

# Effect of alkali treatment on the properties of *Agave augustifolia* v. *marginata* fibre

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**Abstract** - The novel lignocellulosic fibre *Agave augustifolia* (AA) leaf fibres hitherto unexplored in terms of its chemical composition & other properties has been studied. AA fibres were treated with NaOH solution with varied concentrations (2 – 20%) at room temperature for 1h. Chemical composition and physical properties were evaluated after alkali treatment. The hemicellulose content of the alkali treated fibres decreased as the alkali concentration increased. Fibres were characterized by scanning electron microscopy (SEM), XRD analysis, Thermal degradation (TGA). Tensile strength and moisture content were also analysed. The alkali treated fibres between 2% - 15% showed all round improvement in the properties as compared to those of untreated raw fibres.

**Key Words:** NaOH, Chemical composition, Hemicellulose, SEM, TGA, XRD.

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## 1. INTRODUCTION

There are many plant fibres that are found in abundance in nature are mainly lignocellulosic in nature comprising of cellulose, hemicellulose, and lignin. Apart from these other components like pectins, wax, mineral matters etc. are regarded as surface impurities. Being nature cellulose based fibres, they are gaining increasing attention for their possible application in diversified fields such as building materials and automobile sector, light weight material composites etc. Low cost, biodegradability, low density, CO<sub>2</sub> neutral life cycle and ease of processing and recycling are among the known advantages of plant fibres of such environmentally friendly fibres. Natural fibres are more recently being used in combination with polymers in composites. Natural fibre-reinforced composites can be used in the plastics, automobile and packaging industries to reduce material cost. Many types of natural fibres have been investigated and used in reinforced composites including Flax, hemp, jute, straw, wood fibre, rice husks etc. [1]. Agro based lignocellulosic by products such as rice husks, wheat straw have also been used as raw material for production of bioethanol, chemicals, enzymes, proteins, pharmaceuticals and fibre reinforced polymer composites [2]. Plant fibres are highly polar due to the presence of the hydroxyl groups, which are readily available for the formation of hydrogen bonds with interacting, resin matrices. Fibres are covered with pectin and waxy substances, thus hindering the hydroxyl groups from reacting with polar matrices, and forming mechanical interlocking adhesion with nonpolar matrices. To produce the reactive hydroxyl groups and the rough surface for adhesion with polymeric materials, plant fibres need to undergo physical and/or chemical treatment, to

modify the surface and structure to improve their compatibility with the polymer matrix. Various chemical treatments of natural fibre are carried out such as acetylation, silanization, formaldehyde treatment, isocyanate treatment, acrylation, permanganate treatment and mercerization of fibres. Among these treatments, mercerization or alkali treatment is a versatile and simplest treatment for modification of these fibres. It brings about changes in dimension, fine structure, and morphology of natural fibres [3-8]. Alkali treatment reduced the content of amorphous materials such as hemicellulose, which results in increase of their surface roughness and compatibility to non-polar polymer matrix which in turn improves its mechanical properties [9] & [10].

In the present work a novel fibre is extracted from the plant *Agave angustifolia* v. *marginata* commonly called the "Banded Caribbean Agave". *Agave angustifolia* belongs to the Agavaceae family. This is a very rugged, attractive, eye-catching plant with symmetrical narrow, stiff bayonet leaves liberally margined in creamy white. The rosettes can be 1 m in diameter with several leaves 50-80 cm long ending in 18 mm long terminal spine (Fig. 1). It is grown as an ornamental plant worldwide. Native from Costa Rica to Mexico and North America, the height of the plant can reach upto 3-4 feet. Each rosette develops a trunk 40 cm high and produces offsets around the base, eventually forming clumps. A single plant has 20 to 30 offsets spreading to 15 feet away from the parent plant. Fairly tropical, it grows quickly, but will not tolerate much frost. It grows best in full sun but can adapt to some shade. Caribbean Agave is a hardy survivor, tolerating heat,

drought, and salty seaside conditions [11]. Hence an attempt has been made to investigate the properties of hitherto unexplored this novel AA fibre see its potential to be used as textile as well as composite material. The effect of alkalization on the properties of AA fibres is also studied.



