silanes were used for surface modifications of the fibers. Lignocellulosic *Hibiscus sabdariffa* fibers were initially thoroughly washed with detergent powder and then soaked in distilled water for 3 h. Then the fibers were dried for 48 h in air at room temperature followed by drying at 100°C for 24 h to adjust the moisture content to 1–2% and then stored in a vacuum dessicator.

The dried fibers were designated as untreated fibers. These untreated fibers were subsequently subjected to extraction with acetone for 72 h in a Soxhlet apparatus followed by washing with double-distilled water and air drying to remove waxes and water-soluble ingredients prior to chemical treatment.

Mercerization of *Hibiscus sabdariffa* Fibers

Generally, the first step in chemical treatment of natural fibers is the mercerization process (pretreatment process). Lignocellulosic *Hibiscus sabdariffa* fibers were immersed in 2% NaOH solution for different time intervals (45–315 min) at room temperature for the optimization of time. The fibers were then taken out and thoroughly washed with double-distilled water for removal of any NaOH content. However, the final washing was carried out with 2% acetic acid in order to neutralize the last traces of NaOH. Finally, the fibers were washed again with fresh distilled water and dried in an oven at 70°C to a constant weight. The fibers were then kept in an air drier for 24 hours. After the optimization of time, the mercerization of the fibers was also carried out in different concentration of NaOH solutions (2–14%) at the optimized time period to study the effect of the concentration of NaOH on the mercerization of the fibers.

Silane Treatment

Silane treatment of *Hibiscus sabdariffa* fibers was carried out with aminopropyl triethoxy silane. The solution of 2% of the silanes was prepared by mixing aminopropyl triethoxy silanes with an ethanol/water mixture in the ratio (60:40). This solution was allowed to stand for 1 h. The pH of the solution was maintained between 3.5 and 4, using the Metrepak pHydrion buffers and pH indicator strips. Mercerized *Hibiscus sabdariffa* fibers (2–12% NaOH) were dipped into this solution and were allowed to stand for 1.5 h. The ethanol/water mixture was drained out and the fibers were dried in air and then in an oven at 70°C to a constant weight.

Evaluation of Physical and Chemical Properties

Surface modification of natural fibers through chemical treatments has been found to affect the crystalline regions of cellulosic materials, hence it also affects many physical and chemical properties of fibers such as swelling, thermal behavior, moisture absorbance, water uptake, and chemical resistance. Silane treated/untreated *Hibiscus sabdariffa* fibers thus prepared were tested for the evaluation of physical and chemical properties such as swelling behavior, moisture absorbance behavior, water uptake, and chemical resistance.

Swelling Behavior in Different Solvents

Swelling behavior of the surface-modified and raw samples of *Hibiscus sabdariffa* fibers was determined by treating with methanol, isobutanol, carbon tetrachloride, water, etc. Known initial weights W_i of the silane-treated samples and raw sample were immersed in 100 mL of solvents at room temperature for 72 h. The samples were filtered and the excess solvent was removed with the help of filter paper, and then the final weight W_f was noted on Libror AEG-220 (Shimadzu) electronic balance.

The percent swelling was calculated from the increase in initial weight in the following manner:

$$\text{Percent swelling } (P_S) = \frac{W_f - W_i}{W_i} \times 100$$

Moisture Absorbance Study at Different Relative Humidities

Moisture absorbance studies on different natural fibers were carried out at various humidity levels (20–100). The silane-treated and raw samples of *Hibiscus sabdariffa* fibers were dried at 50°C in an oven until a constant weight was obtained. Moisture absorbance percentage was found by placing the known weight W_i of dry silane-treated and raw samples in a humidity chamber (Swastika) set at a particular humidity level for about 2 h, and then the final weights W_f of the samples exposed at a particular relative humidity (RH) were taken. The same procedure was repeated at different humidities ranging from 20 to 100%. The percent moisture absorbance was calculated from the increase in initial weight in the following manner:

$$\%\text{ Moisture absorbance } (\%M_{\text{abs}}) = \frac{W_f - W_i}{W_i} \times 100$$

Water Uptake Behavior

In order to calculate water uptake behavior, small wicks of the silane-treated and raw *Hibiscus sabdariffa* fibers were prepared, a mark was drawn on each wick, and then they were dipped into a beaker containing water. The rise of water through capillary action in each fiber wick was noted at different time intervals (12–72 h) in hours.

