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Investigation of Physico-Mechanical and Moisture Absorption Characteristics of Raw and Alkali Treated New Agave Angustifolia Marginata (AAM) Fiber

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The main objective of this paper is to introduce the new natural fiber for use as reinforcement in polymer matrix composites due to their significant properties like lesser weight, biodegradability, load carrying structures, non-toxic and cost effective. The Agave Angustifolia Marginata (AAM) fiber possesses rich cellulose content which is the cheapest and available sufficiently. An effort has been taken in the extraction of AAM leaf fibers followed by an investigation of its physical, mechanical and moisture absorption properties. The outcome of the fiber before and after alkali treatment was analyzed in terms of diameter, length, aspect ratio and density. The AAM fibers were treated with 2 %, 5 %, 10 % and 15 % NaOH solution and subjected to different holding time as 1, 2, 4, 6 & 8 hrs in each combination and the effect of alkali treatments were explored. Analysis of the tensile strength, aspect ratio, weight loss and water absorption of the fibers are also carried out. It is exciting to note that the 5 % NaOH treatment with 1 hour holding time ascertain substantial improvement in tensile properties of the fibers than the others. A Scanning Electron Microscope (SEM) is used to analyze the morphology study of the varying alkali concentration of the fibers with different immersing time.

Keywords: natural fibers, alkali treatment, physico-mechanical properties, moisture absorption, SEM.

1. INTRODUCTION

Nowadays the natural products are essential to protect the earth and the environment. The natural fibers play major roles in producing high performance renewable and biodegradable “green” composites, which in turn provides a platform for solving the ecological problems and eco-friendly issues. The natural fiber-reinforced composites (FRC) is owning the low weight, more strength, easily biodegradable and cost saving applications of renewable materials. Synthetic fibers are having good properties, but non-biodegradable and hazardous to the atmosphere. As an alternative way, the natural FRCs were developed to overcome the problems in which plant fibers such as hemp, bamboo, kenaf, jute, banana, oil palm, and flax were used.

The family name of AAM is Agavaceae and scientific name of AAM is Agave Angustifolia var. Marginata. The Agave Angustifolia Caribbean Agave is a little wonderful Agave with solid short, sword-shaped leaves with cream coloured edges (Fig. 1 a, c, d and e). Height: 50 cm to 90 cm, spread: 50 cm to 120 cm. The leaves vary extensively long contingent upon developing condition, however, can reach up to 60 cm (or more). They are solid, sword-formed, inward and revolute or plicate, blue or blue-greenish 5 – 10 cm wide at their most stretched level. The Leaves have negligible groups of brilliant white, with intermittent cream stripes. The flowers shading looks like Greenish-yellow to white in terminal panicles and roughly 5 cm long (Fig. 1 b). The bloom stalks reach up to 2.5 m in height (Fig. 1 b). Each leaf ends with the sharp spine as shown (Fig. 1 d, e and f) to provide an easy count of the individual leaves thus associated.

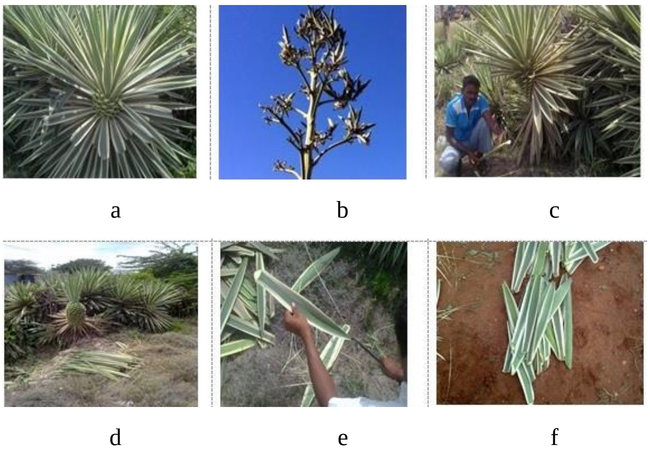

Fig. 1. Collection of leaves from the AAM plant: a – AAM plants; b – flower of AAM plant; c – leaves extracted from AAM plant; d – separated AAM leaves; e – leaves sharp edges were removed; f – the final stage of the leaves (ready to insert into the extracting machine)

