A New Study on Effect of Various Chemical Treatments on Agave Americana Fiber for Composite Reinforcement: Physico-Chemical, Thermal, Mechanical and Morphological Properties

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ABSTRACT

This research is focused to fundamentally understand the benefits of using Agave Americana C. plant as potential reinforcement in polymeric composites. The fibers were extracted from the external part of the bark of the plant, which grows worldwide in pastures, grasslands, open woodlands, coastal and riparian zones. In order to use the natural fiber as reinforcement it is paramount important to probe their chemical composition, microstructural behavior and mechanical properties. Hence, firstly the extracted fibers were chemically treated with NaOH, stearic acid, benzoyl peroxide and potassium permanganate. The chemical composition in terms of cellulose, hemicellulose, lignin and other waxy substances were determined using a standard TAPPI method. FT-IR technique was used to understand the character of molecular bonds, crystallinity and their correlations with various bonds in fiber structure. The thermal stability was investigated through thermogravimetric and differential scanning calorimetric analysis, and the mechanical characterization was performed by applying standard tensile test. The surface morphology of fibers was examined through scanning electron microscopy (SEM) and finally reliability scrutiny of all the analysis was carried out. The results of chemical modification techniques applied on the surfaces of natural fibers allows to produce superior fibers used to form the novel composite materials for light- weight application.

Keywords: Agave Americana fibers; Chemical Analysis; FTIR; TGA; SEM.

1. Introduction

Owing to the customers' demands for more energy efficient, lighter in weight, biodegradable, renewable and carbon sequestering materials, the industry is focusing their attention towards using more ecological and environmentally friendly materials [1]. Adding to these demands, the rapid depletion of fossil fuels and new government policies, which require the use of more materials produced from non-renewable sources, make the green materials attractive particularly in the manufacturing of novel composite materials [2, 3]. In this context, the natural fibers used as reinforcements in polymer-based composites have been found to be very attractive and being used drastically used over the past few decades by several industrial sectors in manufacturing of their commercial products [4]. Natural fibers have attractive features like good thermo-mechanical properties, high electrical resistance, good acoustic insulating behavior and higher fracture resistance properties make them suitable materials to be used in production of many industrial items that include wind turbine blade, window and door frames, electrical and electronic industries, railway sleepers, automotive components, furniture, etc. [5-7]. Several researchers have investigated on different types of natural fibers like coir, hemp, banana, borassus, sisal, jute, tamarind, flax, kapok, kenaf, etc., as possible reinforcements in polymer composites. Although natural fibers possess certain advantages as mentioned above, they suffer some drawbacks while using in polymer matrix composites such as hydrophilic nature, high moisture sorption, poor resistance to high temperatures, variability in fiber properties and weak bonding with hydrophobic polymeric matrices [4, 8, 9]. Thus in order to overcome these problems and to improve the compatibility between natural fibers and polymer matrices certain surface modification processes like physical (surface fibrillation

through mechanical beating, laser treatment, ozone treatment and plasma treatment) and chemical (alkali treatment, saline treatment, acetylation, benzoylation, potassium permanganate treatment, stearic acid treatment and polymer coatings) treatment methods are being used [2, 10].

Chemical treatment methods are generally based on the utilization of reagent functional groups which are capable of reacting superiorly with natural fiber structures as well as effectively remove non-cellulosic materials from the fibers. Thus, this surface modification technique helps the fibers to have better bonding with polymeric matrices [11, 12]. The alkali treatment is one of the most effective chemical treatment technique employed to remove hemicellulose and other impurities from fibers thereby leading to enhanced cellulose content which in turn results in improved mechanical properties [1]. The adhesion properties between hydrophilic fiber and hydrophobic matrix can be enhanced by peroxide treatment. Short sisal fibers were chemically treated with diluted 6% benzoyl peroxide and 4% dicumyl peroxide and resulted in improved tensile strength properties [14]. Potassium permanganate (KMnO4) treatment results in the reaction of permanganate ions with hydroxyl groups and initiates graft copolymerization with the formation of cellulose-manganate. Further, this treatment improves interfacial adhesion characteristics between fibers and matrix [15]. Another important method used for fiber surface modification is stearic acid treatment where the carboxyl groups of stearic acid reacts with hydroxyl groups in the fibers through esterification reaction and results in removal of non-crystalline materials like waxes; oils etc., as well as improves water resistance properties [11, 16]. The modification techniques are going to affect the fiber mechanical properties, their thermal behavior, crystalline structure, and morphology, this has been studied and reported by many researchers in their work.

The fiber studied in the present work is extracted from the leaves of Agave americana C. plant. Agave Americana fibers are one of the strongest, heat & drought tolerant and stiffest. These fibers have a strong potential to be used as reinforcement in polymer composites. It is a monocotyledonae plant belonging to Agavaceae family and is widespread in the tropical, and subtropical regions. It grows up to 3-6 ft. long (90-180 cm) and 6-10 ft. wide (180-300cm). These fibers are being used in used in ropes, carpets, doormats, fish stringers, saddle pads, brush brittles, baskets, headbands, sandals, and textile applications and can be extracted from the leaves either by heating the leaves in hot water or retting them in seawater or through various chemical and mechanical methods [17]. Research findings show that Agave Americana C. plant leaf fibers can be utilized as efficient reinforcements in polymer composite materials [18].

