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Study on properties of new biodegradable plant fiber (agave decipiens) for polymer reinforcement

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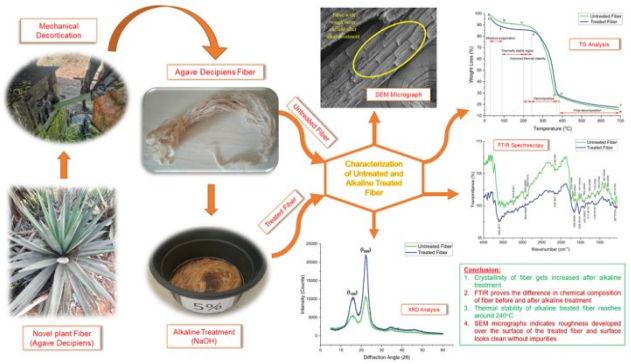
Graphical abstract



Abstract

The article involves in the process of study on novel plant fiber from agave plant species known as agave decipiens. The fiber was mechanically extracted from their leaves and fiber was chemically treated using sodium hydroxide by 5% (w/v). Using various analyses, the fiber was characterized and its properties were obtained. From chemical constituent analysis it was confirmed that hemicellulose, amorphous lignin, and other impurities were removed to some extent, and using x-ray diffraction (XRD), an improvement in crystallinity index (CrI) was observed (i.e. from 47.99% to 52.29%). Increased crystallinity provides better tensile stress from 479.302 MPa to 494.172 MPa, which was confirmed by single fiber tensile test. A change in physical diameter was observed using a digital microscope, the outer diameter was reduced to 117.66µm from 121.84µm. Change in chemical components was identified by Fourier Transform Infrared Spectroscopy (FTIR). Alkaline-treated (AT) fiber sustains a temperature of about 240°C during thermogravimetric analysis (TGA). Study on surface morphology was conducted with help of scanning electron microscope (SEM) Concluding that alkaline treatment made some impact on fiber characteristics and made it suitable for reinforcement.

Keywords: Biomaterial, natural fiber, agave decipiens, alkaline treatment, cellulose, crystallinity

1. Introduction

Materials having excellent mechanical property and less impact on the environment has prompted a slew of studies into bio-sourced polymer composites. Plant-based fibers attract the most scientific interest of any natural fiber because they are abundant, cost-effective, bio-degradable, and they possess reasonable mechanical strength (Santos *et al*., 2022). Characteristics of natural fibers are primarily influenced depending on fiber constituents like hemicellulose, amorphous lignin, cellulose, and other impurities say wax and dust particles. Fiber strength and stiffness were determined by the presence of these chemical components (Krika *et al*., 2021; Ramakrishnan *et al*., 2022).

Mostly all plant fibers were made-up of intricate structure, which contains a center channel as a lumen and covered by the cell wall. Lumen layer is used to transport food and water. The cell wall is made up of 3 layers: primary wall, secondary wall, and middle lamella. Primary wall consists of lignin, cellulose, pectin, and hemicelluloses. Crystalline cellulose will present in the secondary wall and it was surrounded by other chemical constituents. Hemicelluloses are adhered around the cellulose using hydrogen bonding, while the intermediate wall/lamella offers structural properties to the fiber. In plant fiber percentage of micro-fibril angle, cellulose, and hemicelluloses rules the strength of the natural fibers, which also differs from plant to plant (Latif *et al*., 2019). The fiber strength can be improved by various surface modifications, and an altered fiber surface has less attractive to moisture. That is hydroxyl group gets eliminated from the fiber which lowers the hydrophilic property (Kabir *et al*., 2012).

Komal *et al*. (2018) took 5% (m/v) sodium hydroxide solution for treating the banana fiber about 5 hours. The author confessed that fiber resin interfacial bonding and thermal stability were improved after chemical modification. Fractography justifies the change in the percentage of chemical components in untreated (UT) fiber and alkaline-treated (AT) fiber. (Guo *et al*., 2019) modified kenaf fiber using 5% sodium hydroxide followed by other oxidizing agents. The reduction of chemical constituents in the fiber was evident by chemical composition analysis, TGA, and FTIR tests. The author noticed that after chemical treatment, the fiber had less moisture absorption and

improved crystalline index and tensile strength. The author confirmed that solely alkaline treatment itself removes most of the chemical constituents from the fiber, if much improvement is required alkaline followed by hydrogen peroxide treatment was suggested. (Hamidon *et al*., 2019) concludes that surface modification increases the bonding between fiber and resin. Such treatment also reduces water adsorption i.e. hydrophilic property gets diminished. Mostly all surface treatments aided to improve fiber properties, fiber suitability, and fiber-resin adhesion. Mentioned that chemical treatment up to certain concentrations increased the mechanical properties and also provides better bonding between fiber and resin. Over the optimum concentration, alkaline treatment reduces mechanical characteristics. Water absorption also gets reduced because of the chemical treatment. Changes in fiber characteristics are observed using SEM and FTIR analysis and thermal behavior using TGA/DTG analyses (Venkatachalam *et al*., 2016).