Low concentrated NaOH solution (Alkali) and steam explosion technique were employed to remove the surface impurities such as Lignin, wax & Hemicellulose and resulted in improved physical, chemical and mechanical properties of the fiber [1, 2]. This study declared that the increased concentration of the chemical treatment drastically reduced the tensile strength of fibers due to the removal of the fiber gums that act as a matrix [2]. FTIR and XRD analysis supported the steam explosion treatment as it aided in the removal of lignin and hemicellulose gradually from the fiber surface and increased thermal stability [3, 4]. Environmentally friendly methods were also adopted to treat fiber surface instead of chemical treatments [5]. It is noteworthy that the marginal

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concentration of the alkali treatment decreases the surface layers of fiber and higher concentration damages fiber surfaces [1, 6, 7]. The fibers treated in 5 % was observed the adequate tensile strength and improved density factors. The 10 % and 15 % NaOH treated fiber notified more concentration of chemicals, which causes the fiber surface damage and higher tenacity values. The 5 % alkali treated fiber was recommended to fabricate the lightweight composite for different applications [8]. The chemical treatment conveyed the improvement of Cellulose percentages from 31 to 96. It is evident that the low concentration chemical treatment improved cellulose content, adhesion with matrix and mechanical properties [8 – 10]. NaOH-Clay method was used to treat the fiber surface. The result produced maximum tensile strength, reduced water absorption and removed the lignin, hemicellulose and wax content [11]. Relatively less and increased porosity fraction of the SCF used for light weight applications and in thermal and acoustic insulation Purpose [12]. Thermal degradation test post chemical treatment on the wheat husk showed 235 °C and rye husk showed 210 °C respectively, which seems to be an appreciable increment [13]. The NaOCl/NaOH found to be very effective surface treatments in the removal of hemicellulose and providing improved Physico-chemical properties of the fibers [14]. It is noteworthy that the best ratio of Alkali treated fibers increases the optimum strength of the fiber, physical, chemical and mechanical properties. The density value of the agave fiber was found to be lower than other natural fibers [15, 16]. The natural fiber composites had higher water absorption properties than other synthetic fibers [17]. It had been found that 3 % of NaOH treatment ineffective and more than 9 % of NaOH treatment inducing damage to the fiber and 6 % NaOH resulting in enormous variations in physicochemical and mechanical properties [18]. It is also evident and noteworthy that marginal Alkali treated hybrid composite possesses higher tensile, flexural and compressive strength than others [19].

The AAM fibers were utilized as a part of assembling twines and ropes for fishing and rural purposes. Further, these fibers can be utilized for the generation of nets, rugs, floor coverings, door mats, packs, sacks, fish stringers, furniture webbing, wrap, upholstery cushioning, saddle cushions, pad stuffing, brush bristles, wicker bin, wrist trinkets, headbands, shoes, ornamental things, garments and other woven Products. There can be a potential use of AAM fiber in composites and additionally in Nonwovens. Bio-plastics, Geotextiles, floor coverings, fiber sheets, dart sheets and formed furniture can also be made using AAM fibers. Susheel Kalia et al. Investigated on chemically Rehabilitated fibers that can be utilized to plan composites with the green picture, as materials for the textile enterprises as well as promising antimicrobial materials [5]. This paper investigates the extraction process on AAM leaf fibers and its length, diameter, density factors, weight loss in varying concentration of NaOH treatment. The tensile strength and water absorption properties of raw and alkali treated fibers are carried out. Morphological analyses were performed by using Scanning Electron Microscope (SEM). Based on the AAM fiber properties of different concentration of Alkali treatment with the varying soaking time period, the best weight percentage of NaOH treated fibers is revealed out for the natural FRC to the numerous applications.

2. EXPERIMENTAL

2.1. Extraction of AAM fiber

In this experiment, the fiber is separated by a procedure known as decortication as shown in Fig. 2 a. Where leaves are crushed, beaten, and brushed away by a turning wheelset with sharp blades, so that exclusive fibers remain as shown in Fig. 2 b.