Fig. 1. Agave angustifolia v. marginata plant

## 2. MATERIALS

The fibres extracted from the leaves of the AA plant and harvested from the rural areas of Maharashtra, India, were used without any pretreatment or purification. The chemical reagents such as sodium chlorite, sodium bisulphate, glacial acetic acid, sodium hydroxide, ethanol and benzene of analytical grade were procured from SD Fine Chemicals Ltd., India.

## 3. EXPERIMENTAL WORK

### 3.1. Fibre extraction

The fibres were extracted from the matured leaves of the AA plant. These matured leaves were separated from the thorns on the edges and tip of the leaf, with sharp knife. The leaves were then subjected for water retting for 15 days. There after the leaves were beaten with a wooden hammer to loosen the fibres. These fibres were washed continuously under running tap water to get clean fibres. This was followed by sun drying, combing and hand brushing to get uniform fibres suitable for physical, chemical and morphological analysis.

### 3.2. Alkali treatment of fibres

The AA fibres were chopped to a length around 10cm and treated with different concentrations (2%, 5%, 10%, 15%, 20% w/v) of aqueous solutions of NaOH at room temperature for 1hr using material to liquor ratio 1:30. The fibres were then removed and washed thoroughly with water, neutralized with dilute acetic acid and washed again with fresh water. The fibres were there after dried at 65 °C in hot air oven for 24 h.

### 3.3. Determination of chemical composition

The chemical analysis of the fibres was carried out as per the standard procedure used by Chattopadhyay & Sarkar [12]. The preweighed chopped fibres were used for analysis and lignin,  $\alpha$ -cellulose and hemicellulose content were determined using this method. The ash content was

determined by standard method of TAPPI T 211 om-02 (2002).

### 3.4. Morphological studies

The scanning electron micrographs of the fibre surface were recorded on a microscope (JEOL JSM 6380LA, Japan) from Institute of Chemical Technology. The fibre samples were sputter coated with platinum before recording the micrographs.

### 3.5. FTIR analysis

The IR spectra of raw AA fibre sample was recorded using FTIR spectrophotometer (Shimadzu 8400s, Japan) using ATR sampling technique by recording 45 scans in % transmittance mode in the range of 4000-600  $\text{cm}^{-1}$ .

### 3.6. XRD analysis

The crystallinity of raw AA fibres was studied using an X-ray diffractometer (Shimadzu 6100, Japan) equipped with  $\text{CuK}\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ) in the  $2\theta$  range 5-30°. The experiments were performed in the reflection mode at a scan speed of 2°/min in steps of 0.02°. The crystallinity index (CrI) of the fibre was calculated according to the empirical method shown in the following equation [13].

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

where  $I_{002}$  and  $I_{am}$  are the peak intensities of crystalline and amorphous materials, respectively.

### 3.7. Thermal analysis

The samples of untreated and alkali treated AA fibres were cut into small pieces and thermogravimetric analysis (TGA) was carried out. The thermograms were recorded on Shimadzu 60H DTG machine using aluminium pan between temperature range 30-500 °C under the inert atmosphere of nitrogen at a flow rate of 50ml/min.

### 3.8. Tensile properties and Moisture regain

The tensile properties of the fibres were measured according to ISO 5079; 1996 method in terms of the breaking load, percentage elongation at breaking using Tinius Olsen tensile testing machine. A gauge length of 20 mm with a speed of 5 mm/min were used for the testing. Approximately 15 fibres were tested for their tensile properties. The fibre sample was tested according to ASTM standard method 2495, for estimation of moisture content.

