Journal of Polymer Science and Technology 6(1) 2021:1-10
ISSN: 2550-1917

INFLUENCE OF ALKALI TREATMENT ON MECHANICAL AND MORPHOLOGICAL PROPERTIES OF SINGLE *AGAVE ANGUSTIFOLIA* FIBRE

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ABSTRACT

The effect of alkali treatment on physical, tensile, and morphological properties of single *Agave Angustifolia* fibre was investigated. The fibre was extracted from *Agave Angustifolia* leaves using a fibre extracting machine. Alkali treatment using NaOH by reflux method for 2 hours was performed on the single fibre of *Agave Angustifolia*. The treated and untreated single *Agave Angustifolia* fibre's mechanical properties were tested using tensile tests. The alkali-treated single fibres show higher tensile strength, Young's modulus, and elongation at break than that of the untreated fibres. The crystallinity index and crystallite size increased after alkali treatment from X-ray diffraction analysis. Scanning electron and optical microscopy micrographs showed that *Agave Angustifolia* fibres' diameter decreased with alkali treatment. Besides, micrographs also reveal the reduction of the fibre's surface roughness with alkali treatment. Comparing the treated and untreated *Agave Angustifolia* fibres' mechanical properties with other well-known fibres show that this fibre has excellent potential to reinforce materials in various types of polymer matrices.
Keywords: Physical properties, SEM, Single fibre test; Tensile test, X-ray diffraction

INTRODUCTION

Growing awareness of the environmental problems has led researchers to focus on studies based on biomaterials. Thus, the study's focus contributes to increased research based on new biocomposite production. Biocomposites are made up of polymers and fibres from natural sources. Natural fibres can be obtained from various plant parts, such as leaves, bark, stems, seeds, and fruits. Compared with synthetic fibres, natural fibres are biodegradable, lighter, low density, renewable, relatively good mechanical properties, readily available at low cost. Natural fibres' excellent nature has encouraged its use in various applications such as construction, automobiles, electronics, sports equipment, and packaging. However, natural fibre production is challenging to meet today's growing industrial demand [1]. Therefore, many new natural fibres have been introduced as a potential reinforcing material, such as *Chrysanthemum morifolium* [2], *Grewia damine* [3], *Centaurea solstitialis* [4], Kigelia africana fruit [5], *Derris scandens* stem [6], *Acacia nilotica L.* [7] and many more.

One of the less exploited natural fibres is *Agave Angustifolia*. *Agave Angustifolia* fibre consists of 67% α-cellulose, 25% hemicellulose, 6% lignin, and 2% extractives [8]. Cellulose from

Agave Angustifolia is effective as a reinforcing agent where the mechanical, thermal, and degradation properties of biocomposites increase with its addition [9-10]. However, its use as a reinforcing agent still does not get researchers' attention, and no studies are reporting on its mechanical properties. The study of fibres' mechanical properties is essential to know their potential applications. Natural fibres' mechanical properties depend on the chemical composition, age, location of the plant, density, cell dimensions, extraction process, and the fibre's internal structure [11-12].

Although natural fibres exhibit some excellent properties compared to synthetic fibres, they also exhibit some drawbacks. Among the main drawbacks that limit its use as a reinforcing agent is its hydrophilic nature. As opposed to polymer matrices' hydrophobic properties, this results in a weak interface between natural fibres and polymer matrices. Therefore, various surface treatment methods such as alkali, silane, peroxide, and acetylation treatments have been applied to natural fibres [13]. Among all these treatments, alkali treatment using NaOH is the most widely used treatment because it has been proven to improve the fibres' mechanical and thermal properties [14-15]. Furthermore, this treatment is simple with low cost. The alkali treatment acts by removing hemicellulose and lignin from natural fibres [16-17]. The reactions and results of alkali treatment are as follows:

$$\text{Fiber}-\text{OH} + \text{NaOH} \longrightarrow \text{Fiber}-\text{O}-\text{Na}^{+} + H_2O + \text{Hemicellulose} + \text{Extractives}$$

In this study, natural fibres were extracted from the *Agave Angustifolia* plant's leaves, subsequently undergoing alkali treatment using NaOH. The mechanical, physical, and morphological properties of alkali-treated *Agave Angustifolia* fibres were compared to untreated fibres.