In this study, leaf fibers extracted from Agave Americana C. plant were subjected under four different chemical treatment methods (NaOH, peroxide, permanganate and stearic acid) and their effects on the chemical composition, structural characteristics, crystallinity, thermal degradation, tensile properties and surface morphology were explored by chemical analysis, Fourier transform-infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), universal testing machine (UTM), and scanning electron microscopy (SEM) respectively.

2. Materials and Methods

2.2. Extraction of Agave Americana C. fibers

The fresh leaf fibers from Agave Americana C. plant were collected from the village of Virudhunagar district situated near Madurai Road, Tamil Nadu, India. The fibers from the obtained leaves were extracted through distilled water method. First the leaves were subjected to a hydrolysis treatment by using distilled water. Later fibers were removed from the matrix by calendering the leaves. Finally, the fibers were washed with distilled water. This fiber removal process was carried out under the following conditions: temperature of 120^0 C for duration of one and half hour [19]. All other chemicals for surface modification were obtained from Sri Raghavendra Chemicals, Bengaluru, Karnataka, India.

2.3. Fiber treatment of Agave Americana leaf Fibers (AAFs)

The AAFs were treated with four different chemical solutions and these are discussed in this section [1].

2.4. NaOH Treatment

Dried AAFs were immersed in a 6% (w/v) NaOH (sodium hydroxide) solution for 45 min. They were then washed with condensed water to eradicate any traces of NaOH from AAFs. The treated AAFs were finally dried in an oven at 80^0 C for 1 h.

2.5. Benzoyl Peroxide–Treatment

AAFs were soaked with 6% benzoyl peroxide and acetone solution for 30 min. The treated fibers were then dried in air for 24 h.

2.6. Potassium Permanganate–Treatment

The dried AAFs were rinsed in 0.5% potassium permanganate with acetone for 30 min and later were dried in air.

2.7. Stearic Acid–Treatment

First the dried AAFs were placed in a stainless steel container and 1% stearic acid solution liquefied in ethyl alcohol was added drop by drop through continuous stirring for 45 min. The treated fibers were finally dried in an oven at 80^0 C.

3. Characterization Study

3.1. Chemical Analysis

The chemical analysis is carried out to understand the AAFs cellulose and other amorphous contents. Cellulose, hemicellulose and lignin were determined using standard TAPPI methods. Ash wax content was determined using ASTM E 1755-01 method while the wax content was quantified using Conrad method. Moisture content was examined through an electron moisture analyzer (Sartorious, model MA45) and density in the raw and various treated AAFs was measured with the help of meltbertoledoxsz05 balance method [21, 22]. In each treatment groups, five fiber samples were considered and average values are reported.

3.2. Fourier Transform-Infrared Spectroscopy (FTIR) Analysis

FTIR analysis was performed using a Shimadzu 8400S FTIR spectrometer. A total of 32 samples were taken for every sample with a resolution of 2 cm^{-1}. Fine powdered 2 mg of dried fibers per 150 mg KBr were pressed into a disc for FTIR quantification.

3.3. Thermogravimetric Analysis (TGA)

Thermograms of raw and modified AAFs in were recorded using a thermogravimetric analyzer (Jupiter simultaneous thermal analyzer, model STA449 F3), at a heating rate of 10^0 C/min in the temperature range of 25^0 C to 700^0 C, under pure nitrogen atmosphere.

3.4. Differential Scanning Calorimetry (DSC) Analysis

Mettler DSC 822e equipment was used for DSC analysis of raw and modified AAFs. About 2 to 3 grams of fiber samples in each treatment groups were conserved in aluminium pans and heating rate was maintained at 10^0 C/min during analysis. The samples were heat scanned at temperature ranging 50^0 C to 700^0 C and placed inside a heat scan holder [25].

3.5. Tensile Testing

Universal testing machine (ASTM D 3379 standard) was used to measure the tensile strength of raw and modified AAFs [26], while the diameter of the fibers was determined using polarized light microscope. All the tests were performed at ambient temperature with relative humidity

of about 65%. A minimum of 20 samples in both raw and modified groups were taken to conduct the tensile tests.

3.6. Scanning Electron Microscopy (SEM)

To evaluate the alterations made on the agave fiber surfaces by various chemical treatments, raw and modified AAFs were scrutinized by VEGA 3 TESCAN scanning electron microscopy operating at 10 kV. All the samples were enclosed in a thin gold coating to avoid electrostatic charge and to enhance image resolution.

3.7. Contact angle measurement

The OCA 15EC data physics-contact angle system was used to measure the water contact angle of the AAFs that measures the wettability of the liquid over the fiber surface. For contact angle studies, 2 μl of distilled water were placed as a sessile drop on the samples. An average of 10 samples were taken as the contact angle values for each treatments.