The extracted fibers were hanged out in the opened space to allow them to dry in sunlight up to 48 hrs (Fig. 2 c). A new decorticating technology allows for simplification of a processing method used so far, improvement of production economics – through reduction of energy and labor consumption during extraction of fiber [13].

a b c

Fig. 2. Extraction of AAM fiber from the leaves using mechanical decorticator machine: a – leaves fed into the fiber extracting machine; b – extracted fiber was kept in sunlight; c – final stage of fiber

2.2. Chemical method (alkali treatment)

Natural fibers are chemically treated to remove the surface impurities like lignin, pectin, wax content and natural oil. The NaOH solution plays major roles to bleach, clean and make changes in the surface structure of the cellulose in plant fibers. AAM fibers were treated with 2 %, 5 %, 10 % and 15 % (2 g/L, 5 g/L, 10 g/L and 15 g/L) sodium hydroxide solution at room temperature and fibers were immersed in the alkali solution as 1 h, 2 h, 4 h, 6 h and 8 h time duration. Then, washed the NaOH treated fibers with distilled water and tap water repeatedly to neutralize the chemicals. Then the fibers were kept in sunlight for up to 48 h. The more concentration of the NaOH Solution resulted in heavy damage to the fiber surface and less concentration of NaOH only removed the meager amount of surface impurities. The moderate concentration of sodium hydroxide was recommended to achieve the best results [1, 6, 7].

2.3. Physical properties of fiber

2.3.1. Breaking strength and elongation

The breaking strength of the raw and treated fibers was tested as per ASTM D 3822 by using the Instron 5500R machine. In this method, single raw fibers and 2, 5, 10 and 15 % of NaOH treated fibers with different holding time were tested separately. The mean breaking strength, CV % strength, mean elongation and CV % of elongation were calculated as per above standard.

2.3.2. Fiber length

The fiber length (L) was measured by using ordinary steel rule. The leaves vary significantly in length according to growing conditions but can reach up to 60 cm (or more).

2.3.3. Diameter

Image analysis was used to calculate the diameter (d) of the raw and Alkali treated fibers. To get accurate results the image analyzer used in 20 different locations of AAM fibers and finally average mean values were taken.

2.3.4. Density

The ASTM D792-13 (water displacement method) standard followed to find the density of the raw and Alkali treated AAM fibers. The one gram amount of fiber was immersed in the water and volumetric displacement was noted. The weight was measured by the high precision weighing scale machine. Finally, based on the weight to volume ratio the density value was measured [15].

2.4. Weight loss of the fiber

The AAM fibers were subjected to different alkali concentration as 2 %, 5 %, 10 and 15 % with varying holding time as 1, 2, 4, 6 and 8 h. The weight loss of the AAM fibers with effect on varying alkali treatment was explored. The loss in weight was measured by high precision weighing scale machine. A fixed amount of dry cleaned fiber as W_1 and the amount of fiber was weighed again after soaking as W_2. The % weight loss was calculated as:

$$\% \text{ weight loss} = (W_1 - W_2) / W_1 \times 100. \quad (1)$$

2.5. Moisture absorption properties (raw and treated)

Water absorption studies on treated and untreated AAM fibers were carried out in accordance with ASTM D570-10. The AAM fiber has the inclination of absorbing water up to its own particular weight. In this case, the raw AAM fibers and NaOH (Alkali) base treated AAM fibers were subjected to an absorption test by immersing the respective specimens in a refined water bath at room temperature (29 °C). The holding period of 24 hours was provided. The natural fiber retains water because the cell wall polymers have certain hydroxyl and other oxygenated groups that draw moisture amid hydrogen holding. The hemicellulose is for the most part in charge of water absorption in the common natural fiber [17]. The weight of the fibers was measured at regular time intervals and the moisture content was calculated as:

$$\text{Moisture content } (\%) = ((W_2 - W_1) / W_1) \text{ x } 100, \quad (2)$$

where W_1 and W_2 denote the initial weight of dried AAM fiber and weight after time t of soaking respectively.

2.6. Scanning electron microscopy (SEM)

The SEM images were captured and analyzed by JEOL-JSM-6390 model. The SEM generates high-resolution images in a raster fashion with a high electron beam. The SEM analysis was carried on both raw and varying concentrations of selected alkali treated fibers.