Neto *et al*. (2019) carried out alkaline and saline treatment for hybrid fibers (jute, sisal, ramie, curaua). Used 2g of NaOH in 100ml of water and soaked the fibers for one hour and followed by a saline solution maintained under a pH of 5. The author admits that alkaline treatment increases fiber roughness and chemical treatment improves the fiber's thermal stability. Sgriccia *et al*. (2008) experimented with kenaf, flax, and hemp fibers by treating them using 5% sodium hydroxide solution for about an hour and followed by saline treatment. Author noted that lignin and hemicellulose are removed from the fiber, and traces of saline coating was observed using SEM. Thespesia Lampas plant fiber was immersed in a 2% (w/v) alkaline solution and left for one day. The finding revealed that mechanical strength of alkaline-treated (AT) fiber was better than untreated (UT) fiber. Fiber roughness was examined using SEM micrographs. Other analyses like chemical analysis, FTIR, XRD, and TGA support the chemical treatment done (Reddy *et al*., 2014). (Prithiviraj and Muralikannan, 2022) performed a surface modification using 5% NaOH alkaline solution for perotis indica fiber under different soaking times say, 1min, 30mins, 45mins, 60mins, and 75mins. The author concluded that fiber tensile value was improved in 60mins of soaking time compared to other timings. From TGA and DTG curves, thermal property of the fiber increased to 20^{o}C. XRD analysis proves that the crystalline index got raised from 48.3% to 55.43%. Presence of cellulosic components was proved by the FTIR spectrogram. The survey indicates that alkaline treatment can affect the surface of the fiber by eliminating undesirable substances, leading to a higher percentage of cellulose. This ultimately increases the fiber's strength, making it a viable material for polymer reinforcement. So, it is an indeed requirement to alter the fiber properties through chemical treatment mostly by alkaline solution.

Natural fiber-reinforced composite opens up new opportunities for all scientists and researchers, with an emphasis on the advantages of generating bio-degradable materials. From decades to the current scenario, enormous novel plant fiber varieties like abutilon indicum, saccharum bengalense grass, vernonia elaeagnifolia, purple bauhinia, furcraea foetida, juncus effusus L., cryptostegia grandiflora, fibers from stems of leucasaspera, cardio spermum halicababum, derris scandens, grewia damine, cissus vitiginea, barks of vachellia farnesiana, areca palm leaf stalk, calotropis gigantea fruit bunch, fiber from ficus religiosa tree roots, etc., were identified, characterized, and providing an alternative resource for composite reinforcement instead of synthetic fibers.

The study introduces a novel fiber (agave decipiens plant fiber) to the list of discovered novel fibers. The properties of agave decipiens fiber were not discovered or studied by any researchers and no significant research had examined predominantly in field of natural fiber composites. The study involves in the process of investigating the vital properties of untreated (UT) and alkaline-treated (AT) agave decipiens fiber to find its reinforcing capability in polymer composites

2. Resources and techniques

2.1. Extraction of fiber

Large, mature, fresh agave decipiens leaves were identified and obtained from the rural areas of Coimbatore district, Tamil Nadu, which was shown in Figure 1. At first, agave decipiens leaves were cut down from the plant, then the thrones at the two edges of the leaves were removed using a normal knife. Fibers were extracted using mechanical decortication method. By feeding the leaves inside a fast-rotating cylindrical roller, the pulps from the leave were removed by leaving the fiber alone. Next, the fiber was washed 2-3 times to remove the contaminates and it was allowed to dry in open sunlight. Figure 2, shows the extraction of agave decipiens fiber (Kathirselvam *et al*., 2019; Kumar and Sekaran, 2014; Thirumalaisamy and Subramani, 2018).

Figure 1. Agave Decipiens plant

Figure 2. Extraction of fiber from leaves

2.2. Surface treatment

The extracted raw fiber contains impurities and unwanted chemical substances over the fiber surface. So it cannot be directly used for reinforcement. If raw fiber was utilized for reinforcement without chemical processing there occurs a poor interlocking between fiber and resin, which obviously decreases the laminate strength. Water absorbance will easily take place because of the presence of hydroxyl functional group (–OH) in the form of hemicellulose. Considering these negative impacts, it is preferred to perform a chemical treatment. There are various types of fiber chemical treatment available and from which alkali treatment provides better results (Hamidon *et al*., 2019; Ramshankar *et al*., 2023; Venkatachalam *et al*., 2016).

For this research work, based on the literature study alkaline (NaOH) chemical treatment was performed for the new cellulosic agave decipiens plant fiber. At first, the fiber was cleaned with fresh water and immersed in a sodium hydroxide solution prepared with 5% (w/v) concentration and allowed to soak for up to 3 hours. Figure 3 demonstrates the alkaline treatment for agave decipiens fiber. The fiber was then rinsed once again with fresh water to eliminate any remaining sodium hydroxide traces, and dried at 100°C for half an hour. (Guo *et al*., 2019; Komal *et al*., 2018; Prithiviraj and Muralikannan, 2022; Sgriccia *et al*., 2008).

Figure 3. Alkali (NaOH) Treatment 5% (w/v)

2.3. Experimental analysis

2.3.1. Diameter measurement

Fiber diameter measurement is an essential function by which change in the diameter after alkaline treatment can be observed and it was very useful in calculating the fiber tensile value based on equation 1 (Guo *et al*., 2019; Puspita *et al*., 2023). A digital microscope was used for diameter measurement. Measurement was taken at various points and the average value can be calculated. To do this 25 samples of fibers each from both untreated (UT) and alkaline-treated (AT) fibers were used.