Chemical Resistance in 1 N NaOH and 1 N HCl

In an effort to calculate chemical resistance, 100 mg of silane-treated and raw *Hibiscus sabdariffa* fibers were put in 100 mL of 1 N NaOH and 1 N HCl, losses in weights were studied at different time intervals (12–72) in hours, and the percent chemical resistance (P_{cr}) was calculated as per the method reported earlier:

$$\text{Percent chemical resistance } (P_{\mathrm{cr}}) = \frac{T_w - W_{\mathrm{aci}}}{T_w} \times 100$$

where T_w = total weight and W_{aci} = weight after certain interval.

Characterization of Raw/Surface-Modified *Hibiscus sabdariffa* Fibers

Infrared Spectroscopy (IR)

IR spectra of the raw and surface-modified *Hibiscus sabdariffa* fibers were taken with KBr pellets on a PerkinElmer RXI spectrophotometer

Scanning Electron Microscopy (SEM)

Scanning electron microscopic (SEM) studies of raw *Hibiscus sabdariffa* fibers were carried out on Electron Microscopy Machine (LEO 435 VP). The excitation energy used was 5 keV. Since these materials are nonconducting, to achieve good electric conductivity all samples were first carbon sputtered followed by sputtering a gold/palladium mixture before examination. Scanning was synchronized with a microscopic beam in order to maintain the small size over a large distance relative to the specimen. The resulting images had a great depth of field. A remarkable three-dimensional appearance with high resolution was obtained.

Thermal Analysis

Thermal analysis of natural and synthetic polymers gives us a good account of their thermal stability. Thermogravimetric analysis (TGA), differential thermal analysis (DTA), and derivative thermogravimetric (DTG) studies of the samples were carried out in nitrogen atmosphere on a thermal analyzer (PerkinElmer) at a heating rate of 10°C/min. TGA is used to characterize the decomposition and thermal stability of the materials under a variety of conditions and to examine the kinetics of the physicochemical processes occurring in the sample. At the same time, DTA involves comparison of the precise temperature difference between a sample and an inert reference material while heating both. DTG is a type of thermal analysis in which rate of material weight change upon heating versus temperature is plotted and is used to simplify reading of weight versus temperature (thermo$_{\text{max}}$) and the peak onset temperature (T_e). The area under the DTG curve is proportional to the mass change, and the height of the peak at any temperature gives the rate of the mass change at that temperature. DTG curves are frequently preferred when comparing results with DTA curves because of the visual similarity.

RESULTS AND DISCUSSION

It has been reported that the availability of the number of active sites on the polymer backbone of natural fibers affects the extent of the surface modifications such as grafting, mercerization, and silane treatment. The most important variables that affect mercerization and silane treatments are the reaction parameters like reaction time and concentration of NaOH. Extensive studies were made to optimize these parameters for further carrying out the silane treatment of *Hibiscus sabdariffa* fibers for application in various fields.

Effect of Reaction Time

The reaction time parameter very much affects mercerization. Reaction time has been optimized by determining the maximum weight loss for *Hibiscus sabdariffa* fiber. Percentage weight loss of *Hibiscus sabdariffa* fibers as a function of time is presented in Table I.

Maximum mercerization of *Hibiscus sabdariffa* fibers was observed at 180 min, as shown in Table I. The decrease in weight loss beyond optimum reaction time is due to the excessive deposition of sodium ions

Table I. Effect of reaction time on mercerization of *Hibiscus sabdariffa fibers*

Sr. no.	Time (min)	% Wt. loss
1	0	0
2	45	3.76
3	90	5.49
4	135	8.04
5	180	9.52
6	225	8.90
7	270	8.94

over surface modification and other side reactions, which may not favor further weight loss.

Evaluation of Physical and Chemical Properties of Silane-Treated *Hibiscus sabdariffa* Fiber

Silane-treated and raw *Hibiscus sabdariffa* fibers were subjected to evaluation of physical and chemical properties such as swelling behavior, moisture absorbance behavior, water uptake, and chemical resistance.