3. RESULTS AND DISCUSSION

3.1. Physical properties of AAM fiber

3.1.1. Fiber density

Boopathi et al., investigated the tensile force and tenacity estimations of 5 % NaOH base treated fibers were higher than that of the untreated fibers, 10 % NaOH treated and 15 % NaOH treated fibers [8]. The tensile strength of the 5 % NaOH concentration fibers found to be higher when compared with the raw fiber, 10 % NaOH and 15 % Alkali treated fibers. Moreover, 5 % Alkali base treated fibers revealed higher tenacity quality. The findings revealed that 10 % and 15 % NaOH base treatment resulted in the etching of fiber surfaces, subsequently yielding lower fiber tensile strength. The density values, thus improved by using Alkali treatment and its outcome is shown in Table 1.

Table 1. Diameters, density and tensile strength of raw and alkali treated fibers in various immersion timings

NaOH wt.%	Soaking, h	Average length, mm	Average díameter, mm	Aspect ratio, L/d	Tensile strength, MPa	Density,g/cc
0 wt.%	UT**	500	0.186	2688	512	1.608
2 wt.%	T*-1h	500	0.152	3289	522	1.617
	T-2h	500	0.147	3401	530	1.619
	T- 4h	500	0.145	3448	545	1.626
	T-6h	500	0.143	3497	563	1.630
	T-8h	500	0.141	3546	590	1.639
5 wt.%	T-1h	500	0.134	3731	626	1.641
	T-2h	500	0.132	3788	624	1.645
	T-4h	500	0.130	3846	621	1.647
	T-6h	499	0.127	3929	619	1.649
	T-8h	498	0.125	3984	618	1.655
10 wt.%	T-1h	498	0.123	4049	587	1.660
	T-2h	497	0.121	4107	568	1.663
	T-4h	497	0.120	4142	563	1.665
	T-6h	496	0.118	4203	560	1.669
	T-8h	495	0.115	4304	559	1.702
15 wt.%	T-1h	497	0.114	4360	540	1.705
	T-2h	495	0.112	4420	534	1.709
	T-4h	494	0.110	4491	529	1.712
	T-6h	493	0.109	4523	516	1.714
	T-8h	493	0.107	4607	509	1.717
*T-Treated ** UT-Untreated						

Density values were found to be lesser due to the effect of NaOH treatment. The NaOH treatment leads to densification of fiber cell walls as an outcome of elimination of surface layers (less dense fats and waxes) and filling of pores with grafted molecules. Eventually, we could suggest that this property could be recommended for the AAM fibers that can be used as reinforcements in making polymer composite structures in light weight. Additionally, the supplementary feature of the AAM fiber is biodegradability.

3.1.2. Aspect ratio (L/d)

The uniformity of the natural fiber is hard to maintain the growth of the fiber depends on the region and climatic conditions. Hence, the measurand of the fibers was taken on an average basis to maintain the regularity. Different samples of fibers were taken and each measurand is noted in different positions on the same samples to arrive at the average values as shown in Table 1. The average values were computed for length and diameter. This procedure is followed on both raw fibers and all the samples of alkali treated fibers. From the result, it was shown clearly when the Alkali concentration goes higher, the fiber diameter decreases rapidly and the lengths are also reduced.

3.1.3. Effect of tensile strength of alkali treated AAM fiber

The tensile test was conducted for raw fiber and different concentration of Alkali treated AAM fibers. It was exciting to note that, all the fibers were moved to 0 – 8 h of soaking period, after which moisture content and tensile properties were carried out. Compared to raw fibers, the 5 % of Alkali treated AAM fibers with one hour holding time proved to possess excellent improvements in tensile strength than 2 %, 10 % and 15 % of NaOH treated fibers with different soaking time as shown in Fig. 3. The tensile strength increased as a result of improved cellulose crystallinity. Furthermore, when subjected to more wt.% of NaOH and holding time one could figure out the adverse effect on the cell walls of the fiber surface, reduced cellulose crystallinity and pores developed in the surface area. The above said adverse effects causes a reduction in tensile strength of the fiber. It was proved that the tensile strength of the 5 % alkali treated fibers found to be higher than others [8].