EXPERIMENTAL

Materials

The leaves of the *Agave Angustifolia* plant were harvested from Sungai Ramal Dalam, Kajang, Selangor, Malaysia. Sodium hydroxide (NaOH) was supplied from Systerm, Malaysia.

Preparation of *Agave Angustifolia* Fibre

The thorns on the Agave leaves' edges were removed. Then, the leaves were dried under the sun for a week to reduce the leaves' moisture. *Agave Angustifolia* fibres were extracted from the leaves using a fibre extraction machine. Figure 1(a) shows the Agave plant, and Figure 1(b) shows freshly extracted Agave fibre using a fibre extraction machine. The extracted fibres were then retted with water for a week to dissolve most cellular tissue and pectin around the fibre-bundles. Water was changed two times a day. The fibres were then dried at an outdoor ambient temperature and were combed after dried.

Fig. 1 (a) Plant and (b) freshly extracted fibre of *Agave Angustifolia*

Akali treatment

The long Agave fibres were treated with an aqueous 4% NaOH solution. The reflux process was carried out for 2 hrs at a temperature of 80 to 90 ºC. After 2 hrs, the mixture was left to cool and then filtered. The filtered Agave fibres were washed with distilled water several times to remove the alkali solution.

Characterisation of Raw and Alkali-Treated Agave Fibre

Density measurement

The treated and untreated Agave fibres' density was determined through the liquid displacement method based on the Archimedes principle. Both fibres were weighed first and then immersed in distilled water of known initial volume. Throughout the experiment, special care was taken to prevent air bubbles in the immersion medium. The final volume was recorded, and the density was calculated by taking an average of three specimens for each Agave fibre sample.

X-ray diffraction analysis

XRD analysis was performed to study alkali treatment's effect on Agave fibre's crystallinity. This analysis was performed using a Bruker AXS diffractometer model D8 Advance. Before the analysis was carried out, all the long fibres were ground into a fine powder and then flattened on the sample holder to obtain a uniform X-ray exposure. The source of Cu-Kα radiation ($\lambda = 0.1541$ nm) was shot on the sample at 2θ range from 5° to 50° generated at 40 kV and 30 mA. The crystallinity index (CrI) for alkali-treated and untreated Agave fibres was calculated according to Segal expression as in equation (1) [18].

$$\mathrm{CrI} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

I_{am} and I_{002} in expressions represent the minimum intensity at 18° to 19° and the maximum intensity at 22° to 23°, respectively. The crystallite size was calculated according to equation (2) [19].

$$\text{Crystallite size } = \frac{K\lambda}{\beta \cos\theta} \tag{2}$$

Where K = 0.84 (Scherrer constant), λ = 0.154 (X-ray wavelength), θ refers to the Braggs' angle, and β refers to the entire width of the peak at 22° to 23°.

Morphological properties

The fibres' surface changes before and after alkali treatment were observed through optical microscopy (OM) and scanning electron microscopy (SEM). The OM model DinoCapture 2.0 was used to measure a single fibre's diameter before performing tensile testing. Morphological changes on the fibre surface were recorded with the SEM microscope model Leo 1450VP. All samples were placed on the aluminium stub plane's surface and dried in an oven at 60 °C for 30 min. All fibres were coated with 0.01 to 0.1 µm gold using a splash coating model SC 500 to increase the specimen's ability to conduct electricity. All the samples were recorded at a voltage of 3 to 15 kV.

Single fibre test

The tensile properties of treated and untreated Agave fibres were assessed through a single fibre test using an Instron machine model 5566. For each sample, Agave fibres were glued on a cardboard with a gauge length of 25 mm. A total of twenty specimens for each sample were pulled at a crosshead speed of 3 mm/min, according to ASTM D3822/D3822M-14(2020).

