Comparison Of Fibers Properties Of Azadirachta Indica And Acacia Arabica Plant For Lightweight Composite Application

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COMPARISON OF FIBRES PROPERTIES OF AZADIRACHTA INDICA AND ACACIA ARABICA PLANT FOR LIGHTWEIGHT COMPOSITE APPLICATIONS

POREĐENJE OSOBINA VLAKANA BILJAKA AZADIRACHTA INDICA I ACACIA ARABICA ZA PRIMENE KOD LAKIH KOMPOZITA

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Keywords
- Azadirachta Indica
- Acacia Arabica
- physical analysis
- chemical analysis
- X-ray diffraction
- Fourier transform-infrared spectroscopy
- thermogravimetric analysis
- SEM

Ključne reči
- Azadirachta Indica
- Acacia Arabica
- fizička analiza
- hemijska analiza
- Rentgenska difrakcija
- Furijeova transformacija-infracrvena spektroskopija
- termogravimetrijska analiza
- SEM

Abstract

In this paper, the bark of Azadirachta Indica and Acacia Arabica plant is analysed for investigating and comparing. The aim is to investigate the potential use of these fibres as reinforcements in polymeric materials. The physicochemical properties of Azadirachta Indica fibres (AIFs) and Acacia Arabica fibres (AAFs) are examined by chemical constitutions, X-ray diffraction, thermogravimetry analysis, Fourier transform infrared spectroscopy analysis, and surface morphological analysis. AIF has a cellulose content of 68.42 wt.%, density of 740 kgm^{-3}, crystallinity index of 65.04%; AAF has a cellulose content up to 68.1 wt.%, density equal to 1028 kgm^{-3} and crystallinity index of 51.72% respectively. The maximum peak temperature obtained in differential thermogravimetry (DTG) curve is 321.2°C for AIF, and 345.1°C for AAF. The physicochemical results confirm the structural application of AIF and AAF for several industrial fields.

Izvod

U radu su date analize kora biljaka Azadirachta Indica i Acacia Arabica radi istraživanja i poređenja. Cilj je da se istraži potencijalna primena ovih biljnih vlakana kao ojačanja u kompozitnim materijalima. Fizikohemijske osobine vlakana Azadirachta Indica (AIFs) i vlakana Acacia Arabica (AAFs) dobijene su ispitivanjem hemijskog sastava, Rentgenskom difrakcijom, termogravimetrijskom analizom, Furijeovom transformacijom – infracrvenom spektroskopijom i analizom morfologije površine. AIF ima celulozni sastav 68,42 % tež., gustina je 740 kgm^{-3}, indeks kristaliteta 65,04%; AAF ima celulozni sastav do 68,1% tež., gustina je 1028 kgm^{-3}, i indeks kristaliteta 51,72%. Najveća dostignuta temperatura na krivoj diferencijalne termogravimetrije (DTG) jeste 321,2°C za AIF i 345,1°C za AAF. Fizikohemijski rezultati potvrđuju strukturnu primenu AIF i AAF kod nekoliko industrijskih primena.

INTRODUCTION

Recently, growing environmental care and sustainability needs throughout the world have increased the attention of researchers to search for better alternatives to synthetic fibres (such as glass, carbon and aramids) /1-4/. Referring to environmental aspects in fact, these synthetic fibres have some disadvantages /5/, but fortunately nature always comes in handy for providing several good alternative materials /6/. These difficulties are overcome by good fibres yielding plants which are cost-effective without compromising mechanical properties. The applications of natural fibres are growing in many sectors such as aerospace, automotive, construction, and packaging industries /7-8/. This is mainly due to their excellent features compared to synthetic fibres, i.e., low cost, low density, cost-effectiveness, high toughness, non-toxic, renewable, recyclable, non-abrasive and biodegradable properties /9-13/. The chemical constituents of bark fibres are strongly dependent on the age, local environmental conditions of the plants /14/. Natural fibres can be extracted from different parts of plants such as stems, leaves, roots, fruits, and seeds. Among all the plant fibres, bark fibres are longer and possess highest degree of compatibility between fibre and matrix. Fibre extracts from the bark of Acacia planifrons, Prosopis juliflora, Grewia optiva, Grewia tiliifolia, Cordia dichotoma, Sterculia urens, Acacia leucophloea, and Thespesia lampas proved as evidence for good reinforcements in polymer composites /2, 4, 13, 15-21/.