## 3. RESULTS AND DISCUSSIONS

### 3.1. Determination of chemical composition

The results of analysis of chemical composition of untreated and alkali treated AA fibres are displayed in (Table. 1). The results indicate that the alkali treatment caused distinct change in composition of the fibre with respect to  $\alpha$ -cellulose, hemicellulose and lignin content. The untreated and alkali treated fibres were first dewaxed by extracting the chopped fibres in benzene/ethanol solvents (2:1 v/v). The percentage of extractable matter decreased as the alkali concentration increased from 2% to 20%. This might be because the alkali treatment removed most of the wax from the fibres. The hemicellulose content also distinctly decreased with increase in the concentrations of the sodium hydroxide solution which may be attributed to its relatively much more sensitive nature to the action of aqueous sodium hydroxide at room temperature than lignin or  $\alpha$ -cellulose [14]. The hemicellulose content of the AA fibres was found to be reduced from 25% in raw fibre to 10% for AA fibres treated with 20% NaOH solution. It is well known that hemicellulose dissolves in alkali and hence, such alkali treated AA fibre showed consequent increase in relative percentage of  $\alpha$ -cellulose and lignin. Ash content also decreased from 1.5% to 0.5% as most of the impurities such as wax, pectins, fatty substances etc. were removed during the alkali treatment.

**Table -1:** Chemical composition of raw and NaOH treated AA fibres.

NaOH conc %	Extractives in solvent (%)	Lignin %	Hemi-cellulose %	Cellulose %	Ash content %
0*	2.5	6.5	25	64	1.5
2%	2	8	23	66	1.5
5 %	1	11	20	67	1
10%	1	12	18	68	1
15%	0.5	14	15	70	0.5
20%	0.5	15	10	74	0.5

\*Raw, untreated fibre.

### 3.2. SEM analysis

The micrographs of untreated and alkali treated AA fibres (refer Fig. 3) indicate that the surface of raw fibre was irregular and covered with impurities such as hemicelluloses, pectin, and other waxy substances. On alkali treatment the micrographs showed the removal of impurities from the surface layer of the fibres [15]. This is because alkali dissolves the hemicellulose and the removal of the hemicellulose content increased as the concentration of NaOH increased. The surface of the fibre became smoother due to removal of surface impurities (Fig. 3b, 3c) upto 5% NaOH treatment. However, further increase in the NaOH concentrations resulted disrupting the surface making it rough (Fig. 3d, 3e, 3f). Such rough surfaces of course are expected to promote good interfacial bonding between the fibres and matrix if used in the fibre reinforced composites [16] & [8]. The diameter of

the fibres and aspect ratio (L/D) (refer Table. 2) indicate that the diameter of the AA fibre decreased as the alkali concentration increased. This might be due to the removal of hemicellulose from the fibre surface [17] which resulted in better packing of the cellulosic chains.

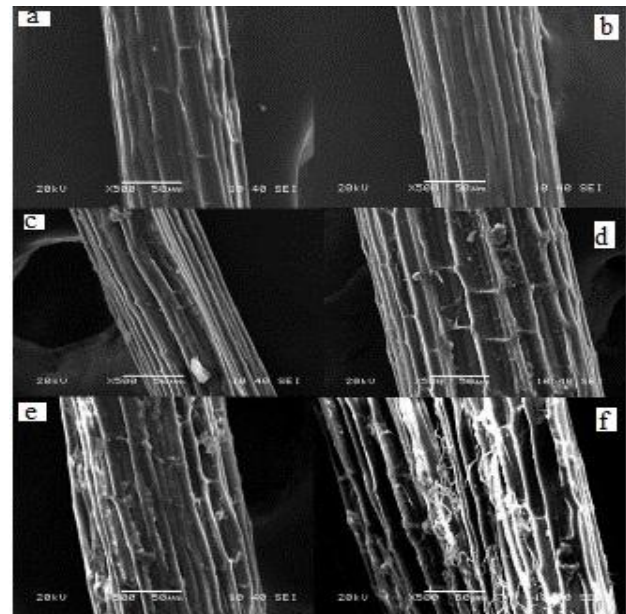


Fig. 3. a) Raw fibre, b) 2% NaOH treated fibre, c) 5% NaOH treated fibre, d) 10% NaOH treated fibre, e) 15% NaOH treated fibre f) 20% NaOH treated fibre.