2.3.2. Single fiber tensile test

As per ASTM D3822-07 (Ding *et al*., 2022; Moshi *et al*., 2020), tensile strength of agave decipiens fiber (ADF) was tested using Zwick Roell Z010 tensile tester. 25 numbers of single fiber were randomly taken in both UT and AT fibers. Maintained a gauge length of around 75mm by constantly moving the crosshead at 5mm/min. The tensile test results provide the maximum load at which the fiber fails with respect to % of elongation.

$$\text{Tensile stress} = \frac{\text{Maximum Load at Failure}}{\text{Area of the fiber}}$$
$$\text{Tensile stress} = \frac{F_{max}}{\frac{1}{4}\pi d^2} \quad (1)$$

Where F_{max} is the maximum force at fiber failure and d denotes the diameter or cross-section (considering fiber has a circular cross-section)

2.3.3. X-Ray diffraction analysis

Fiber crystallinity was evaluated by Rigaku Ultima IV X-Ray diffractometer having Cu as target element used for the XRD measurement. The radiation was measured at 2θ between 5° and 60° under the rate of 2°/min with 0.001° step size. Equation 2 is known as Segal empirical method (Ding *et al*., 2022; Prithiviraj and Muralikannan, 2022; Vijay *et al*., 2020) which was used to calculate the CrI based on the peak intensities observed in the XRD chart. Using Debye–Scherrer equation 3 (Babu *et al*., 2022; Liu *et al*., 2019; Madhu *et al*., 2019) the crystalline size (CrS) of the fiber was calculated.

$$\%\ \text{of CrI} = \left(\frac{I_{200} - I_{100}}{I_{200}}\right) X\,100 \quad (2)$$

Where, I_{200} – Amorphous Peak Intensity, I_{100} – Crystalline Peak Intensity.

$$\text{CrS} = \frac{k\lambda}{\beta \cos\theta} \quad (3)$$

Where, I_{200} – Amorphous Peak Intensity, I_{100} – Crystalline Peak Intensity In equation (3), the Scherrer constant (0.89) is provided for k., λ known as X-ray radiation's wavelength (0.154 nm), β is referred to as full width at half maximum (FWHM) diffraction peak obtained from XRD peak, Bragg angle was denoted by θ.

2.3.4. Physico-chemical analysis

Chemical composition analysis of fiber is important to predict the presence of various chemical components, which gives the percentage of constituents that have been removed through alkali treatment. Based on the survey, pycnometer was used to measure the fiber density, where liquid toluene a known density (0.866 g/cc) was used as an immersion liquid (Kathirselvam *et al*., 2019; Ravindran *et al*., 2020). Cellulose was identified by Kursher and Hoffer's method and Conrad method was performed to identify the wax content (Manimaran *et al*., 2018; Khan *et al*., 2021; Rajeshkumar *et al*., 2021; Udhayakumar *et al*., 2023; Vijay *et al*., 2022). Hemicellulose and lignin were predicted using

NFT 12-008 (Ganapathy *et al*., 2019; Rajeshkumar *et al*., 2021; Vijay *et al*., 2022) and Klason method respectively (Shanmugasundaram *et al*., 2018; Vijay *et al*., 2021). Finally, Sartorius MA45 moisture analyzer was used to find the moisture content and ash content known by ASTM E1755-01 (Khan *et al*., 2021; Moshi *et al*., 2020; Vinod *et al*., 2021).

2.3.5. Fourier transform infrared spectroscopy

Chemical components in the form of chemical/functional bonds were observed using Schimazu IR Affinity 1S FTIR spectroscopy. With the help of functional group identification, change in the chemical components before and after alkaline treatment was identified using FTIR spectroscopy. To do this 2mg of fiber was crushed and mixed with zinc selenide (ZnSe) crystal for spectrum absorbance in a pallet shape and size. The spectral wavelength was measured between 4000cm^{-1} to 400cm^{-1} with a resolution of 0.5cm^{-1}.

2.3.6. Thermogravimetric analysis

SDT Q600 analyzer (TA Instruments) was used to investigate the thermal stability of agave decipiens fiber. A platinum crucible containing 2.5mg of powdered fiber was used for TG analysis, experiment was conducted between the temperature of 25^{o}C and 700^{o}C maintaining a constant heating rate of 10^{o}C/min under the influence of nitrogen environment. Due to change in the temperature, constituents present in the fiber gets decomposed, which results in weight reduction.

2.3.7. Differential scanning calorimetry

DSC Q20 (TA Instruments) was utilized to investigate the different thermal transition phases in untreated (UT) and alkaline treated (AT) agave decipiens fiber. To perform this analysis, fiber measuring of 2mg was taken in an aluminum pan and a heating rate of 10^{o}C/min was maintained. The complete heat flow study was done under a nitrogen gas atmosphere within the temperature range of 20^{o}C to 400^{o}C.

2.3.8. Scanning electron microscope

Outer structure, presence of porosity, and fiber surface roughness were observed using scanning electron microscope (SEM). Micrographs of UT and AT fiber were obtained using ZESSIS FESEM SIGMA VP 03-04 scanned at 2 kV. Meanwhile, natural fiber is a non-conductive material before conducting SEM analysis sputtering coating was performed to get clear views of micrographs.