Azadirachta Indica, well known as Neem tree is pertaining to the mahogany family Meliaceae. It is native in India and it typically grows in tropical and semi-tropical regions. Numerous products made from Azadirachta Indica have been used in India over two millennia because of its inherent medicinal properties. The inter-node length, width, and thickness of average untreated AIF were found to be around 120 mm, 1.27 mm and 1.84 mm, respectively /22/. Acacia Arabica plant was brought to India in 1860 as fuel wood, as the native trees in India in arid and semiarid regions could not fulfil the demands for fuel. It had the capacity to grow in all climatic conditions except in the frost zones of the Himalayan region. It could survive where the annual rainfall was between 150 and 750 mm and maximal temperature 40-45°C and also, they can survive without water for several months /23/. These fibres can be used for making green composites which will be helpful in the applications like light weight sports goods, roofing sheets, door panels, furniture panels, storage tanks, bath units, chairs, partitions, trays, tables, etc /8/.

A pycnometer is used to assess the density of fibres and a chemical analysis is performed to determine their lignin, cellulose, wax, moisture and ash content. The chemical analyses of Azadirachta Indica (AI) and Acacia Arabica (AA) fibres are determined, using X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analysis. The thermal degradation properties are investigated, using thermogravimetric analysis (TGA). A microscopic examination is carried out with a scanning electron microscope (SEM).

MATERIALS AND METHODS

Materials

The fresh and seasoned barks of A. Indica are collected in K. Vellakulam village, situated near Kallikudi in Thirumangalam Taluk, Madurai district, Tamil Nadu, India, Fig. 1(a). The barks of A. Arabica are collected in Lakshmipuram, Tirumangalam Taluk, Madurai district of Tamil Nadu, India, Fig. 1(b). The barks of the plant are submerged in water for a maximum period of 3 weeks to allow microbial degradation, and the fibres are extracted by water-retting processes. The fibres are segregated from the degraded barks with a metal teeth brush and thoroughly washed in fresh water. Then the extracted fibres are dried for 1 week at atmospheric temperature to allow maximal removal of moisture for further testing, /6/.

Physical Analysis

The fibres cross-section and their microstructural characteristics and density are presented in this section. SEM is used to analyse the fibres cross-section. Their dimension is calculated by considering the images of a primary and secondary wall of fibres, by means of an image processing software (Image J, NIST) using the SEM images, /24/. The average cross-section diameter is determined by using the 100 mm fibre length considering at least 5 SEM images, /24/. The fibres density is estimated by pycnometer (Mettler Toledo xsz05 balances) for solids by means of an immer

Figure 1. a) Azadirachta Indica tree, and b) Acacia Arabica tree.

sion liquid known as the Toulon. Initially, the fibres are dried for 48 h in an impermeable no hygroscopic desiccator containing calcium chloride; then impregnated in Toulon for 2 h to remove the microbubbles present in the fibres before measuring the density of the fibres. The fibres are chopped into length of 10 mm and kept into the pycnometer /25/. The fibres density, ρ_{Fibres}, is calculated by the following equation, /25-28/:

$$\rho_{Fibers} = \frac{m_2 - m_1}{(m_3 - m_1)(m_4 - m_2)} \rho_T \ , \qquad (1)$$

where: m_1 is the mass of the empty pycnometer (kg); m_2 is the mass of the pycnometer filled with chopped fibres (kg); m_3 is the mass of the pycnometer filled with Toulon (kg); and m_4 is the mass of the pycnometer filled with chopped fibres and Toulon solution (kg).

Chemical Analysis

The fibres density is evaluated using the Mettler Toledo xsz05 balances method /25/, and its wax content is calculated by Conrad's method, /29/. The chemical composition of fibres cellulose, hemicelluloses, lignin and ash content is determined by standard test methods, /30-31/.