4. Results and Discussion

4.1. Chemical constituents and density analysis

All lignocellulosic fibers are renowned by their crystalline and amorphous structure. The strong hydrophilic hydroxyl groups and hemicellulose, lignin, pectin and other waxy substances present in this crystallite and amorphous regions are going to lower the compatibility with the hydrophobic matrix [11. 12, 27]. Furthermore, cellulose constituent plays a very important role

in providing the necessary strength and stiffness to natural fibers and hemicellulose is accountable for moisture absorption and thermal degradation in the fibers. The fiber structural behaviour and its morphological property are authorized by its lignin content [28]. The chemical composition and density of raw and different chemically treated AAFs was determined and capitulated in Table 1. From the results it is clear that the raw fibers have high percentage of hemicellulose and lignin content, while have lower cellulose content. Upon various chemical modification techniques used, the hemicellulose and lignin content decreases while the cellulose content has increased considerably in all the treated fibers showing partial hydroxylation and depolymerization in hemicellulose and lignin content respectively [19]. The wax and moisture content of all the treated AAFs show ascending values compared to raw fibers which implies that treated fibers will provide improved adhesion and closed crystalline packing between fiber and matrix. The decreased ash contents in chemically treated AAFs directly impacts on its mechanical behavior [29]. It is observed that the density of AAFs increased upon chemical treatments and can be attributed due to densification of fiber cell walls and filling up of pores by grafted molecules [3].

Table 1. Chemical constituents and density of raw and various chemically treated AAFs

Fiber	Cellulose (wt.%)	Hemicelluloses (wt.%)	Lignin (wt.%)	Wax (wt.%)	Moisture Content (%)	Ash (wt.%)	Density (Kg/m^3)
Raw	68.54±3.46	18.41±2.28	6.08±3.39	0.56±0.23	9.32±1.02	3.29±2.11	1035±23.32

NaOH	78.65±2.68	8.47±3.11	4.65±2.38	0.46±0.21	8.04±1.78	4.43±2.36	1041±22.68
SA	81.65±3.59	6.31±2.13	3.43±1.38	0.37±0.35	7.04±2.78	5.21±1.28	1043±13.78
BP	80.26±3.33	7.42±2.11	4.33±1.67	0.42±0.11	7.79±0.69	4.65±1.78	1039±12.44
PP	79.78±2.48	6.67±3.32	4.10±2.44	0.22±0.11	7.98±0.22	4.99±0.78	1044±24.56

4.2. FT-IR Analysis

The existence of several functional groups in raw and chemically treated AAFs is detected using FTIR analysis. FTIR spectra of raw, NaOH, stearic acid (SA), benzoyl peroxide (BP) and potassium permanganate (PP) treated AAFs are described in Figure 1. The band peaks have been assessed from 4000 and 500 cm^{-1}. The typical wide peak between 3427 to 3100 cm^{-1} in all treated and raw AAFs is linked to OH stretching of α-cellulose and can be attributed to hydrogen bonded cellulose structure while, the peak 2917 cm^{-1} is ascribed to C-H stretching vibration group of cellulose [31]. The absorption band peak 2357 cm^{-1} is evidenced only in raw AAF and barely visible in modified AAFs exemplifies the presence of wax and other impurities in raw fibers and is related to C ≡ C stretching of wax. The decreasing peaks 1602 cm^{-1} and 1552 cm^{-1} is seen in all treated and raw fibers and corresponds to C=O stretching vibration of the amide group in hemicellulose and lignin respectively. The chemical treatments usually lessen the hydrogen bonds from cellulose hydroxyl group along with the elimination of carboxyl group in fibers and this is noticeable around the peak 1022 cm^{-1} and is correlated with C-OH stretching of lignin [2, 5]. The peaks and their allocations are shown in Table 2.