Swelling Behavior Study of *Hibiscus sabdariffa* Fibers

Swell ability is the relationship between void structure in backbone polymer and size of solvent molecules both for protic and aprotic solvents.[25] It has been observed that after silane treatment, the swelling behavior of raw fibers decreases. Raw *Hibiscus sabdariffa* fibers showed maximum swelling in water (110.71%) followed by swelling in methanol (64.00%), isobutanol (59%), and carbon tetrachloride (44.53%). However, silane-treated fibers showed less swelling, as seen in Table II. Further, it has been observed that 6% mercerized silane-treated *Hibiscus sabdariffa* fibers showed lowest swelling followed by 8, 10, 4, and 2% respectively. From the above discussion, it has been observed that raw *Hibiscus sabdariffa* fibers showed maximum swelling in water over other solvents. This is due to greater affinity of water for hydroxyl groups present in natural fiber cellulose, resulting in more penetration of water into its matrix than in other solvents. In the case of silane-treated fibers, it has been observed that swelling behavior varies as a function of percentage mercerization (Pm) and nature of solvent. In silane-treated fibers, water and alcohols do not interact to the same extent as with raw fibers due

Table II. Effect of silane treatment on % swelling behavior of *Hibiscus sabdariffa* fibers in different solvents

		% Swelling (in terms of weight)			
Sr. no.	% Mercerization	Water	Methanol	Isobutanol	Carbon tetrachloride
1	0	110.71	64.00	59.47	44.53
2	2	100.14	52.42	47.53	39.28
3	4	76.71	46.36	34.93	29.37
4	6	60.14	29.37	26.38	22.37
5	8	65.28	35.82	30.38	25.38
6	10	71.71	37.79	33.48	29.48

to blockade of the active sites on the natural fiber backbone by silane treatment, which causes change in the sorption behavior. In particular, for raw or treated fibers, the percentage of swelling found decreased from H_2O to methanol and then to isobutanol and carbon tetrachloride, respectively. This may be due to the fact that the nonpolar hydrocarbon part, i.e., alkyl group, resists the formation of hydrogen bond and such resistance increases with the size of alkyl group.

Moisture Absorbance Study of *Hibiscus sabdariffa* Fibers

Raw *Hibiscus sabdariffa* fibers showed 1.57, 1.98, 2.92, 3.29, and 3.85% moisture absorbance at 20, 40, 60, 80, and 100% humidity levels, respectively. However, silane-treated fibers exhibited less moisture absorption, as shown in Table III. Further, it has been observed that 6% mercerized silane-treated *Hibiscus sabdariffa* fibers showed lowest moisture absorbance followed by 8, 10, 4, and 2%, respectively.

From the above data, it has been observed that silane treatment of raw *Hibiscus sabdariffa* fibers has a great impact on the moisture absorbance behavior. There was a decrease in percent moisture absorbance (% Mabs.) with increase in silane treatment (% Mercerization), which is due to the fact that with increase in Pm, the sites for maximum moisture absorbance are blocked after incorporation of silane chains through surface modification by showing less affinity for moisture than original fibers.

Water Uptake Behavior of *Hibiscus sabdariffa* Fibers

Raw *Hibiscus sabdariffa* fibers showed water uptake of 7.03, 9.97, 12.17, 14.21, 16.04, and 17.03 cm at 12, 24, 36, 48, 60, and 72 h respectively. However, silane-treated fibers showed less water uptake, as shown in Table IV. Further, it has been observed that 6% mercerized silane-treated

Table III. Effect of silane treatment on % moisture absorption behavior of *Hibiscus sabdariffa* fibers at different relative humidity levels

		% Humidity levels (in terms of weight)				
Sr. no.	% Mercerization	Hum = 20	Hum = 40	Hum = 60	Hum = 80	Hum = 100
1	0	1.57	1.98	2.92	3.29	3.85
2	2	1.39	1.75	2.64	3.21	3.64
3	4	1.07	1.68	2.55	2.98	3.53
4	6	0.87	1.41	2.26	2.54	2.92
5	8	0.95	1.49	2.37	2.67	3.27
6	10	1.02	1.57	2.42	2.78	3.41

Table IV. Effect of silane treatment on water uptake behavior of *Hibiscus sabdariffa* fibers at different time intervals

		Water uptake (in terms of cm)					
Sr. no.	% Mercerization	12 h	24 h	36 h	48 h	60 h	72 h
1	0	7.03	9.97	12.17	14.21	16.04	17.03
2	2	6.14	8.35	11.54	14.04	15.86	16.87
3	4	5.87	7.97	11.62	13.64	15.63	16.69
4	6	5.38	7.68	10.53	13.07	15.07	16.05
5	8	5.54	7.79	10.83	13.38	15.35	16.31
6	10	5.69	7.88	11.38	13.53	15.51	16.49

fibers showed lowest water uptake, followed by 8, 10, 4, and 2%, respectively.