3. Results and discussion

3.1. Diameter measurement

Sample measurement of fiber diameter using a digital microscope was given in Figure 4. Alkali treatment will have an impact on the fiber diameter. After alkaline treatment, fiber diameter gets reduced due to the elimination of unwanted impurities, cellulosic components, and waxy substances present over the fiber's outer surface (Chakravarthy *et al*., 2020; Guo *et al*., 2019; Maache *et al*., 2017; Moshi *et al*., 2020; Udhayakumar *et al*., 2023). Diameter values of UT and AT of the agave decipiens fiber were given in Table 1.

Table 1. Diameter values and experimental results of single fiber tensile test

Fiber Type	Diameter (µm)	The maximum force (F_{max}) (N)	% of elongation
UT fiber	121.84	5.5 N	2.8
AT fiber	117.66	5.3 N	2.3

3.2. Single fiber tensile test

As mentioned in the methods session, output of tensile test for a single fiber was obtained in the form of maximum load at fiber failure with respect to % of elongation. From the experimental values of UT and AT fiber, the average maximum force and percentage of elongation at break were statistically calculated and tabulated in Table 1.

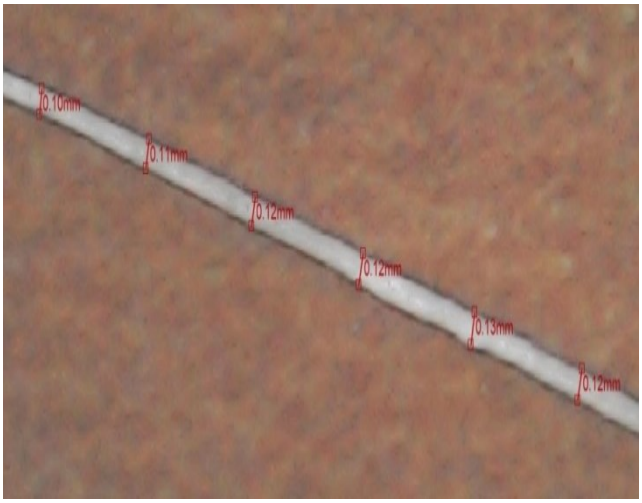


Figure 4. Sample diameter measurement of agave decipiens fiber (ADF)

Fiber tensile strength can be calculated using equation 1 and using hook's law, the elastic modulus of the fiber can be calculated, which was given in Table 2.

Table 2. Mechanical property of the UT and AT fiber

Fiber Type	Strain	Tensile Strength (MPa)	Elastic modulus (GPa)
UT fiber	1.028	479.302	0.466
AT fiber	1.023	494.172	0.483

From Table 2, it can be clearly understood that untreated fiber possesses less tensile value and less elastic modulus compared to alkaline-treated fiber. By the removal of unwanted substrates after chemical treatment, new hydrogen bonds are created with cellulose elements which results in the tight packing of elements between the interfibrillar region. Thus making the fiber more resistant to the load applied (Maache *et al*., 2017; Reddy *et al*., 2014; Udhayakumar *et al*., 2023).

3.3. X-Ray diffraction analysis

Using diffractogram, it was observed that there are two peak curves one is at minimum called amorphous peak (I_{100}) which contains cellulose, hemicellulose, amorphous lignin, and other impurities, and another peak at a maximum value called crystalline peak (I_{200}) which contains α-cellulose (Ding *et al*., 2022; Prithiviraj and Muralikannan,

2022; Vijay *et al*., 2020). Figure 5 provides the diffractogram of UT and AT fiber. From the analysis, it was noted that I_{100} (Intensities) for UT and AT fiber was 5591.67 (2θ=16.12°) and 10473.3 (2θ=16.06°) respectively, then I_{200} (Intensities) for UT and AT fiber was 10751.7 (2θ=22.16°) and 21,955 (2θ=22.24°) respectively. Calculated values of CrI and CrS were tabulated in Table 3.

Table 3. Calculated values of CrI and CrS for UT and AT fiber

Type of Fiber	Crystallinity Index	Crystalline Size (nm)
Untreated Fiber	47.99%	2.980
Treated Fiber	52.29%	4.721

Using equation 1, the crystallinity index of UT and AT of agave decipiens fiber (ADF) was determined to be 47.99% and 52.29%, respectively. From the graph, it can be noted that untreated ADF has a lesser intensity peak compared to alkaline-treated ADF, which proves that after alkali treatment mostly all amorphous constituents were removed and the crystallinity of fiber gets improved due to the increase in cellulose percentage. An increase in CrI is directly proportional to the increase in fiber mechanical properties and this could be the cause for the increase in tensile value of the AT fiber. Higher CrI indicates the reduction in moisture due to the absence of -OH molecules, which was proved by the crystalline size (CrS). Untreated fiber has 2.980nm and alkaline-treated fiber has 4.721nm of CrS. The crystalline size was increased after alkali treatment which signifies that the hydrophilic property gets reduced (Babu *et al*., 2022; Liu *et al*., 2019; Madhu *et al*., 2019).