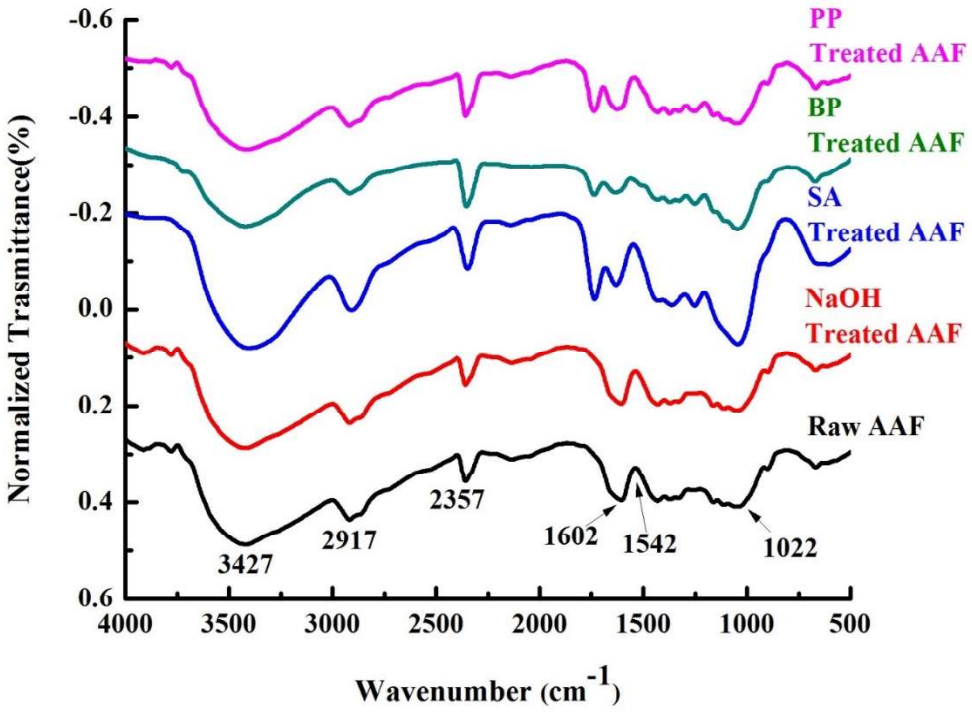


Figure 1. FTIR spectra of raw, NaOH, SA, BP and PP treated AAFs

Table 2. The peaks used for FT-IR analysis and possible allocations of raw, NaOH, SA, BP and PP treated AAFs

Raw AAF	NaOH	SA	BP	PP	Possible allocations
3427	3427	3427	3427	3427	OH-stretching
2917	2917	2917	2917	2917	CH stretching
2357	-	-	-	-	C ≡ C stretching
1602	1602	1602	1602	1602	C=O stretching (Amide)
1552	1552	1552	1552	1552	C=O stretching
1022	1022	1022	1022	1022	C-OH stretching

4.3. TGA

TGA curves of raw and treated AAFs are depicted in Figure 2. From the curves, it is evident that thermal degradation occurred in 3 stages. The initial weight loss was seen at below 100^0 C and the weight loss was around 5-8%. The 1^{st} stage of thermal degradation process was experienced between 50^0 C and 185^0 C and could be due to the eradication of moisture content and some waxy materials from the fibers and the weight loss was around 15% in this temperature range. Next, the 2^{nd} degradation stage progressed at the temperature ranging 220^0 C and 340^0 C and may be due to degradation of hemicellulose and α- cellulose [31-33]. The corresponding weight loss in this temperature ranges for raw and NaOH treated fiber was around 50% and for SA, BP and PP treated fibers was around 55-60%. The last degradation stage was seen at temperature range 370^0 C and 490^0 C and corresponded to the degradation of lignin and other non-cellulosic materials present in the fibers [34]. TGA curve in Figure 3 confirm that AAFs have better thermal stability properties.