Chemical Resistance Behavior of *Hibiscus sabdariffa* Fibers

Raw *Hibiscus sabdariffa* fibers showed 17.52, 22.85, 30.96, 35.79, 43.85, and 48.64% weight loss in 1 N HCl and 23.47, 29.45, 38.94, 45.23, 51.62, and 56.94% weight loss in 1 N NaOH at 12, 24, 36, 48, 60, and 72 h respectively. However, silane-treated fibers showed less weight

Table V. (a) Effect of silane treatment on chemical resistance (in terms of % wt. loss) of *Hibiscus sabdariffa* fibers at different time intervals against 1 N HCl, (b) Effect of silane treatment on chemical resistance (in terms of % wt. loss) of *Hibiscus sabdariffa* fibers at different time intervals against 1 N NaOH

Sr. no.	% Mercerization	12 h	24 h	36 h	48 h	60 h	72 h
			(a)				
1	0	17.52	22.85	30.96	35.79	43.85	48.64
2	2	14.47	21.54	29.47	34.73	42.57	47.26
3	4	13.85	20.93	28.09	33.87	41.26	45.95
4	6	11.96	18.25	25.84	31.28	38.95	42.64
5	8	12.65	19.83	26.67	32.35	39.75	43.94
6	10	13.28	20.07	27.62	33.27	40.27	44.37
			(b)				
1	0	23.47	29.45	38.94	45.23	51.62	56.94
2	2	19.54	28.23	37.98	44.04	50.37	55.63
3	4	18.64	26.91	36.12	43.62	49.13	54.23
4	6	15.35	23.57	32.93	40.93	44.78	52.17
5	8	16.64	24.86	34.72	41.27	46.83	53.01
6	10	17.93	25.83	35.02	42.29	47.94	53.42

loss, as seen in Table V. Further, it has been observed that 6% mercerized silane-treated fibers showed lowest weight loss followed by 8, 10, 4, and 2%, respectively.

Characterization of Raw and Surface-Modified Natural Fibers

Infrared Spectroscopy (IR)

IR spectra of raw *Hibiscus sabdariffa* fiber showed a broad peak at 3415.4 cm^{-1} due to the bonded OH groups and at 2923.3, 1426.5, and 1031.0 cm^{-1} due to the –CH2, C–C, and C–O stretching, respectively. In the case of silane-grafted fibers, additional broad intense bands around 1220 and 1125 cm^{-1} were obtained due to the stretching of Si–O–cellulose and –Si–O–Si bonds, respectively.

Morphological Study

It has been observed that surface modification of *Hibiscus sabdariffa* fibers through silane treatment gave surface coating to the fibers, and surface features of fibers are not clearly visible. The SEM micrographs easily verified the difference between *Hibiscus sabdariffa* fibers and surface-modified fibers (Figure 1).

Thermal Analysis

In thermal analysis of *Hibiscus sabdariffa* fibers, the initial decomposition temperature (IDT) has been found to be 199°C (percent weight loss 7.65) and the final decomposition temperature (FDT) to be 500°C (percent weight loss 85.71). In the case of silane-treated fiber, the IDT has been found to be 197°C (percent weight loss 9.30) and the FDT to be 512°C (percent weight loss 56.46) (Figures 2 and 3).

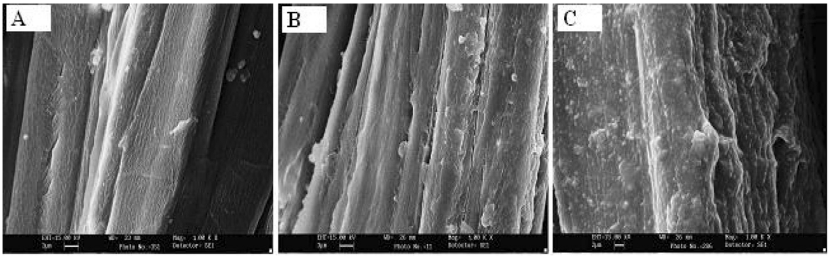

Figure 1. SEM of raw, mercerized, and silane-treated *Hibiscus sabdariffa* fibers (A, B, and C).