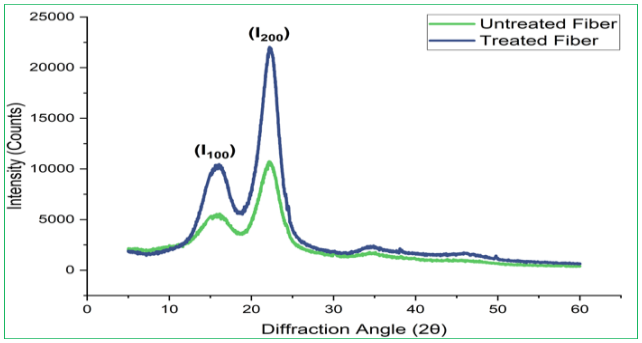


Figure 5. Diffractogram of UT and AT of agave decipiens fiber

3.4. Physico-chemical composition analysis

Every plant fiber consists of cellulosic (cellulose, hemicellulose, and lignin) and non-cellulosic (wax and impurities) substances. Depending on the type of plant, the climate, the soil in which the plant is grown, etc., the distribution of these components will change. Based on these components fiber properties like mechanical strength and thermal stability were determined (Jaiswal *et al*., 2022; Vijay *et al*., 2022). Table 4 gives the percentage distribution of chemical components in agave decipiens fiber.

After alkaline treatment, it can be seen that the decrease in hemicellulose was from 27.82% to 23.67%. Since hemicellulose readily reacts with NaOH (alkaline treatment) and gets detached from the fiber. Lignin has less sensitive to the action of sodium hydroxide so the removal of lignin was less which is about from 12.36% to 10.23%. The elimination of hemicellulose and some percentage of lignin after alkaline treatment decreases moisture absorption property of the fiber that is from 15.73% to 10.58%, in other words, hydrophilic characteristics get diminished, and fiber is now well suited for reinforcement with a matrix. Due to the elimination of contaminates after alkali treatment, the overall percentage of cellulose gets increased which is from 61.79% to 69.10%. An increase in cellulose percentage will significantly improve the fiber crystalline property that directly increases the fiber's mechanical strength.

Table 4. Various chemical constitutions of UT and AT fiber

Chemical constitutions	UT Fiber	AT Fiber
Cellulose %	61.79	69.10
Hemicellulose %	27.82	23.67
Lignin %	12.36	10.23
Wax %	0.48	0.25
Ash %	3.71	3.22
Moisture %	15.73	10.58
Density (g/cc)	0.89	1.17

Raw agave decipiens have higher cellulose content compared to other novel fibers like saccharum bengalense grass, Ficus religiosa, Grewia damine, phoenix pusilla, and thespesia lampus. Phaseolus vulgaris, Derris scandens, Calpotropis gigantean fruit, Cissus vitiginea, and perotic indica have higher cellulose than raw agave decipiens fiber. Table 5 compares the density and presence of various chemical components of agave decipiens with other natural fibers.

During reinforcement, wax content may lead to poor bonding between fiber and resin, that lower the mechanical property as well as the tribological property of the composite. After alkaline treatment, wax content was reduced to 0.25%. The percentage of components present in the fiber was likely removed after chemical treatment, as evidenced by the decrease in ash content from 3.71% to 3.22%.

Finally, fiber density was increased from 0.890 (g/cc) to 1.17 (g/cc) after chemical modification. This is because of the removal of non-cellulosic particles having smaller density values and the filling of chemical molecules in between the voids and pores on the fiber surface. Agave decipiens fiber has lesser density, so it can be employed in lightweight applications. All of the preceding statements disclose that the alkaline treatment has an influence on the agave decipiens fiber and that it is appropriate for reinforcing in polymer composites.

3.5. Fourier transform infrared spectroscopy

FTIR was analyzed for both UT and AT fiber which was given in Figure 6 and various absorbance peaks with respective functional groups was tabulated in Table 6. The wave absorbance between 3800cm^{-1} and 3000cm^{-1} shows the existence of (-OH) hydroxyl functional group, which indicates that moisture content was available in both AT and UT fiber (Madhu *et al*., 2019; Madhu *et al*., 2020; Manimaran *et al*., 2018; Narayanasamy *et al*., 2020; Ravindran *et al*., 2020). Two peak absorbances say 2924.08cm^{-1} and 2860.43cm^{-1} were noticed between the

wave band 3000cm^{-1} and 2500cm^{-1}. This peak occurrence happened by stretching of methyl functional groups that are (CH-) and (CH_2-). In alkaline-treated fiber, less absorbance was noticed in the respective two peaks because of the elimination of hemicellulose after alkaline treatment (Babu *et al*., 2022; Liu *et al*., 2019; Madhu *et al*., 2019; Narayanasamy *et al*., 2020; Sarala *et al*., 2020). Stretching vibration of alkyne functional group (C≡C) can be absorbed in the peak wavenumber 2158.35cm^{-1}, which relates to the wax substance present in the fiber (Madhu *et al*., 2019; Madhu *et al*., 2020; Moshi *et al*., 2020; Sarala *et al*., 2020).

Table 5. Comparison of density and various chemical compositions of agave decipiens fiber with other fibers from different sources