CHARACTERIZATION METHODS

X-ray diffraction (XRD) analysis

Today the use of X-ray techniques for qualitative and quantitative examination of materials is very useful in view of the scatter, emission and absorption properties of X-radiation (X-ray). The phase identification of fibres can be done by XRD analysis. This analysis is carried out by means of a Bruker Eco D8 Advance AXS system. An X-ray tube generates monochromatic intensity of Cu Kα radiation in the 10° to 80° range at a scan speed of 5 °/min. The generator works at 40 kV and 30 mA. The crystallinity index (CI) is assessed through the following equation, /32-33/:

$$I_c = \left(1 - \frac{I_{am}}{I_{002}}\right) \times 100\%, \quad (2)$$

where: I_{002} is the intensity of crystalline peak; and I_{am} is the intensity of amorphous peak in the XRD spectrum. The crystallite size (CS) of the fibres is worked out by the following equation:

$$CS = \frac{K\lambda}{\beta \cos\theta} \quad (3)$$

where: $K = 0.89$ is Scherrer's constant; β symbolizes the peak's full-width at half-maximum; and λ denotes the wavelength of the radiation.

Fourier transform-infrared (FTIR) analysis

FTIR spectrum of fibres is recorded by using Shimadzu spectrometer (FTIR-8400S, Japan) to determine the presence of free functional groups on the fibres. In this analysis, powdered fibre sample of potassium bromide (KBr) pellets are used. The FTIR spectrum was recorded in the range of 4000-500 cm^{-1} region at a temperature of 30 °C and at 65% humidity. The spectrum is identified with a scanning rate of 32 scans·min^{-1} to achieve an acceptable signal-to-noise ratio with a resolution of 2 cm^{-1}.

Thermogravimetric analysis

Thermogravimetric analysis measures the amount of fibres weight changes as a function of increasing temperature in an atmosphere of nitrogen. Thermal stability of the fibres is analysed by TGA using a Jupiter simultaneous thermal analyser (Model STA 449 F3, Netzsch, Germany), by studying the weight loss and transformation. The measurement is performed in high purity nitrogen atmosphere at a flow rate of 20 mL·min^{-1}, and the weight loss is recorded at a heating rate of 10 °C·min^{-1} in the temperature range of 30-600 °C. Measurements are made by using an alumina crucible to maintain a good contact between the sample and thermocouple.

Surface morphological analysis by SEM

A scanning electron microscopy (Carl Zeiss EVO 18) was operated with an accelerated voltage of 20 kV and magnification of 300 × for fibres visualization. In order to avoid the accumulation of electrical charges during analysis, the sample is covered with a thin gold layer.

RESULTS AND DISCUSSION

Physical analysis

The fibres cross-section of both AI and AF are approximately round in shape and used to determine the diameter of the fibre. The fibre cross-section varied by varying fibres length and they are determined by SEM at various location. Five samples are used to measure the diameter of the fibre at four points with equal spacing through 100 to 1000 mm long fibres. The fibre diameter evaluated is up to 40-250 μm and 30-180 μm for AIF and AAF, respectively. Figure 2 shows the SEM images of AIF and AAF.

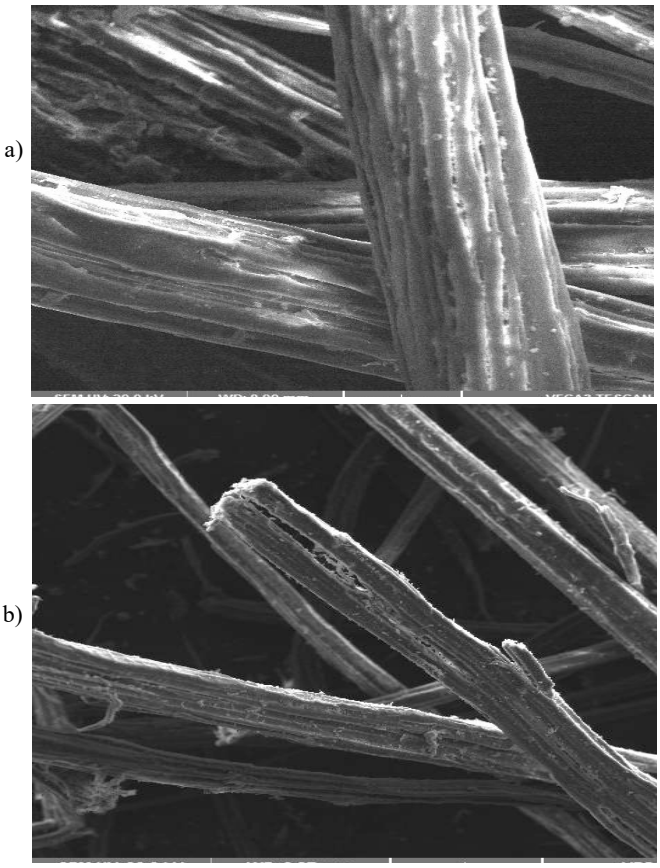

Figure 2. SEM images of the longitudinal section of: a) AIF and b) AAF.