**Table -2:** Physical properties of the fibres

NaOH concentrations, (%)	Average length L (cm)	Average diameter D (cm)	Aspect ratio L/D
Untreated	10	0.01952	512
2	10	0.01651	605
5	10	0.01430	699
10	10	0.01144	874
15	10	0.01070	934
20	10	0.00987	1013

### 3. FTIR analysis

The FTIR spectra of untreated and alkali treated AA fibre (Fig. 4) showed vibrations bands with corresponding to the fibre components such as cellulose, hemicellulose and lignin. All of them are summarized in Table 3. The peak between 1245  $\text{cm}^{-1}$  to 1259  $\text{cm}^{-1}$  almost disappeared after alkalization, indicating elimination of hemicellulose completely [8]. Also the presence of peak between 1500 –1600  $\text{cm}^{-1}$  in the alkali treated fibres confirmed that lignin component was intact. In general no other appreciable changes were observed in the vibrations of alkali treated fibres with respect to untreated AA fibre.

**Table -3:** Possible assignment of frequencies (cm<sup>-1</sup>) of groups in untreated and NaOH treated fibres functional

Untreated cm <sup>-1</sup>	2% NaOH	5% NaOH	10% NaOH	15% NaOH	20% NaOH	Possible assignment of functional groups
3390	3332	3350	3368	3381	3352	OH stretching $\alpha$ - cellulose [18 & 19]
2930	2923	2925	2923	2921	2924	Alkyl CH stretching [20]
2858	2885	2883	2880	2879	2884	CH <sub>2</sub> symmetric stretching (cellulose & hemicellulose) [20]
1731	-----	-----	-----	-----	-----	C=O stretching (carboxylic acid and ester groups) Hemicellulose [9 & 21]
1650	1645	1648	1646	1652	1653	Adsorbed OH water [22]
1541	1540	1542	1541	1540	1540	Aromatic skeletal vibrations of benzene ring in lignin [23-25]
1423	1419	1425	1425	1421	1421	CH <sub>2</sub> Bending [26 & 27]
1378	1372	1375	1378	1377	1375	C-H bending [8]
1321	1315	1316	1317	1315	1317	O-H deformation and CH <sub>2</sub> Wagging [26 & 27]
1255	-----	-----	-----	-----	-----	CH bending of hemicellulose [19 & 21]
1157	1155	1156	1157	1157	1159	Asymmetric C-O-C stretching of lignin [19 & 21]
1020	1023	1027	1025	1026	1026	C-O stretch/C-C stretch [28, 29, 24]
896	894	894	895	896	896	$\beta$ - glucosidic linkage [28, 29, 24]

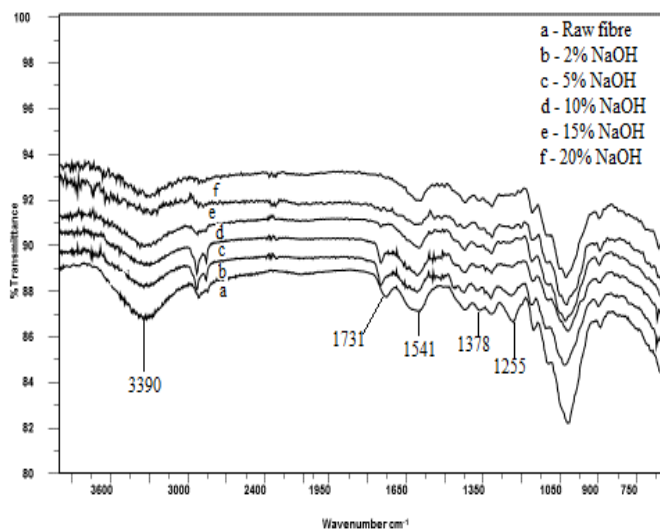


Fig. 4. a) FTIR spectra of untreated AA fibre. b) FTIR spectra of 2% NaOH treated AA fibre. c) FTIR spectra of 5% NaOH treated AA fibre. d) FTIR spectra of 10% NaOH treated AA fibre. e) FTIR spectra of 15% NaOH treated AA fibre. f) FTIR spectra of 20% NaOH treated AA fibre.