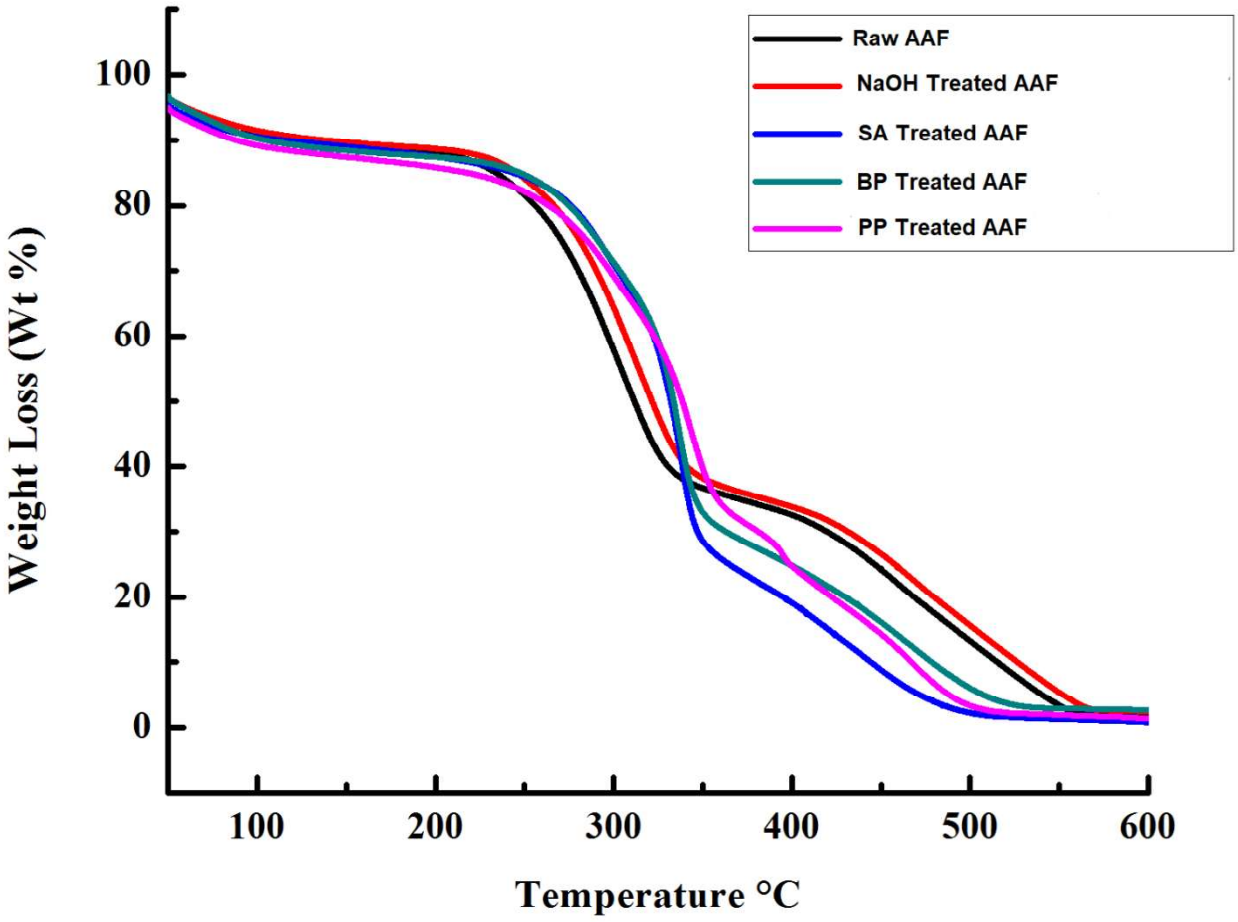


Figure 2. TGA curves of raw and chemically treated AAFs

4.4. DSC Analysis

DSC analysis curves of raw and treated AAFs are depicted in Figure 3. This curve justifies the improvement in cellulose and amorphous fractions (hemicellulose and lignin) degradation temperature and authenticates the elimination of hydrophilicity properties in chemically treated fibers and also shows overall high heat generation in them. The endothermic peak was seen at around 310^0 C and 400^0 C and represented the loss of moisture from the fibers and

exothermic peak at around 570^0 C endorses the complete decomposition of cellulose, hemicellulose and lignin in treated and raw AAFs.

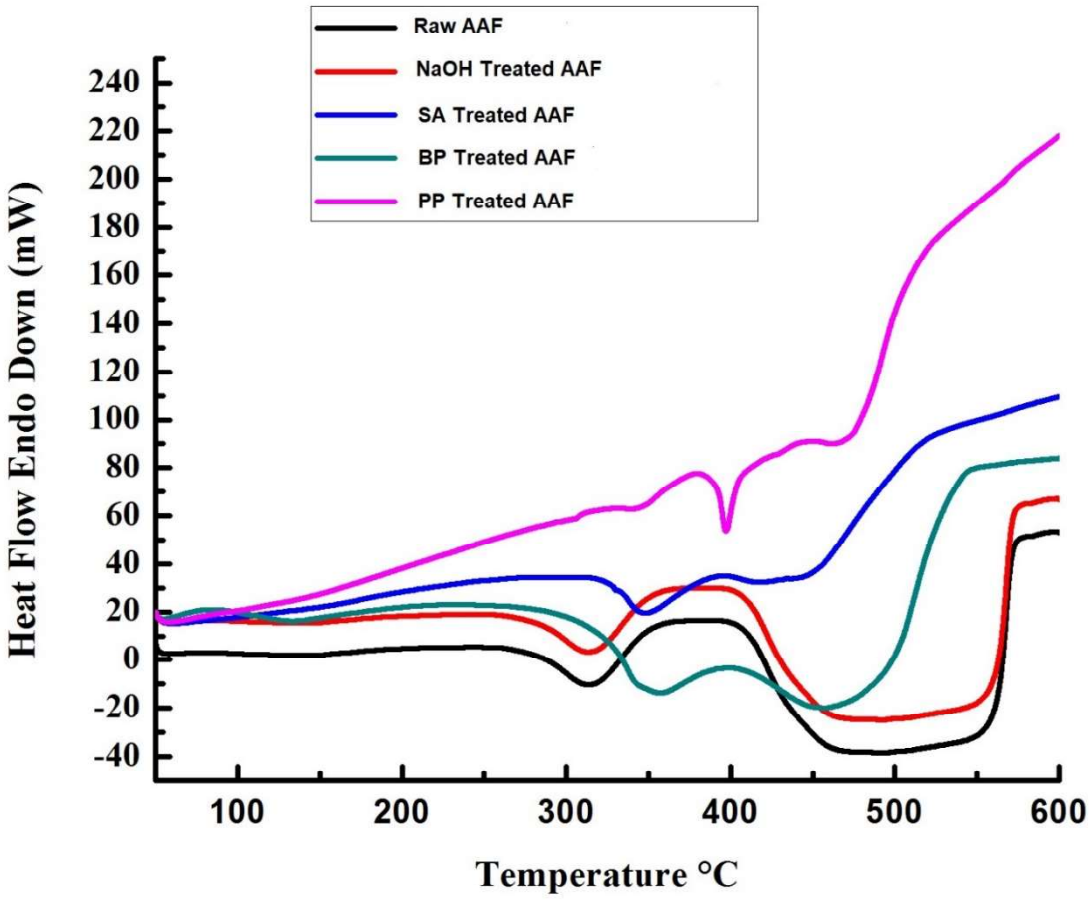


Figure 3. DSC curves of raw and modified AAFs

4.5. Tensile Testing

The tensile properties (strength, young's modulus and elongation at break) and diameter of raw and NaOH, SA, BP, PP treated AAFs are determined and tabulated in Table 3.

From the Table 3, it is seen that all the treated fibers diameters were simultaneously decreased compared to raw fibers. The 6% concentration of NaOH was most effective surface modification among all the treatments.