Chemical analysis

The chemical composition of fibres is compared with various other bark fibres reported in scientific literature and listed in Table 1. The chemical composition of the fibres, cellulose, hemicellulose and lignin are strongly affected by the age of the plant, soil characteristics, extraction conditions and methods used to determine the composition, /34/. The assessed AIFs cellulose (68.42 wt.%) is better than AAFs cellulose (68.1 wt.%), but both of them are greater than other bark natural fibres such as Prosopis juliflora (61.65 wt.%), Cordia dichotoma (59.7 wt.%), Grewia tilifolia (62.8 wt.%), and Oil palm empty fruit bunch (41 wt. %) /2, 16, 17, 35/. Usually, fibres tensile strength and Young's modulus are proportional to the cellulose content, /36-37/. The hemicellulose content of AIFs is equal to 13.72%; its degradation usually leads to fibres collapse into

Table1. Comparison of chemical compositions of raw AIFs and AAFs with various natural fibres.

Fibre name	Cellulose (wt.%)	Hemicelluloses (wt.%)	Lignin (wt.%)	Wax (wt.%)	Moisture content (%)	Density (kg/m^3)	Ash (wt.%)
AIFs	68.42	13.72	13.58	0.43	-	740	-
AAFs	68.10	9.36	16.86	0.49	-	1028	-
Acacia planifrons	73.1	9.41	12.04	0.57	8.21	660	4.06
Prosopisjuliflora	61.65	16.14	17.11	0.61	9.48	580	5.2
Sansevieriacylindrica	79.7	10.13	3.8	0.09	3.08	915	-
Cyperuspangorei	68.5		17.88	0.17	9.19	1102	-
Grewia Tilifolia	62.80	21.20	14.90	-	2.3	-	-
Oil palm empty fruit bunch	65	29	17.5	4	-	-	
Henequen	60	28	8	0.5	-	-	-
Agave	68.42	4.85	4.85	0.26	7.69	1200	-
Sansevieria ehrenbergii	80	11.25	7.8	0.45	10.55	887	0.6

cellulose microfibrils resulting in lower strength due to the linking effect. Hemicellulose content (9.36 wt.%) of the AAFs is considered a compatibilizer between cellulose and lignin. The lignin content of AIFs is 13.58 % and it influences the fibre structure, properties, and morphology. Higher lignin content is evaluated for AAFs (16.86 wt.%). It saves water in the fibres, acting as a protection against biological attack and contributing to the fibres structure and morphology; it provides good rigidity compared to the other fibres too, /38/. The lowest wax content is evaluated for AIFs (0.43 %). It affects the interfacial bond between fibres and matrix during fabrication. Therefore, to improve the interfacial bonding between the AIFs and resin, fibres chemical treatment has to be done in order to remove the wax and moisture content, /25/. However, AAFs contain endurable wax (0.49 wt.%), which may lead to poor interfacial bonding between fibres and polymer matrices, /19/. Of significance are both densities, 740 kgm^{-3} for the AIFs, and 1028 kgm^{-3} for AAFs. This analysis reveals that AIFs will be much useful to make light-weight composite structures.