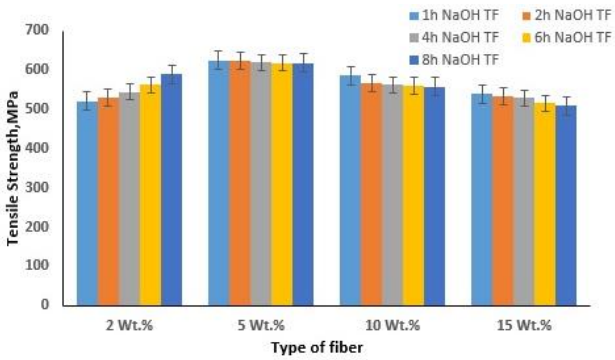

Fig. 3. Tensile strength vs fiber type. The tensile strength was increased in 5 % NaOH with 1 hour immersion treated fibers than others, tensile strength was going down due to more concentrations of NaOH

3.1.4. Effect of weight loss of alkali treated AAM fiber

The continuous increment in weight loss of AAM fiber could be noticed with expanding concentrations of NaOH and increase in treatment time. The reason for the weight reduction was likely because of the slow evacuation of hemicellulose and lignin. The Alkali treatment breaks hydrogen bonds between the hydroxyl bunches (–OH) of the cellulose, hemicellulose and lignin and prompts defibrillation, the breakdown of the fiber group into smaller fibers. In this chemical treatment, the 15 % and 8 hour holding time alkali treated fibers observed heavy weight loss than the other concentration of NaOH treated fibers as shown in Fig. 4. The weight loss, increased gradually due to the high concentration of NaOH % and more treatment time. The Brígida reported the treatment with NaOH was the most proficient in hemicellulose evacuation and in cellulose exposition.

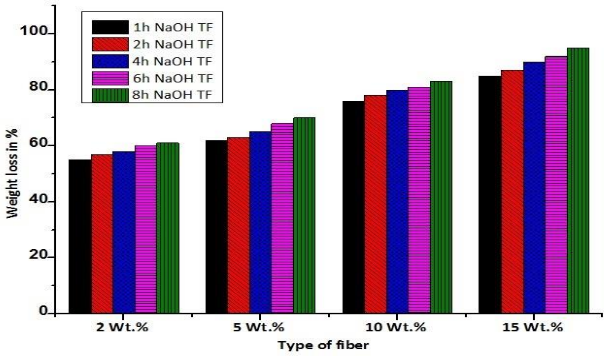

Fig. 4. Weight loss vs fiber type

A high measure of cellulose on fiber surfaces gives free hydroxyl bunches that can respond with the epoxide bunches and weight loss, increased due to strong surface damage because of the high concentration of alkali treatment [14].

3.1.5. The effect of moisture absorption behavior of various % of alkali treated AAM fiber

The water absorption range was lesser in 5 % with 1 h immersing period of alkali treated fibers than others (Fig. 5). Moreover, it was observed that the water absorption rate increased in the case of 10 – 15 % alkali treated fibers which conclude that increase in NaOH content more than 5 % rapidly increases the water absorption range. It can be found a wide range of natural fibers, water uptake was much higher than the synthetic fibers with expanding soaking time period [13].

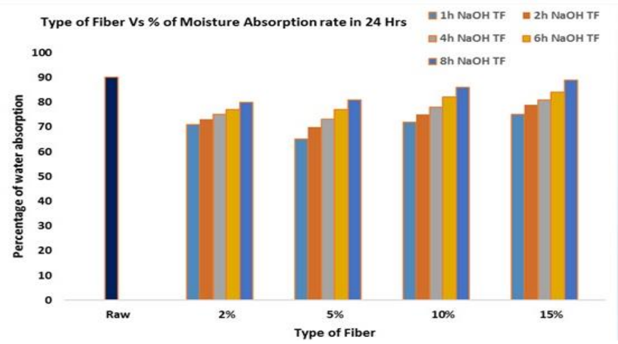

Fig. 5. Type of fibers vs % of water absorption rate in 24 h, percentage of water absorption, increased due to more concentration of NaOH with more treatment time

3.2. Surface morphology of the AAM fiber

The main objective of this study was to understand the difference between the raw and varying concentration of alkali treated fibers. Fig. 6 a shows the raw AAM fibers containing surface impurities such as white membrane layers, wax and hemicellulose. Fig. 6 b shows the 2 % NaOH and 8 h soaking period of AAM fibers and revealed the presence of impurities on the fiber surfaces due to inadequate NaOH concentration. Fig. 6 c shows the 5 % NaOH with 1hour soaking period of the Alkali treated fiber and with better removal of surface impurities when compared with the raw and 2 % NaOH treated fibers. Fig. 6 d and e) shows the 10 % with 1 hour soaking time and 15 % with 8 hours soaking time of NaOH treated fibers.