The average of five samples were chosen from raw and various treatments for tensile test, respectively. From Table 3, it is quantified that the average tensile strength and elongation at break of chemically treated AAFs are higher than those of raw fibers and this can be credited to the fibers tendency towards becoming closely packed due to the removal of hemicellulose by various chemical treatments. The young's modulus is found to be higher in raw fibers, while there is not much significant difference among the chemically treated fibers. The tensile values of AAFs exhibit good properties and tend to be suitable reinforcements in polymer matrices [35, 36].

Table 3. Tensile properties of raw and chemically treated AAFs

Parameter	Raw	NaOH	SA	BP	PP
Diameter (μm)	190±2.76	170±1.98	162±2.43	166±2.46	169±1.88
Tensile strength (MPa)	282±9.34	288±6.54	293±5.67	290±98	289.98±3.78
Young's modulus (GPa)	8.4±2.67	7.99±3.45	7.83±2.65	7.60±3.56	7.89±2.11
Elongation at break (%)	3.4±2.56	3.55±2.78	3.88±2.22	4.1±3.44	3.97±3.43

4.6. SEM Analysis

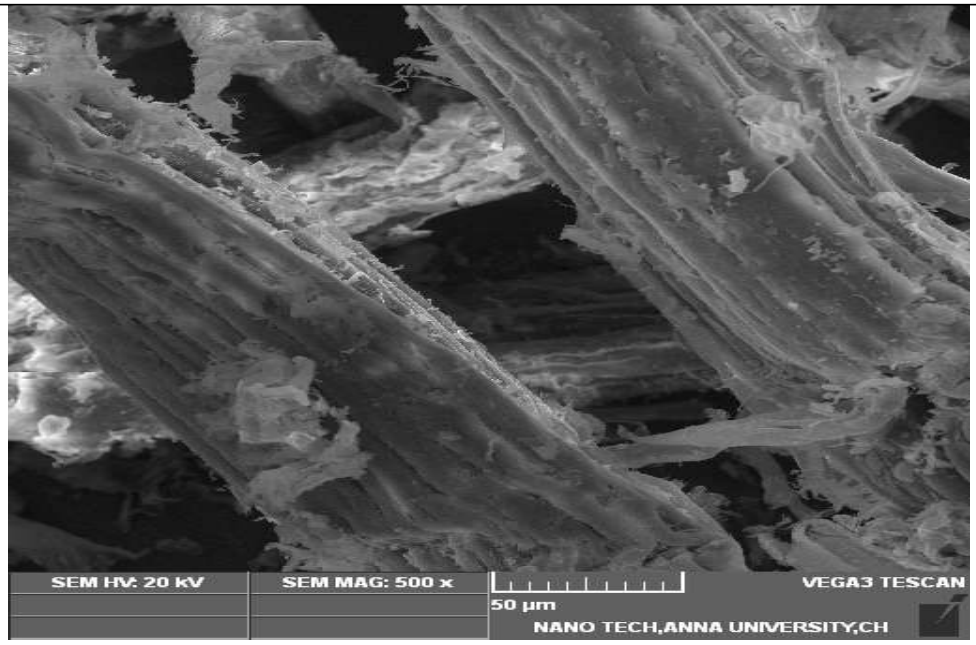
Scanning electron microscopy provides an excellent technique to examine the morphology of the fiber surface. Figure 4 (a) presents the surface morphology of raw AAFs and Figure 4 (b) to (e) shows the micrographs of AAF treated with sodium hydroxide, stearic acid, benzoyl peroxide, and potassium permanganate respectively. Figure 4 (b) to (e) presents the

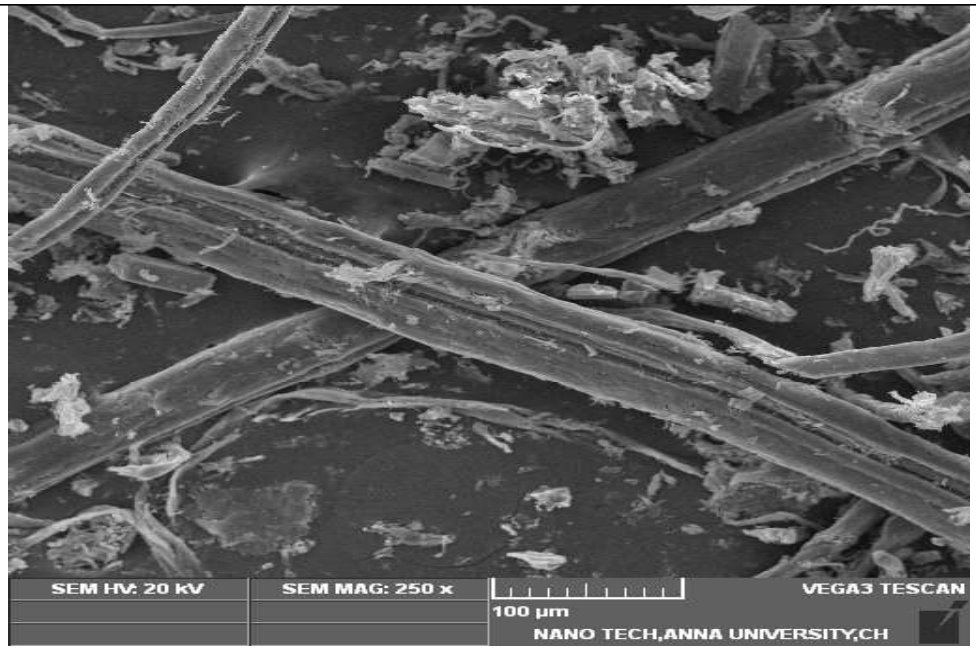
micrographs of AAF treated with sodium hydroxide, benzoyl peroxide, potassium permanganate and stearic acid, respectively.