X-ray diffraction (XRD) analysis

Figure 3 presents X-ray diffraction pattern of AIFs and AAFs. It displays two clear diffraction peaks at 2θ: 15.03° and 22.64° for AIFs, and 22.45° and 15.08° for AAFs, which are commonly witnessed for natural fibres. In AIFs, the peak 15.03° reveals the occurrence of non-cellulosic materials such as hemicellulose, amorphous cellulose, pectin and lignin in the fibres, while the peak 22.64° indicates the content of cellulose in the fibre, /37/. However, in AAFs, the minimal intensity peak is observed at 18.39°, which contains a higher percentage of amorphous fraction (lignin, pectins, hemicellulose, and amorphous cellulose). The calculated CI for AIFs is 65.04 % and CI for AAFs is 51.72 % which are much higher than that of Prosopis juliflora (46 %) and Acacia leucophloea (51 %), and lower than that of Acacia planifrons (65.38 %), jute (71 %) and hemp (88 %) fibres, /13/. The crystallite size (CS) of AIFs is estimated by Scherer's formula as specified in Eq.(2) and the value of CS is found to be 2.75 nm. CS in AIFs poses chemical reactivity and water absorption capacity properties. Besides, the CS of the AAFs estimated as 15 nm reduces the water absorption capacity and chemical reactivity of the fibre, yet it is comparable to that of flax fibre (2.8 nm) and quite close to that of ramie fibre (16 nm). This result reveals that these fibres will be used when cost reduction is the prime consideration, and can effectively replace the relatively expensive materials.

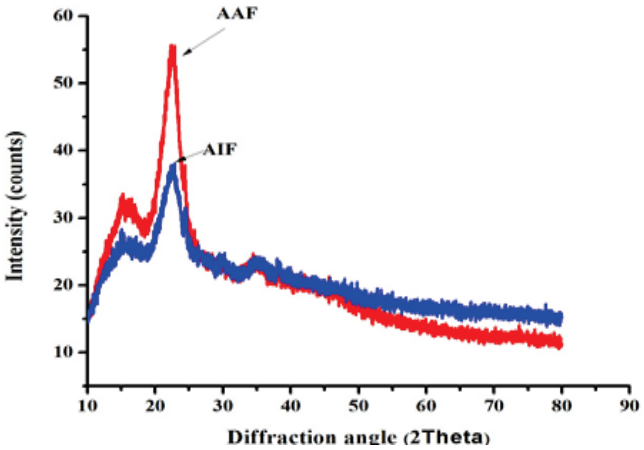

Figure 3. X-ray diffractrogram of AIFs and AAFs.

Fourier transform-infrared (FTIR) analysis

Figure 4 summarizes the functional groups of the AIFs spectrum in the range of wave numbers from 4000 to 500 cm^{-1}. The FTIR spectrum shows absorption bands of different chemical groups of bio-fibre components such as cellulose, hemicellulose and lignin. The major components are alkenes, phenolic hydroxyl group, aromatic groups, and several functional groups having oxygen (ester, ketone and alcohol), /39/. In AIFs, A 'U' shape arises about 3700-3000 cm^{-1} which are related to the hydrogen bonded O-H (alcohol group) stretching vibration from the cellulose arrangement of the AIFs, /40/. The peak at 2917 cm^{-1} characteristic bands for the C-H stretching vibration from CH in cellulose and the peak at 2366 cm^{-1} showed the existence of wax which is allotted to the less ordered band of the alkyl chain. The CH_2 symmetric bending band is estimated at a peak of 1413 cm^{-1}, /41/. The carbonyl region is absorbed in the peak at 1618 cm^{-1} is allocated to O-H bending vibration in hemicellulose region. The bending vibration C-O group of aromatic ring lignin shows peaks at 1048 cm^{-1} stretched vibration. However, one of the most noticeable peaks in the AAFs spectrum appeared at 3442 and 1022 cm^{-1} corresponding to O-H stretching and O-H bending frequencies, respectively, /20/. The two sharp peaks at 2922 cm^{-1} and 2854 cm^{-1} are assigned to pronounced C-H stretching vibration of CH and CH_2 in cellulose and hemi-

cellulose components, /42/. The absorption peak at 1741 cm^{-1} is attributed to the carbonyl group stretching vibration of the alpha-keto carboxylic acid in lignin or the ester group in hemicellulose. The carbonyl region is projected at 1700 cm^{-1} and the peak at 1649 cm^{-1} is assigned to C-O stretching vibration of the acetyl group in lignin and hemicellulose. The bending vibration of C-H and C-O group of the aromatic ring of hemicellulose and lignin are observed at 1462 cm^{-1} and 1325 cm^{-1} stretching vibration.