Different natural fiber		Cellulose%	Hemicellulose%	Lignin%	Wax%	Moisture%	Density(g/cc)	Reference
Agave Decipiens	UT Fiber	61.79	27.82	12.36	0.48	15.73	0.890	Current work
	AT Fiber	69.10	23.67	10.23	0.25	10.58	1.170	
Saccharum bengalense grass		53.45	31.45	11.7	1.3	2.1	1.165	Vijay *et al*., 2020
Ficus religiosa		55.58	13.86	10.13	0.72	9.33	1.246	Moshi *et al*., 2020
Grewia damine		57.78	14.96	16.65	0.59	-	1.378	Ravindran *et al*., 2020
Phoenix pusilla		59.46	18.56	8.28	0.33	-	0.211	Madhu *et al*., 2019
Thespesia lampus		60.63	26.64	12.70	0.76	10.83	1.412	Reddy *et al*., 2014
Phaseolus vulgaris		62.17	7.04	9.13	0.36	6.1	0.934	Babu *et al*., 2022
Derris scandens		63.3	11.6	15.3	0.81	6.02	1.430	Sarala *et al*., 2020
Calpotropis gigantean fruit		64.47	9.64	13.56	1.93	7.27	0.457	Narayanasamy *et al*., 2020
Cissus vitiginea		65.43	14.61	10.43	0.39	8.47	1.287	Chakravarthy *et al*., 2020
Perotic indica		68.4	15.7	8.35	0.32	9.54	-	Prithiviraj and Muralikannan, 2022

Peak absorbance between 1750cm^{-1} and 1500cm^{-1}, 1730.14cm^{-1} peak was exhibited in UT fiber which shows the stretching of carboxyl and ester functional groups (–COO), indicating the existence of hemicellulose and lignin. Meanwhile, this peak absorbance was zero in AT fiber because of the elimination of hemicellulose and some amount of lignin (Ding *et al*., 2022; Liu *et al*., 2019; Shaker *et al*., 2020; Vijay *et al*., 2020). The peak absorbance noticed at 1653cm^{-1} and 1656.85cm^{-1} in the UT fiber and AT fiber respectively signifies the vibration of carbonyl and acetyl functional groups (C=O), from this presence of some percentage of hemicellulose and lignin, can be confirmed (Babu *et al*., 2022; Moshi *et al*., 2020; Ravindran *et al*., 2020;). Stretching of alkene functional group (C=C) occurs at the wavelength of 1529.55cm^{-1} and 1517.97cm^{-1} in the AT and UT fiber respectively, which indicates the presence of aromatic lignin (Ding *et al*., 2022; Vijay *et al*., 2020).

Three peaks absorbance say, 1402.25cm^{-1}, 1317.38cm^{-1}, and 1261.45cm^{-1} were noticed between the wavelength 1500cm^{-1} to 1250cm^{-1}, which attributes to methyl and acetyl functional groups. This indicates the existence of cellulose (CH_2-) and aromatic lignin, hemicellulose (C-O) respectively (Sgriccia *et al*., 2008; Shaker *et al*., 2020). Peak absorbance at 1317.38cm^{-1} was present only in alkaline-treated fiber indicating the increase in cellulose percentage after alkali treatment. Wavelength absorbance between 1250cm^{-1} and 1000cm^{-1}, a sharp narrow peak absorbance can be seen in untreated fiber at 1122.57cm^{-1} and 1022.27cm^{-1}, which attributes to the bending of (C-O-C) and (C-O) which indicates the existence of polysaccharides of cellulose and pyranose ring of cellulose respectively. A board peak absorbance was noticed at 1138cm^{-1} wavelength because of the improvement in cellulose percentage in AT fiber (Guo *et al*., 2019; Moshi *et al*., 2020).

The β-glucosidic linkage between the monosaccharide of cellulose occurs at the peak wavelength of 806.25cm^{-1} and 887.26cm^{-1} (Chakravarthy *et al*., 2020; Liu *et al*., 2019; Reddy *et al*., 2014; Maache *et al*., 2017). Final peak can be witnessed at 607.58cm^{-1} which was accredited to (C-OH) and occurs due to out-of-plane bending of cellulose (Ding *et al*., 2022; Guo *et al*., 2019; Sarala *et al*., 2020). FTIR analysis confirmed the presence of chemical components in the fiber with the aid of functional groups It was also possible to determine how the chemical composition changed in proportion followed by alkaline treatment. FTIR inference proves that cellulose percentage was increased due to chemical treatment.

Table 6. Wavenumber and presence of functional groups identified using FTIR analysis

Wavenumber (cm^{-1})	Absorbance		Chemical composition	Functional groups
	UT Fiber	AT Fiber		
3800 - 3000	All peaks within the range		(-OH)	Hydroxyl groups
3000 - 2500	2924.08, 2860.43		(CH-) & (CH_2-)	Methyl groups
2500 - 1750	2158.35		C≡C	alkynes
1750 -1500	1730.14	-	(–COO)	carboxyl and ester
	1653	1656.85	(C=O)	carbonyl and acetyl
	1517.97	1529.55	(C=C)	alkene
1500 - 1250	1402.25		(CH_2-) & (C-O)	methyl and acetyl
	-	1317.38		
	1261.45			
1250 - 1000	1122.57		(C-O-C) & (C-O)	polysaccharides of cellulose and pyranose ring of cellulose
	1022.27	1138		
1000 - 750	887.26, 806.25		-	β-glucosidic linkage of cellulose
750 - 500	607.58		(C-OH)	Out-of-plane bending of cellulose

Table 7. Consolidated TGA results of both fibers

Curve points		Temperature (°C)	% of weight loss	Decomposition
Untreated Fiber	Treated Fiber			
a to b	x to y	25 to 80	6% and 4%	Moisture removal
b to c	y to z	80 to 200 and 80 to 240	Negligible	Thermally stable
c to e	z to v	200 to 340 and 240 to 380	60%	Cellulose decompose
e to f	v to t	above 360	14% and 11%	Wax and amorphous lignin