From Figure 4 (a), it can be clearly seen that AAF has a cylindrical shape and consists of a parallel set of microfibrils. In addition, there are waxes, oils and other considerable impurities on the surface of the fiber. In general, it is believed that chemical treatments remove non-cellulose materials and impurities from the surface of the fiber.

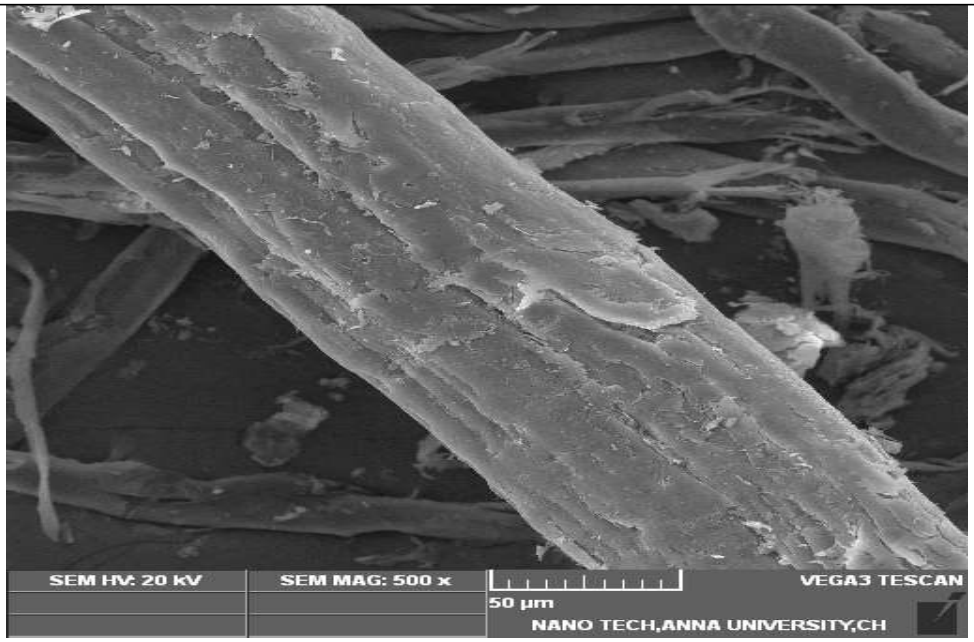
From Figure 4 (b) to (e), it can be seen that several chemical treatments caused an increase in the number of pores in the surface of the fiber. It also showed a very clean surface and the absence of impurities was very obvious. In addition, the treatment also damaged the surface of the fiber; these changes have benefits in the manufacture of composite materials when the fibers are used as reinforcement materials. The rougher surface improves the mechanical interlocking adhesion between the fibers and the matrix. In fact, after treatment with PP, it is observed that waxes, oils and other impurities are completely removed without damaging the surface of the fiber (Figure 4 (e)). It is concluded that the effect of chemical treatments that can significantly eliminate impurities due to the reduction of hydrophilicity.