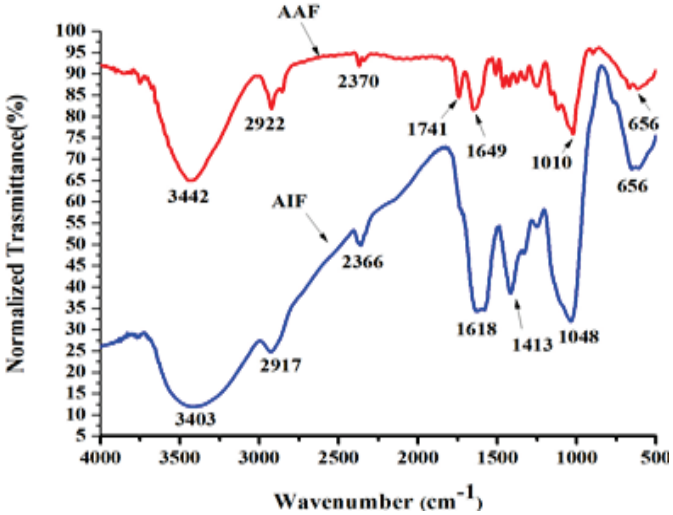

Figure 4. FTIR analysis of AIFs and AAFs.

Thermogravimetric analysis

The thermal stability performance of fibres is investigated using TG curves as shown in Fig. 5. The initial degradation of AIFs is spotted in range from 50 to 230 °C, /42/. The second degradation of AIFs developed from the temperature range of 240-400 °C in which a peak fairly near to 300 °C associated to thermal depolymerization of hemicellulose with 13.54 % of mass loss is detected. Another noticeable peak at 321.2 °C indicates the degradation of cellulose with a foremost weight loss of about 34.51 % /43/. Comparable results are also detected at 331.1 °C, 345.1 °C and 333.02 °C for Prosopis juliflora, Acacia Arabica, Sansevieria ehrenbergii, respectively /2, 4, 43/. The ending region from 450 to 500 °C denoted significance of oxidative degradation of the charred residue. The weight loss may be associated with dehydration as well as degradation of lignin content in fibre. Typically, two stages of degradation are observed during the AAFs thermal stability study. The initial degradation noticed (at 84.4 °C) between room temperature and 100 °C could be due to the evaporation of moisture present in the AAFs. The second major degradation progressed from the temperature region of 240-400 °C in which a peak quite close to 300 °C related to thermal depolymerization of hemicellulose with 16.20 % of mass loss is noticed, /38/. A very prominent peak at 345.1 °C showed a major mass loss of about 52.63 % due to the degradation of cellulose. Similar results are also observed at 346.8°C, 331.1°C, 321°C, 308.2°C, 298.2°C and 309.2°C for A. leucophloea, P. juliflora, bamboo, hemp, jute, and Kenaf fibres, respectively, /43/. However, the degradation of the aromatic structure of lignin occurred slowly under the initial temperature up to 600 °C, /45/.

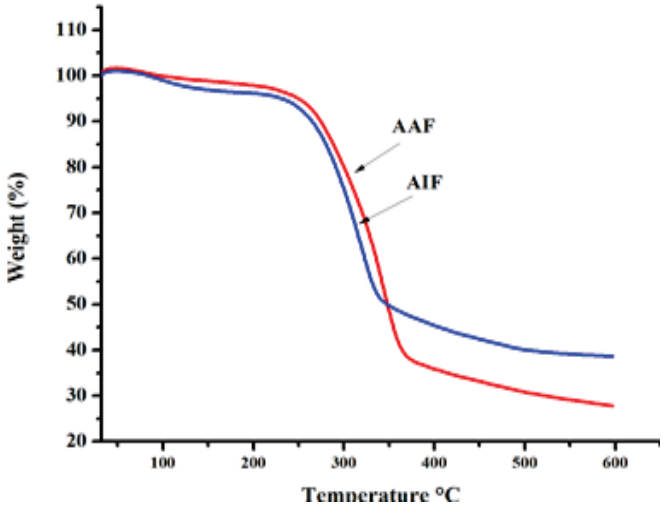

Figure 5. TG curves of AIFs and AAFs.