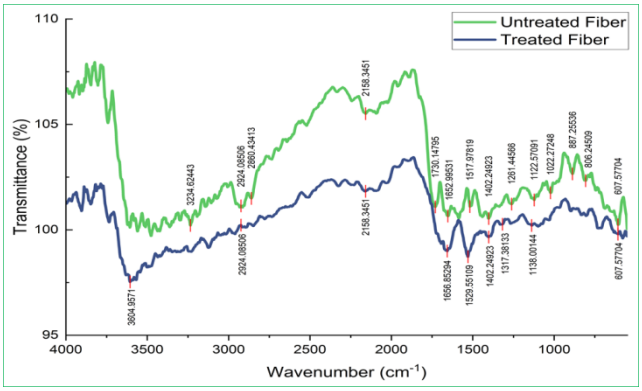


Figure 6. FTIR spectroscopy for UT and AT fiber

3.6. Thermogravimetric analysis

Decomposition of elements and fiber's thermal stability can be studied using thermogravimetric analysis. Figures 7 and 8 gives the detailed interpretation of TGA and DTG curves for UT and AT agave decipiens fiber and summarized values of TGA results were tabulated in Table 7. Thermal stability can be identified by the percentage of weight loss at an increasing order temperature (Narayanasamy *et al*., 2020; Shaker *et al*., 2020).

The curve can be divided into 4 regions. In the first region viz., presence of moisture in the fiber absorbs the temperature and evaporates which results in slight weight reduction, which can be seen in the curve from a to b and x to y (Rajeshkumar *et al*., 2021; Udhayakumar *et al*., 2023; Vijay *et al*., 2021). At this stage, weight loss will be around 4% to 6% for the temperature rise from 25°C to 80°C. When the temperature was raised to above 80°C, the fiber has very negligible weight loss which can be considered as a straight line that is from point b to c and y to z., showing that fiber was thermally stable between 80°C to 200°C. When compared to untreated fiber, alkaline-treated fiber has greater thermal stability and can withstand temperatures up to 240°C. (Arun Ramnath *et al*., 2023; Binoj *et al*., 2016; Jebadurai *et al*., 2019).

Next in 3rd region viz., from c to e and z to v heavy weight loss can be noticeable which was about 60% for the temperature rise of 200°C to 380°C. During this phase hemicellulose, α-cellulose, and lignin gets decomposed. Most decomposition of cellulose occurs above 320°C (Ganapathy *et al*., 2019; Rajeshkumar *et al*., 2021; Vinod *et al*., 2021). Alkaline-treated fiber consumes an additional 20°C to 30°C of temperature to decompose compared to UT fiber, this is because of the elimination of non-cellulosic contaminates using alkaline treatment. This was also evidenced by DTG curve as shown in Figure 7. UT fiber takes 340°C of temperature to get weight loss, whereas treated fiber takes up to 360°C of temperature, which proves that thermal performance was improved after alkaline treatment (Ganapathy *et al*., 2019; Kathirselvam *et al*., 2019; Manimaran *et al*., 2018).

Final decomposition takes place at the temperature range of 380°C to 700°C, where the remaining amorphous lignin and waxy substances get decomposed. The weight loss was found to be 14% and 11% for UT and AT fiber respectively. The elimination of an undesirable component from the fiber during the alkaline treatment accounts for the variation in weight loss (Vijay *et al*., 2021; Vinod *et al*.,

2021). As a result, both TGA and DTG curves were very useful in finding the thermal stability for both fibers.

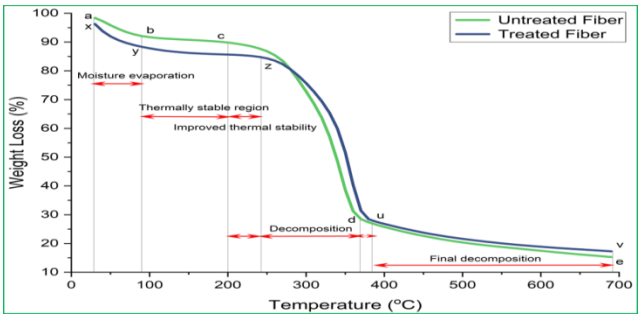


Figure 7. Thermogravimetric analysis of UT and AT of agave decipiens fiber

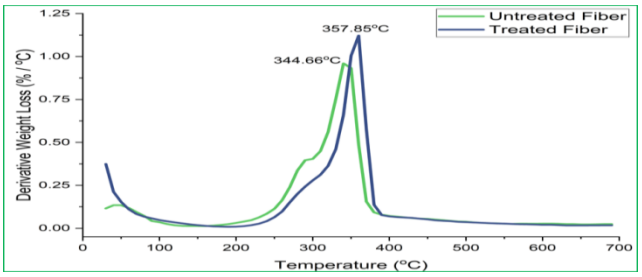


Figure 8. Derivative thermogravimetric of untreated and treated agave decipiens fiber