RESULTS AND DISCUSSION

Physical and Structure Properties

Table 1 shows the density of raw and alkali-treated *Agave Angustifolia* fibre. The density of raw *Agave Angustifolia* fibre is 1.15 g/cm^3. This density value is almost the same as Agave Americana fibre density, which is 1.159 g/cm^3 [20]. The low density of fibre is beneficial in manufacturing a light-weight material. The density of *Agave Angustifolia* is low when compared to cotton, flax [21], and *Agave Sisalana* [22] but higher when compared to kenaf and rice husk [23]. Alkali treatment increased *Agave Angustifolia* fibre density, where the density is 1.56 g/cm^3. Similar trends were recorded by Pitchayya Pillai et al. [24], Ganapathy et al. [25], and Kathirselvam et al. [26]. The increase of density is due to the removal of hemicellulose and increased α-cellulose content in alkali-treated Agave fibre [8]. The α-cellulose has a higher weight density than other lignocellulosic components [1], thus increasing the density of alkali-treated fibre.

Table 1 Physical and structural properties of raw and alkali-treated *Agave Angustifolia* fibre

Agave Fiber	Density (g/cm^3)	Crystallinity Index (%)	Crystallite size (nm)
Raw	1.15 ± 0.02	59	1.02
Alkali treated	1.56 ± 0.03	66	1.40

In addition to the removal of hemicellulose, the increase of alkali-treated fibres' density is also due to the increased crystallinity index (CrI), as seen in Table 1. The CrI of *Agave Angustifolia* fibre is 59%, which is almost the same as cotton (60%) [27], higher than *Agave Americana* fibre (13.36%) [20] but lower than *Agave Sisalana* (70.9%) [28]. The CrI indicates that *Agave Angustifolia* fibre's crystal structure is more ordered than the *Agave Americana* fibre. The increase of %CrI of *Agave Angustifolia* fibres after alkali treatment is due to a better close-packed and stress relaxation on the cellulose chain due to the removal of hemicellulose [29-30]. Moreover, this increase is also due to the rearrangement of crystalline areas [31]. Meanwhile, *Agave Angustifolia* fibres' crystallite size was also found to increase with alkali treatment. The crystallite size of raw *Agave Angustifolia* fibre is 1.02 nm, which is higher than sago frond (0.53 nm) [32] and lower than cotton (5.5 nm) [27]. The increase in fibre crystallite size with alkali treatment is similar to the study reported by Ganapathy et al. [25].

Morphological Analysis

The changes in the morphology of the surface of raw and alkali-treated *Agave Angustifolia* fibres are shown in Fig. 2 and Fig. 3. Both micrographs in Fig. 2 and Fig. 3 clearly show that the alkali treatment leads to the changes in the *Agave Angustifolia* fibres' morphology. The size of raw and alkali-treated *Agave Angustifolia* fibres is measured through the optical micrograph, as shown in Fig. 2. The *Agave Angustifolia* fibre diameter is between 60 to 250 µm, while the size of alkali-treated *Agave Angustifolia* fibres is between 5 to 120 µm.