Surface morphological analysis by SEM

Morphology studies of AIFs and AAFs are carried out using a scanning electron microscope to evaluate the fibre surface structure, Fig. 6(a) and (b). The changes in morphology are significant to forecast the fibre interaction with the polymer matrix in composites. In general, all-natural cellulose fibres are multi-cellular, where lignin and hemicellulose bind a bundle of individual cells, /40/.

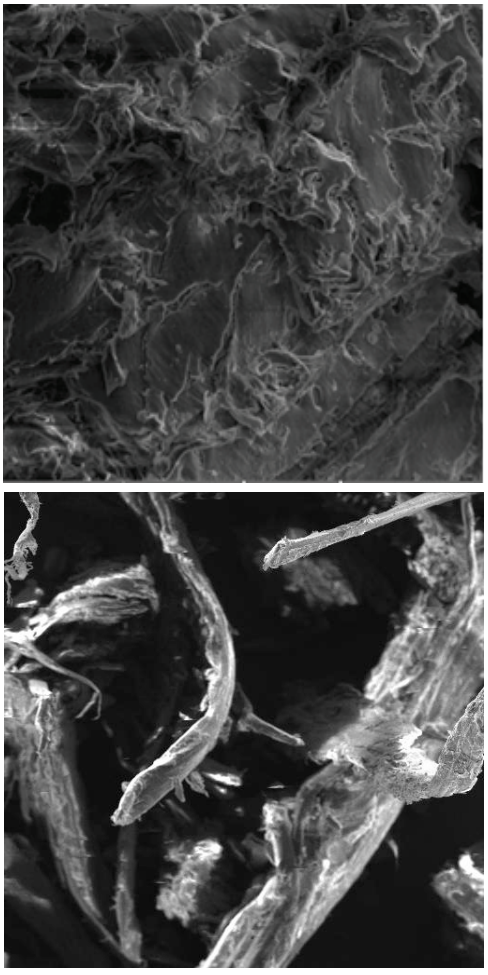

Figure 6 (a). SEM images of cross-section of AIFs.

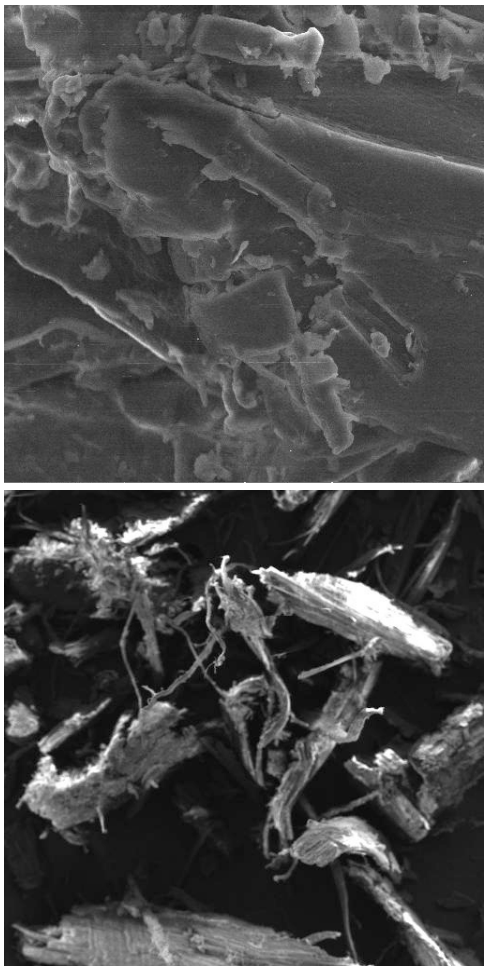

Figure 6 (b). SEM images of cross-section of AFFs.

CONCLUSION

In this investigation, the chemical composition, FTIR analysis, crystalline characteristic, SEM and thermal degradation of AIFs and AAFs are studied. The high amount of cellulose in AIFs and AAFs can provide a relatively high crystallinity index and the lower density of AIFs and AAFs exposed to make them suitable for lightweight composite applications. The thermal stability analysis demonstrated AIFs and AAFs cellulose degradation temperature. The characterization results clearly show that the AIFs and AAFs are a better alternate reinforcement of making composites. These characterization results also confirm its usage of AIFs and AAFs for various applications.

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