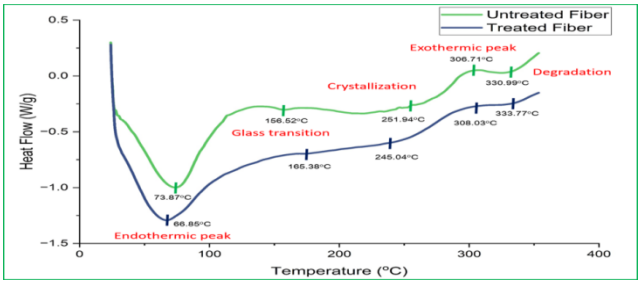


Figure 9. Differential Scanning Calorimetry for UT and AT fiber

3.7. Differential scanning calorimetry

Figure 9 shows the DSC curvature for untreated and alkaline-treated agave decipiens fiber. DSC curve is used to support the TGA analysis for the same fiber. From Figure 9 it can be noted that an endothermic peak occurs between 60°C to 80°C in both fibers. During this phase, the moisture absorbs the heat supplied to the fiber and gets evaporated (Madhu *et al*., 2019; Madhu *et al*., 2020; Narayanasamy *et al*., 2020). Temperature around 150°C to 170°C is known as glass transition phase. Fiber starts changing its phase to crystallinity where most of the hemicellulose and other unwanted substances get degraded. The curve moves upward at the temperature above 250°C, where the crystallinity peak occurs. At this phase, a small amount of amorphous lignin and cellulose were removed and above 350°C, the exothermic peak occurs during which all the constituents get burnt up (Ganapathy *et al*., 2019; Kathirselvam *et al*., 2019; Madhu *et al*., 2020; Zakikhani *et al*., 2014). The DSC curve well agrees with the TGA results for UT and AT fiber.

3.8. Scanning electron microscope

SEM micrographs for both UT and AT of agave decipiens fiber were obtained from examination, which was shown in Figures 10 and 11. SEM morphology is a good method to understand and study the outer structure of the fiber, mainly it is useful in investigating the change in the outer surface before and after surface modification.

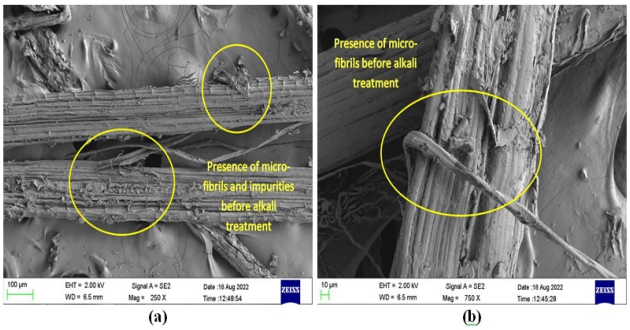


Figure 10. Fiber with impurities and micro-fibrils

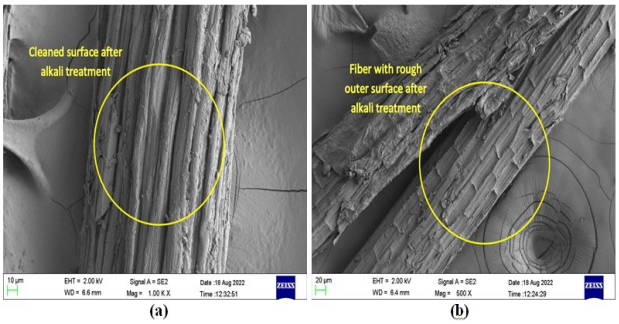


Figure 11. Fiber with a clean and rough surface

In the UT fiber small micro-fibrils and other surface impurities can be clearly visualized in Figure 10 (a) & (b). Absence of micro-fibrils and non-cellulosic impurities was prominently able to be seen in the micrograph (i.e.) in Figure 11 (a). Compared to UT fiber, AT fiber looks clean and roughness has been developed on the fiber surface which was given in Figure 11 (b).

Presence of roughness over the surface helps the fiber to properly merges with the resin during reinforcement (Arun Ramnath *et al*., 2023; Manimaran *et al*., 2018; Manimaran *et al*., 2022; Shanmugasundaram *et al*., 2018). From SEM micrographs it was able to understand that alkaline treatment made some impact on the agave decipiens fiber.

4. Conclusion

Agave decipiens a new plant fiber was successfully extracted by mechanical decortication method and imparted to chemical treatment using sodium hydroxide with 5% (w/v) concentration. In this research work characterization of both UT and AT of agave decipiens fiber was executed. Amount of chemical components present in both fibers was obtained using chemical composition analysis, in which the cellulose content gets improved due to the deduction of unwanted cellulosic components and impurities. This change in chemical composition was supported by FT-IR spectroscopy analysis. An increase in cellulose content directly increases the crystallinity in the fiber which was proved by the X-Ray diffractogram. Tensile test on single fiber confirms the increase of tensile modulus in alkaline-treated fiber. The thermal decomposition of UT and AT of agave decipiens fiber was studied using TGA, in which fiber treated with alkaline solution withstands higher temperature than untreated fiber. Using SEM micrographs

the morphology of the fiber was studied. In untreated fiber, small micro-fibrils and impurities were able to be identified. Meanwhile, in alkaline treated fiber, the surface looks clean and roughness was created because of the impact produced by the NaOH reaction with fiber. This change was able to identify by outer diameter measurement from that reduction of diameter can be witnessed in alkaline treated fiber. From this research study, various properties of newly identified plant fiber (agave decipiens fiber) were successfully characterized for both untreated and alkaline-treated fiber. Experimental results proved that the agave decipiens fiber can be used as a reinforcement after performing chemical treatment. In a future study, the impact of varying concentrations of alkaline solution or different chemical treatments can be investigated and compared.

Conflict of Interest

Declaring no conflict of interest.

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