### 3.4. XRD analysis

The X-ray diffractograms of untreated and alkali treated AA fibres (Fig. 5) showed intense reflections obtained at  $2\theta$  values around  $16^\circ$  and  $22^\circ$ . The low angle reflection at  $16^\circ$  was of low intensity, representing I(am) of amorphous material and the high angle reflection at  $22^\circ$  showed higher intensity representing I(002) of crystalline material in lignocellulosic fibres.

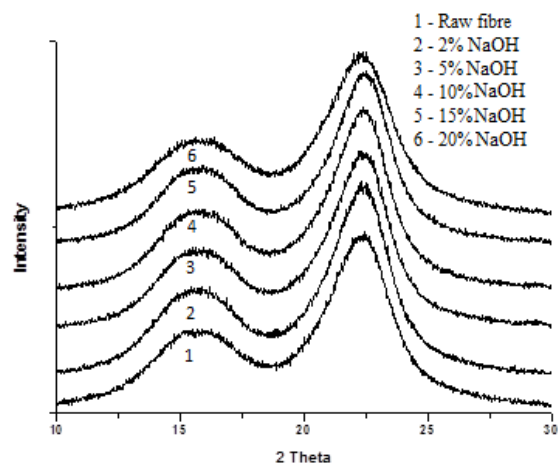


Fig. 5. XRD diffraction pattern of raw and NaOH treated fibres.



**Table -4:** Crystallinity Index of raw and NaOH treated AA fibres

Sample	I (am) at 2θ 15.5°	I (002) at 2θ 22°	CrI%
Raw AA	500	1129	55.71
2% NaOH	1105	2812	60.70
5% NaOH	1119	3111	64.03
10% NaOH	1112	3194	65.18
15% NaOH	1010	2981	66.22
20% NaOH	3061	1088	64.45

Similar observations were seen in other natural fibres too [30, 7, 8 & 31]. The crystallinity index of the AA fibre increased with the increasing concentrations of alkali treatment (Table 4) as compared to that of the untreated fibre. This may be due to alkali causing removal of the amorphous constituents from the fibre such as hemicellulose, pectin, waxes etc. and resulting in the closer packing and stress relaxation of the cellulose chains [32-34] (Table 4). Only at 20% of alkali treatment, the crystallinity decreased as the amount of the crystalline cellulose distinctly dropped indicating the molecular degradation of cellulose at this concentration [35].

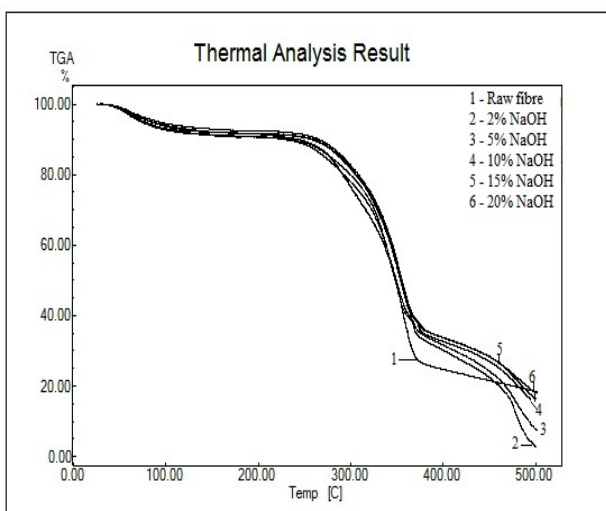


Fig. 6. TGA analysis of raw and NaOH treated fibre

### 3.5. TGA analysis

All lignocellulosic fibre mainly composed of cellulose, hemicellulose and lignin, degrade in three stages