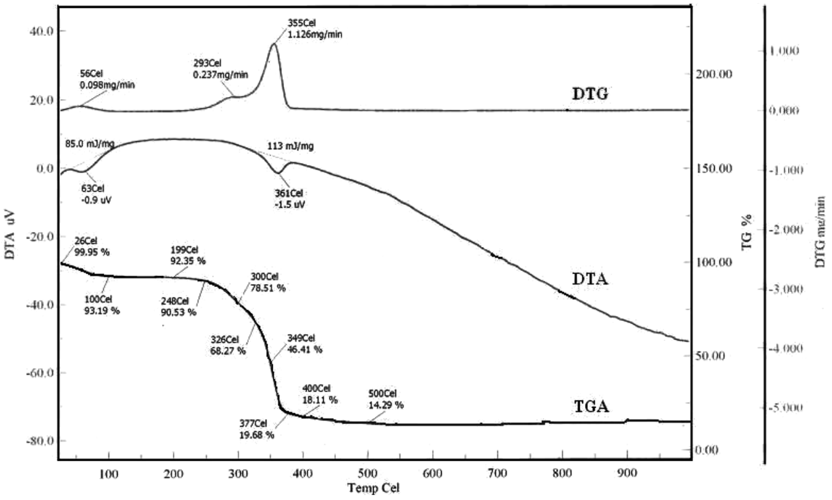

Figure 2. TGA/DTA/DTG analysis of raw *Hibiscus sabdariffa* fiber.

It has been observed that physical and chemical properties of *Hibiscus sabdariffa* fibers are greatly affected by silane treatment. The activation of lignocellulosic *Hibiscus sabdariffa* fibers has been carried by mercerizing these fibers with different concentrations of sodium hydroxide for different intervals prior to carrying out silane treatment so as to open the active sites of the cellulose backbone for surface treatment. Alkaline

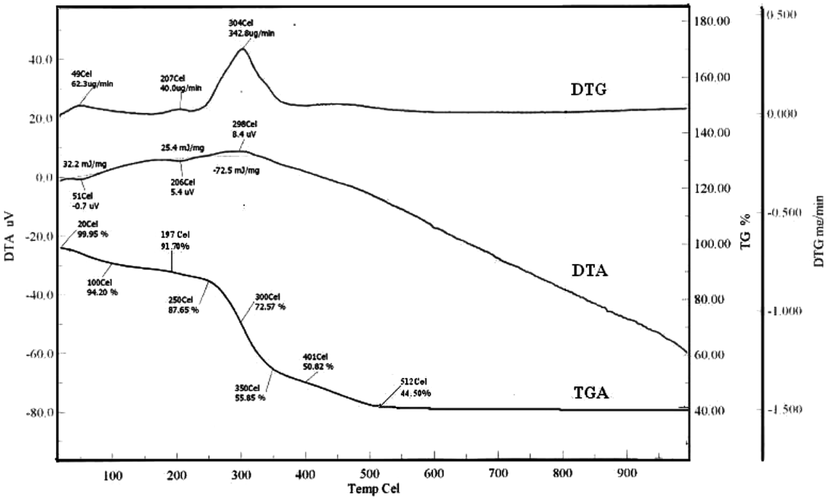

Figure 3. TGA/DTA/DTG analysis of silane-treated *Hibiscus sabdariffa* fiber.

treatment increases the surface roughness in given lignocellulosic natural fiber by disrupting the hydrogen bonding in the network structure. This treatment removes a certain amount of lignin, wax, and oils covering the external surface of the fiber cell wall, depolymerizes cellulose, and