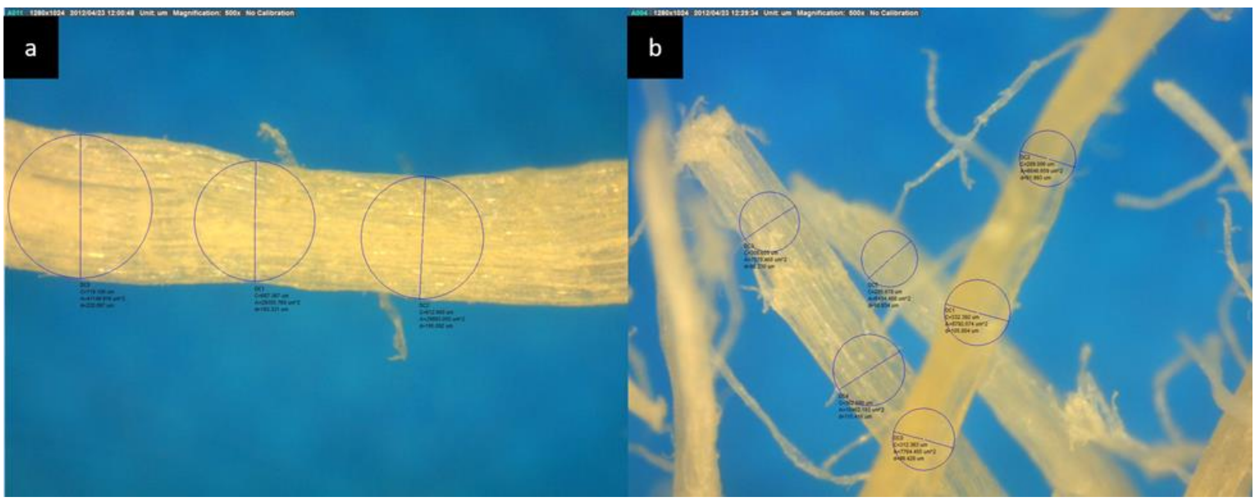

Fig. 2 Optical micrographs of (a) raw and (b) alkali-treated *Agave Angustifolia*

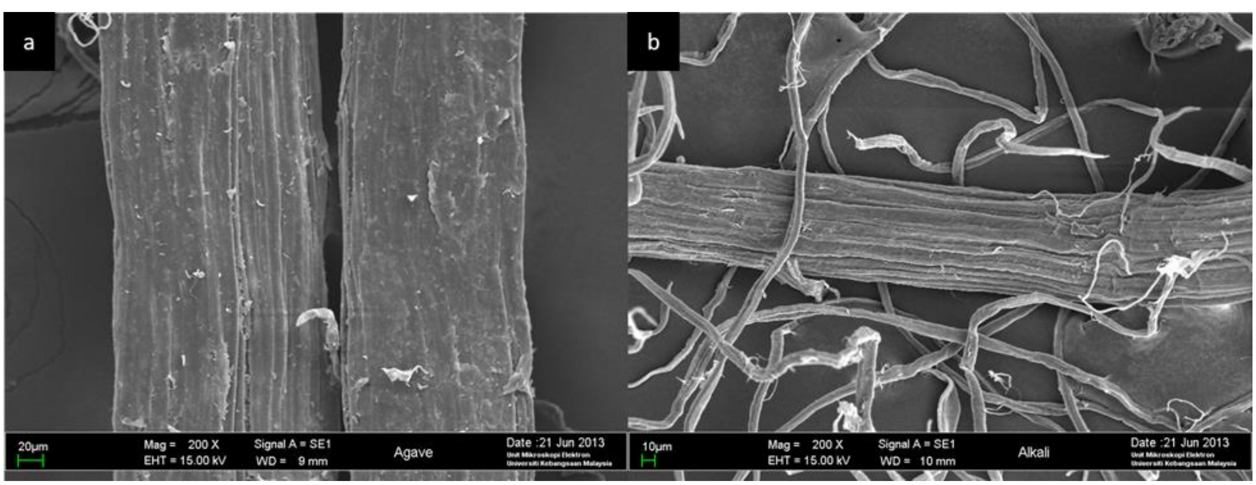

Fig. 3 FESEM micrographs of (a) raw and (b) alkali-treated *Agave Angustifolia*

The SEM micrograph of raw *Agave Angustifolia* fibres in Fig. 3(a) shows a bundle of fibres consisting of long individual fibre cells bound together by hemicellulose and lignin. The raw *Agave Angustifolia* fibre surface is uneven and attached with hemicellulose, lignin, pectin, and wax with other impurities. The micrograph in Fig 3(b) of alkali-treated *Agave Angustifolia* fibres shows that some fibres are separated individually, and some are still bound in the form of fibrous bundles. The separation is due to the removal of the fibrous cell-binding component in which more than 84% hemicellulose is removed during alkali treatment [8]. During alkali treatment, hemicellulose is hydrolysed and subsequently soluble in water [33]. This aids in defibrillation and the fibre bundle's opening, as shown in the micrograph.