(a) RAW AAFs

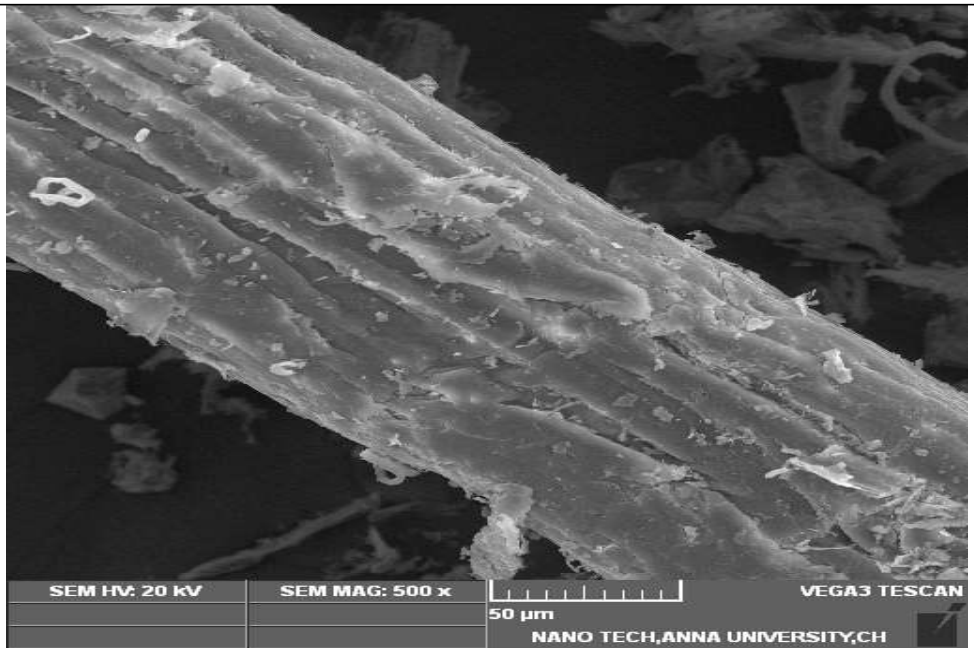

SEM HV: 20 kV
SEM MAG: 500 x
VEGA3 TESCAN
50 µm
NANO TECH,ANNA UNIVERSITY,CH


SEM HV: 20 kV
SEM MAG: 250 x
VEGA3 TESCAN
100 µm
NANO TECH,ANNA UNIVERSITY,CH

(b) NaOH treated AAFs



(c) SA treated AAFs



(d) BP treated AAFs

(e) PP treated AAFs

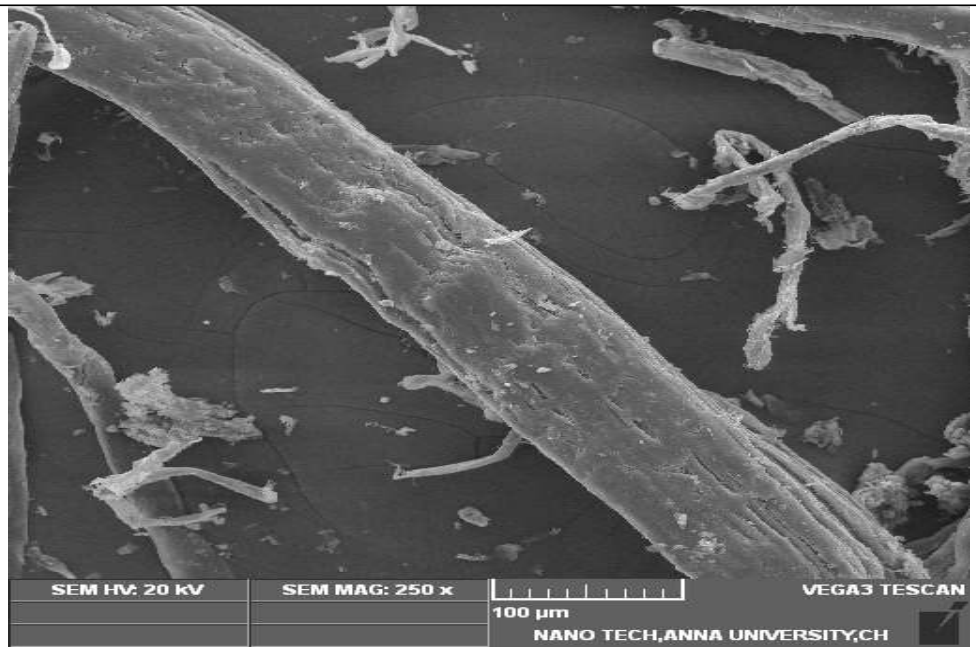


Figure 4. SEM images of Raw and various chemically treated AAFs

4.7. Contact angle measurement

The contact angle of raw and chemically treated AAFs are tabulated in Table 4. There is a marginal increase in the contact angles of the all chemically treated AAFs compared to raw AAF. This behavior is due to the removal of non-polar materials namely hemicellulose, wax, lignin etc. from the surface of treated fibers. And nature of reduction in the contact angle of untreated AAF is due to the presence of hydrophobic constituents. This is evident that the treated fiber surface is rougher and this higher wettability of fiber cause better bonding with matrix material [37].

Table 4. Contact angle of raw and chemically treated AAFs

Fiber	Contact angle (°)
Raw	55.3
NaOH	58.5
SA	59.8
BP	59.4
PP	60.2

Conclusions

The present investigation demonstrates that the application of various chemical treatments on the AAFs fibers generate a paradoxical effect over their morphological, chemical composition, thermal and mechanical properties. The following conclusions was drawn from the study:

- The chemical analysis results indicate that the application of different chemical treatments on the fibers can bring significant reduction in the amorphous contents like

hemicellulose, lignin and other impurities and make them less resistant to water molecules.

- FT-IR analysis confirms that the removal of amorphous contents is produced during the chemical treatments.
- TGA and DSC expose better thermal behaviour in the AAFs after surface modification.
- The tensile test reveals major improved on the tensile strength and elongation at break in all the modified AAFs at the expenses of lower young's modulus which is associated to high variation in fiber dimensions after the treatment.
- SEM measurements provided significant details of hydrophilic nature of raw AAFs inked to various chemical treatments.
- The contact angle analysis shows the enhancement of surface roughness by applying chemically treatment, therefore the treated fibers have a superior wettability.

It is concluded that the chemical modification techniques applied on the surfaces of natural fibers allows to produce superior fibers used to form the novel composite materials for light-weight application.

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