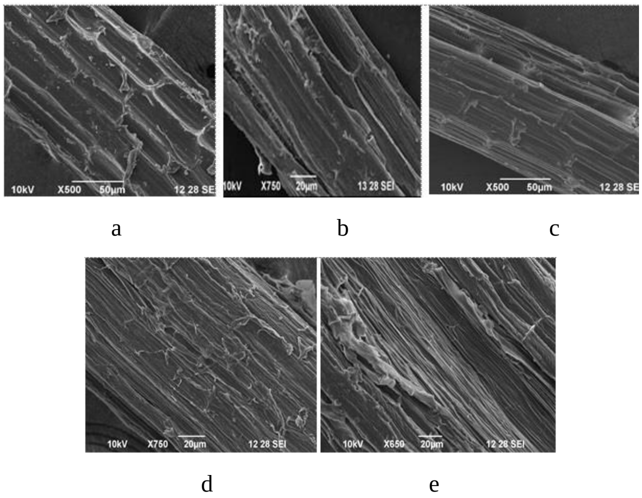

Fig. 6. SEM images of alkali treated AAM fibers: a – raw fiber contains more surface impurities (wax, lignin and hemicellulose); b – the 2 % NaOH and 8 h holding time period treated fibers with the removal of less quantity of surface impurities; c – 5 % NaOH with one hour soaking time period of treated fibers with the total elimination of the surface impurities; d and e – 10 % and 15 % 8 hours NaOH treated fiber reflecting adverse effects: fibers were crushed and cell wall damaged

It was evident that the cell walls were shrunk and small pores were apparently seen and after the NaOH treatment, the fibers were crushed and damaged. If the pore was increased, the mechanical properties were reduced, and the moisture absorption rate and weight loss of the AAM fibers were increased. The best concentration of NaOH treatment was taken as the one which provided reduced surface impurities of the fibers and improved version of physical-mechanical and moisture properties of the fibers.

4. CONCLUSIONS

The physical, mechanical and moisture absorption characteristics of AAM fibers were examined and the results attained are:

1. The fiber diameter, length, and weight ratios were analyzed in the experiments. The diameter and weight of the fibers were reduced due to high concentrations of alkali treatment and a high soaking time period when exceeded more than 5 % NaOH and more than 1 hour soaking time period.
2. The density of the AAM fiber was lesser, but the density was improved in the 5 % alkali treated fiber than the raw fiber due to increased in cellulose crystallanity of the fiber surface. The applications of this fiber were used to make fewer lightweight composites.
3. The NaOH- base treated fibers detached the surface layers of the fibers like lignin, hemicellulose, and other surface impurities. And it is used to make a fiber surface in a smooth manner and bonding between fiber surface matrixes in better effective manner.
4. The tensile strength was found to be greater in 5 % NaOH with 1hour immersing period of treated fibers than others due to the better concentration of Alkali and improved cellulose crystallinity. It was also evident that the fiber got strengthened after the treatment compared to raw fiber. The weight loss of the fibers was increased due to more concentrations of NaOH with respect to high treatment time.
5. The water absorption rate was less in 5 % with the 1hour holding of alkali treated fibers than others. Moreover, it was observed that water absorption, increased in the case of 10 – 15 % alkali treated fibers which show that increase in NaOH content above 5 %, gradually increases the water absorption.
6. The SEM study revealed a clear picture of removal of surface impurities in the 5 % NaOH with 1 hour soaking period than raw fiber and other wt.% of NaOH treated fibers.
7. Finally, four types of alkali treatment were conducted with different immersion timings. Specifically, the AAM fiber and 5 % NaOH with 1 hour treatment gave better mechanical properties, less moisture absorption and removed surface impurities. Hence, 5 % NaOH treated fiber could be recommended for the reinforcement process of composite manufacturing and its applications.

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