Tensile properties

The tensile properties of raw and alkali-treated *Agave Angustifolia* fibres are shown in Fig. 4, and the details are recorded in Table 2. The tensile strength, tensile modulus, and the elongation at break of Agave Sisalana fibre are lower than *Agave Angustifolia* fibre, which is at 303 MPa, 3.6 GP, and 2.8%, respectively [34]. However, fibres' mechanical properties depend on the microfibrils' age and orientation. A comparison of *Agave Angustifolia* fibres' tensile properties with other fibres is shown in Table 2. The obtained results clearly show that the alkali-treated *Agave Angustifolia* fibres exhibit better tensile properties than raw fibre. The increase is due to the removal of hemicellulose and other extractive substances, increasing defibrillation. The removal of hemicellulose caused new hydrogen bonds to form between cellulose fibrils, thus increasing mechanical strength. Besides, high % CrI also contributes to improving these mechanical properties.

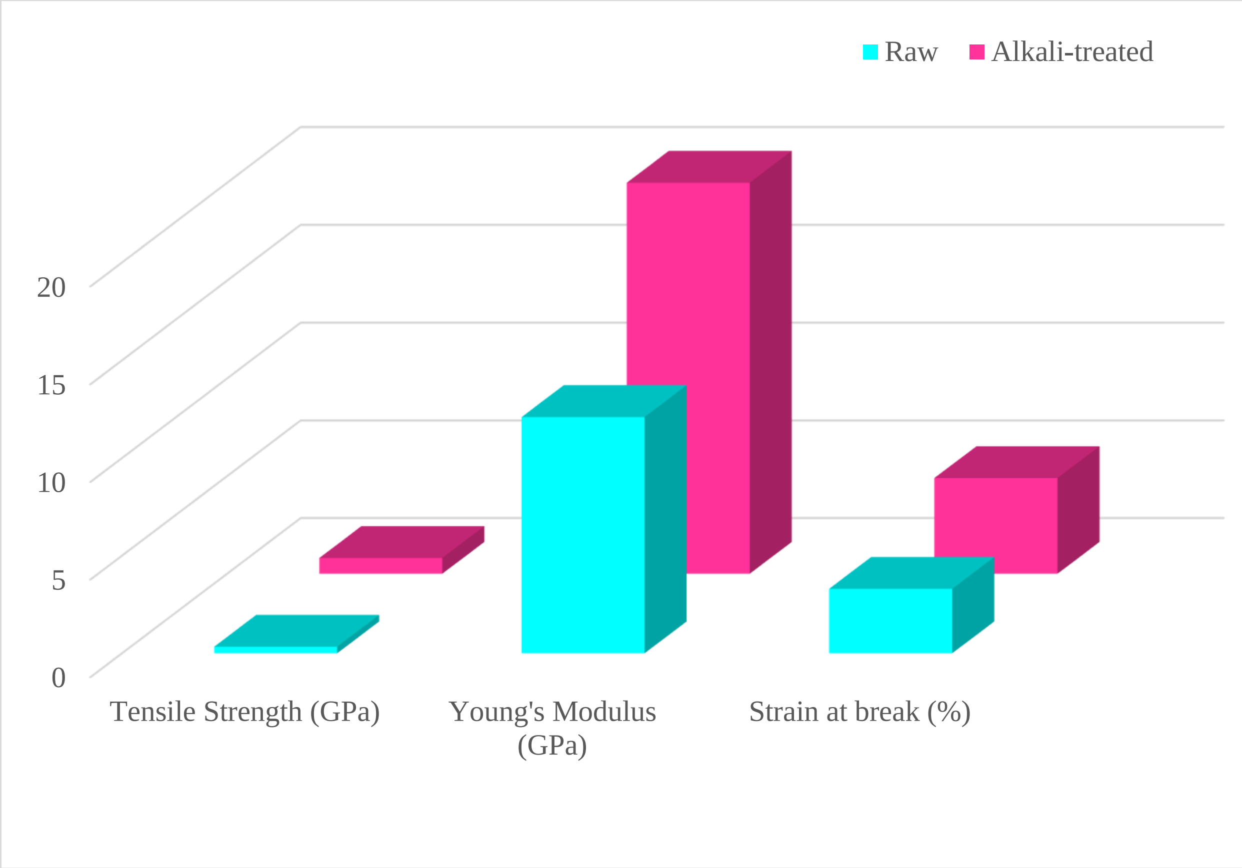

Fig. 4 Tensile properties raw and alkali-treated *Agave Angustifolia* fibres.

Table 2: Comparison of *Agave Angustifolia* fibre's mechanical properties with other natural fibres.

Natural fibre	Chemical treatment	Mechanical properties			Ref
		Tensile strength (MPa)	Young's modulus (GPa)	Strain at break (%)	
Agave Angustifolia	-	338.8 ± 30.6	12.1 ± 3.3	3.3 ± 0.6	Present work
	4% NaOH	806.8 ± 154.9	21.2 ± 4.8	4.9 ± 0.7	
Saharan aloe vera cactus leaves	-	621.8	42.29	2.39	[1]
	5% NaOH	805.5	40.03	2.47	
Date palm fibre	-	233 ± 27.1	7.15 ± 2.0	10.3 ± 1.57	[35]
	6% NaOH	366 ± 36.4	5.45 ± 1.4	12.8 ± 2.4	
Coir fibre	-	150	2.2	15	[36]
	3% NaOH	126	0.9	14.5	
Abaca fibre	-	717 ± 83	18.6 ± 1.9	4.2 ± 0.2	[37]