namely waxes, pectin and hemicelluloses at 180 °C, cellulose at 300 °C and lignin at 400 °C [36 & 37]. The first, second and third stages were found in the temperature ranges of 50–125 °C, 200–300 °C and 300–400 °C respectively. The first stage degradation reflects elimination of moisture. The second stage degradation is reflective of degradation of hemicellulose and some part of the lignin. The third high temperature stage degradation where in α-cellulose and lignin degradation takes place [38 & 39]. Several natural fibres showed similar observations. The degradation temperatures of untreated and alkali treated fibres showed great differences. It was observed that the thermal stability of the alkali treated fibres was found to be greater than that of untreated fibre, which was because the alkalization treatment reduced the hemicellulose content and also gave rise to lignin-cellulose complex which was more stable to heat than that of untreated fibre sample [39]. The removal of the amorphous hemi cellulose causing progressive increase in the crystallinity of the fibres also facilitated improvement in thermal properties.

**Table-5:** Results of TGA analysis of raw and NaOH treated AA fibres

NaOH conc %	Peak temperature °C	Degradation %	Residue at 500 °C Char content, %
0*	83.60	8.62	12.08
	298.12	16.41	
	362.23	62.89	
2%	81.41	8.23	20.86
	294.14	15.60	
	360.17	55.31	
5%	80.21	8.19	22.88
	293.35	14.71	
	359.11	54.22	
10%	79.15	8.11	24.45
	285.05	13.94	
	355.23	53.50	
15%	76.08	7.95	33.16
	280.54	12.62	
	360.06	46.27	
20%	60.11	7.61	31.83
	271.54	10.85	
	359.22	49.71	

\* Untreated fibre

The residual char content at 500 °C also increased from 12.08% to 31.83% on increasing alkali concentration. (Table 5). The fibres treated with 2 to 15% alkali thus showed all round increase in thermal stability. However, at 20% alkali concentration, thermal stability of the fibres

declined resulting in decrease in residual char content which also supported the decrease in crystallinity.

### 3.6. Tensile properties and moisture content

The tensile strength of alkali treated AA fibre increased with increase in NaOH concentration. This increase in tensile strength was maximum at 15% alkali and it is attributed to increase in crystallinity as a result of hemicellulose removal [40].

Also in untreated raw fibre, however the strain remains intact in the cellulose chains because of the presence of hemicellulose which keeps the cellulose chains dissociated from each another, acting as the barrier for load distribution. However, 20% alkali treated sample showed decrease in tensile strength which is attributed to the degradation of cellulose at such high concentrations of alkali resulting in decrease in crystallinity. The elongation and moisture content values however, did not show appreciable changes in their values.

**Table- 6:** Tensile properties and moisture content of raw and NaOH treated fibres

Fibres	Tensile strength gf	Elongation %	Moisture content %
Raw	406.9	4.44	8.6
2% NaOH	411.2	4.65	8.2
5 % NaOH	455.3	4.98	8.1
10% NaOH	495.6	5.11	8.1
15% NaOH	522.4	5.85	7.9
20% NaOH	487.2	5.58	7.6

### 4. CONCLUSION

The AA fibres treated with different concentrations of NaOH showed distinct enhancement in their properties when compared to those of raw AA fibre. The chemical composition showed that the hemicellulose content decreased with increase in alkali concentration. This was due to ability of alkali to dissolves the hemicellulose. Surface morphology of the fibres became rougher due to removal of hemicellulose as a result of treatment with increase in alkali concentration. Further FTIR study confirmed the removal of hemicellulose as the functional groups indicating the presence of hemicellulose were either absent or the peak intensities gradually decreased as the alkali concentration increased.

These results were further supported by the XRD analysis showing increase in the crystallinity upto 15% NaOH treated samples. Beyond that, at 20% NaOH concentration the cellulose degradation took place showing distinct decrease in

the crystallinity of alkali treated AA fibre. TGA analysis also showed that the thermal stability increased as compared to that of raw fibre. It was observed that the residual char content increased in case of increase in alkali concentration till 15%. However, beyond that, at 20% NaOH, it decreased possibly due to the cellulose degradation and lesser crystallinity as observed earlier. Similarly tensile properties were also enhanced till 15% and then decreased at 20% concentration. It was observed that the AA fibre which was treated with 15% alkali was most suitable for further applications as it had over all enhanced properties.

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