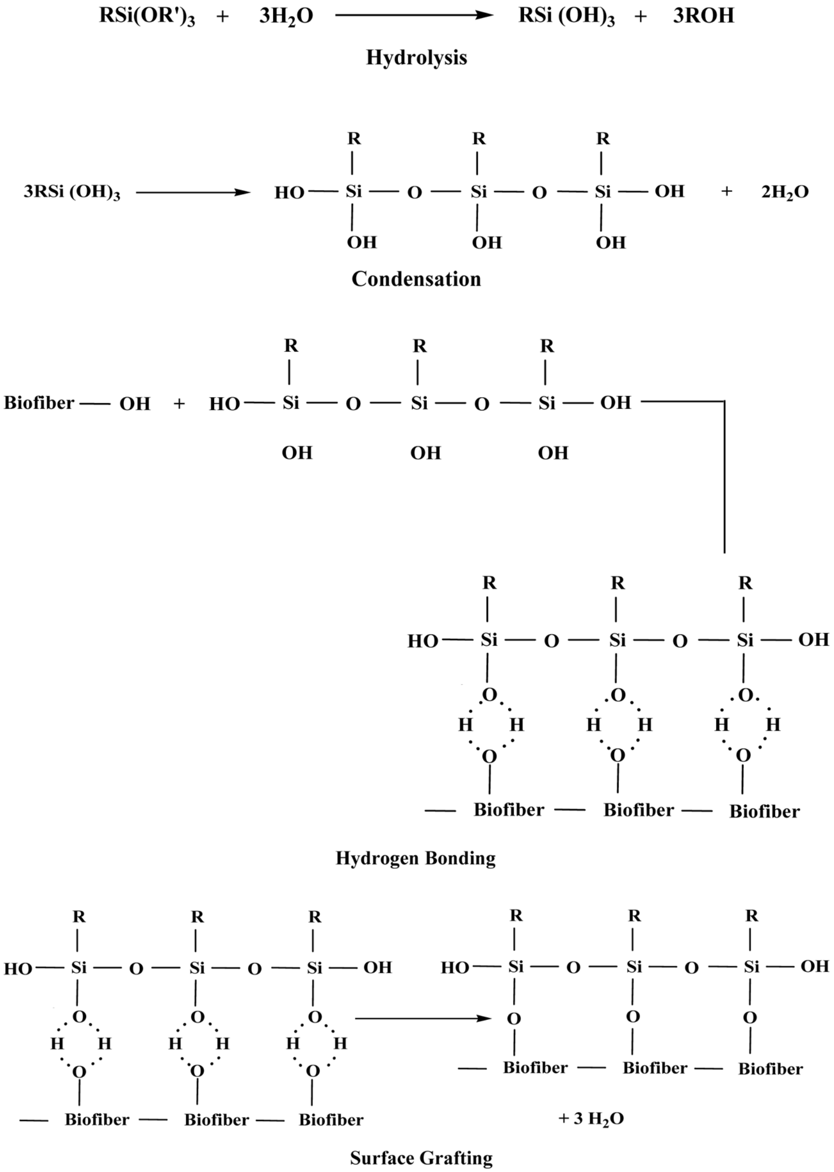

Figure 4. Various steps in surface modification of *Hibiscus sabdariffa* fiber.

exposes the short length crystallites. An addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide.

Otherwise, without such activation only a small amount of surface modification occurred, which is due to the fact that natural fibers, being lignocellulosic fibers, are highly hydrogen bonded polymers, and silane molecules are not in a position to penetrate beyond the surface of the cellulose substrate. In the present study, the modification of the lignocellulosic fibers has been attempted by chemical treatment through silane for the fabrication of natural fiber-reinforced polymer composites. Silane coupling agent helps in improving the degree of cross-linking in the interface region and offers a perfect bonding result. Alkoxy silanes are able to form bonds with hydroxyl groups. Silane coupling agents may reduce the number of cellulose hydroxyl groups in the fiber interface. In the presence of moisture, hydrolyzable alkoxy group leads to the formation of silanols. The silanols then react with the hydroxyl group of the fiber, forming stable covalent bonds to the cell wall that are chemisorbed onto the fiber surface. Therefore, the hydrocarbon chains provided by the application of silane restrain the swelling of the fiber by creating a cross-linked network due to the covalent bonding. Further, since *Hibiscus sabdariffa* fibers exhibited micro pores on their surface, the aminopropyl triethoxy silane coupling agent penetrated into the pores and formed a mechanically interlocked coating on their surface. The chemical treatment of lignocellulosic fibers results in formation of complexes with the hydroxyl group of the cellulose and thus reduces moisture absorption of the fiber. Silanes undergo hydrolysis, condensation, and bond formation stage. Silanols can form polysiloxane structures by reaction with hydroxyl group of the fibers. The reaction schemes for silane treatment of natural fibers of *Hibiscus sabdariffa* are given in Figure 4.

CONCLUSION

Important progress has been made in the past in establishing the fundamental material-process interactions in the field of high-performance natural fibers. However, a significant degree of incongruity still exists especially with regard to the effective utilization of natural fibers such as *Hibiscus sabdariffa*, which have been used in a number of applications for hundreds of years but still have not attracted attention from the research community. Research carried out shows that these fibers are high potential candidates for the effective utilization in a number of applications. It has been observed that the properties such as chemical resistance, moisture absorbance, swelling behavior, and water uptake vary directly with the silane treatment. Surface modification of fibers through silane treatment helps in improving the physicochemical properties of natural fibers. In spite of some limitations, the *Hibiscus sabdariffa* fibers can be a suitable alternative to toxic synthetic fibers. There is a need of a distinct and more comprehensive approach for the development of *Hibiscus sabdariffa* fibers for use in the high technology sector.

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