	5% NaOH	773 ± 119	25.3 ± 6.3	3.2 ± 0.5	
Sugar palm fiber	-	216.80 ± 31.02	3.86 ± 0.67	23.34 ± 3.91	[38]
	4% NaOH	248.04 ± 44.96	4.33 ± 0.96	19.91 ± 3.49	
Red banana peduncle fibre	-	440 ± 13.4	12.41	1.57	[24]
	5% NaOH	650.12	13.56	2.9	
	-	32.3	69.61	2.57	[39]

Perotis indica fibre	5% NaOH	34.9	79.45	2.0	
Banyans' tree aerial roots	- 5% NaOH	19.37 ± 7.72 20.45±12.20	0.70 ± 0.40 0.82 ± 0.32	1.8 ± 0.40 1.6 ± 0.50	[25]
Thespesia populnea barks	- 5% NaOH	557.82 ± 56.29 661.96 ± 33.96	20.57 ± 4.46 22.46 ± 4.09	2.80 ± 0.56 3.08 ± 0.53	[26]

CONCLUSION

In this article, the suitability of raw and alkali-treated *Agave Angustifolia* fibres as a reinforcing material has been verified through physical, mechanical and morphological characterisation. The following conclusion is the outcome of all the analysis that has been conducted:

- The density of *Agave Angustifolia* fibre increases with alkali treatment.
- Alkali treatment increases the crystallinity percentage and crystallite size of *Agave Angustifolia* fibre.
- Fibre bundles undergo a defibrillation process with alkali treatment.
- The rough surface of *Agave Angustifolia* fibres becomes smooth after alkali treatment.
- Alkali treatment increases tensile strength, Young's modulus, and elongation at break.
- Increased crystallinity percentage improves the mechanical properties of fibres.

Based on these results, alkali-treated *Agave Angustifolia* fibre can be a good alternative for polymer composite reinforcing material.

ACKNOWLEDGMENT

The authors acknowledge financial support from the Ministry of Higher Education, Malaysia (MOHE) and Universiti Kebangsaan Malaysia (UKM) under research grants DIP-2018-037 and GUP-2020-048.

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