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Physical, Chemical, Morphological and Thermal Characterisation of Natural Fibers for Sound Absorption

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ABSTRACT

The increasing concern over the waste reduction and health hazards associated with the use of synthetic fibers such as glass fibers, carbon fibers, aramides and other fibers have turned the attention towards natural ecofriendly green fibers. In this paper natural fibers namely – milkweed fiber, Agave americana fiber and Sansevieria roxburghiana fibers have been studied experimentally to determine the physical, chemical, morphological and thermal properties by FTIR, SEM, PLM, X-Ray diffraction, DSC and TGA analysis methods. In this study, Sansevieria fiber was observed to have the highest tenacity of 53.58 g/tex and showed maximum degradation temperature of 375 °C. Milkweed fibers were found to have the lowest density of 0.9 g/cc and possessed hollow structure with smooth surface as observed from SEM and PLM techniques.

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KEYWORDS

Natural Fibers, FTIR, SEM, PLM, TGA

1. INTRODUCTION

Natural fibers have wide range of applications in the field of textile, automotive and aerospace, particularly in the light of the recent global inclination towards eco-friendly textiles. The emerging green economy is based on energy efficiency, renewable feed stocks in polymer products, industrial processes that reduce carbon emission and recyclable materials. Natural fibers are renewable resources and they have good mechanical strength, low weight and low cost, that has made them particularly attractive to the automotive industry [1]. The utilization of natural fibers as a reinforcement for composite materials is an emerging research area. In recent years, attempts have been observed to reduce the use of synthetics and expensive glass, aramid or carbon fibers and also lighten considerably the car's body by taking advantage of the lower density and cost that natural fibers provide [1].

Milkweed is a type of seed hair fiber which consists of single cell, unlike bast fibres. These fibres, which are attached to the side wall of the seed, gets dispersed by wind. The typical seed-hair fibres are similar in morphology to cotton, with long lengths and small diameter. Milkweed fibres are hollow with a thin wall and are therefore lightweight. The hollow structure of milkweed fibre has led to its use in items where good insulation or buoyancy properties are needed. Agave and sansevieria fibers are collected from the bast surrounding the stem of their respective plant.

In a literature, pyrolysis of sugar cane bagasse and coconut fiber was studied by thermal analysis by TGA and DSC methods and chemical constituents were determined by FTIR method [2]. Jute/Gelatin composites were characterized for its physical, mechanical and morphological properties and it showed good results [3]. In another study of hemp and kenaf fibers, the effect of alkali and saline treatment on its physical properties and also their FTIR, XPS and ESEM analysis were carried out to study its properties in comparison with glass fiber [4]. Blends of

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agave, pineapple leaf fibers, polypropylene fibers, non woven and composites were tested for its mechanical and dynamic mechanical properties and observed that agave-polypropylene fibers possessed superior mechanical properties [5]. A review of agave Americana fibers was performed stating its physical properties, extraction, characterization and its applications [6]. Another review of milkweed fibers concluded about its versatility as an industrial textile material due to its suitable physical and morphological properties exhibited by the fibers [7]. A blended non woven form of milkweed and kapok fibers were studied for their acoustic and thermal properties and found a positive correlation between fabric density and sound reduction and a negative correlation between bulk density and sound reduction, and the effect of kapok fibers with thermal conductivity [8]. Sansevieria cylindrica fiber was studied for its dynamic, mechanical, surface and thermal properties and the effect of alkali treatment on fiber properties and corresponding effects on the mechanical and thermal properties were analyzed [9]. Various other studies were done to characterize the properties of natural fibers, its composites and its nonwovens [1, 10-16].

With respect to the materials considered in this paper, structural properties of milkweed fibers have been studied along with cotton blend [17]. Mechanical characteristics of milkweed and its other properties have also been addressed in the literature [7, 18]. With respect to agave Americana, some studies have been carried out to identify its mechanical and crystalline characteristics [19-21]. A few literature deals with analyzing the properties and physical characteristics of sansevieria roxburghiana fibers [22, 23].

The objective of this paper is to characterize the physical, morphological, chemical and thermal properties of three natural fibers namely – Agave Americana, Sansevieria Roxburghiana, and milkweed fibers. The properties of these three natural fibers were determined experimentally and compared.

2. MATERIAL AND METHOD

2.1. Material

The milkweed fibers were obtained from matured pods of the plant asclepias syriaca and the floss was extracted from the pods by hand and partly dried. Complete drying was done by spreading the floss on hessian sheets in the sun for about three days until the floss becomes lustrous in appearance and a fair amount of dust and dirt separates out from the floss. On drying, the floss loses about 5% of its weight. The agave Americana and sanseviera roxbhurghiana were extracted by water retting method. Initially the fibers were retted in stagnant water for 7 days and then extracted by mechanical extraction technique. The bast was passed through the rollers of a specially designed machine, which crushed them to separate the fibers and they were washed in running water and dried.

2.2. Physical Properties

The physical properties such as length, fineness, strength, density and moisture were determined as per the ASTM standard test method. Single fiber properties of milkweed fiber were measured and the fiber lengths were also determined by comb sorter diagram as per BS 4044:1989 standard. Linear density of the material was measured by gravimetric method using the ASTM test method D1577 at standard atmosphere conditions. The fibers were placed flat on a cutting device, using the template for standard length (L) the fiber samples were cut and weighed (W). The denier (D) of the fibre sample was calculated using the equation (1).

$$E = \frac{9000W}{LN} \qquad (1)$$

The tensile properties of fibres were determined using ASTM D3822-01 standard. After pre-conditioning the samples in a standard atmosphere, the testing was carried out with gauge length of 10 mm. The test was repeated with 50 samples and the values were recorded. The fibre density was determined with a density gradient column having a mixture of xylene and carbon tetrachloride as per ASTM D1505-03 standard, after the fibers were cut into small pieces and made into small balls, otherwise leading to very low density values due to air bubbles inside the canal of the

(a) (b) (c)

Figure 1. Extracted fibers (a)Agave Americana (b) Sansevieria Roxburghiana (c) Milkweed

fibers. The moisture regain values were measured as per ASTM D 2654-89a by measuring the weight loss of the sample continuously with an interval of 30 minutes with respect to increasing temperature upto 105 ^{o}C until there was no loss in weight.

2.3. Surface Characterisation

The surface morphology of the fibers was studied to understand the behavior of thermal and acoustic properties of the fiber. The surfaces of fibers were observed using scanning electron microscope. Prior to the test, the samples were coated with a thin layer of gold by a plasma sputtering apparatus. The observation was performed in high vacuum mode with secondary electron detector and accelerating voltage between 5 and 10 kV. With an optical microscope (Leica Projection Light Microscope, Model: DM750P), the longitudinal and cross-sectional views of fiber samples were photographed with a magnification of 200x. Average of fifty randomly chosen readings was taken to compute the mean fiber diameter.

2.4. Chemical Characteristics

The crystallinity of raw, alkali treated and dyed milkweed fibers were evaluated by X-ray diffraction. X-ray diffractograms of the samples were obtained with Lab-X, wide angle X-ray diffractometer, having an X-ray tube, producing monochromatic radiation (λ = 1.54 Å) at 30 kV and 20 mA. To calculate the crystallinity index, software was used to separate the background and the overlapped peaks. The crystallinity index of the fibre was calculated according to the Segal empirical method as given in equations (2) and (3).

$$CrI\ (\%) = \frac{I_{002} - I_{am}}{I_{002}} * 100 \qquad \text{(II)}$$

$$Cr\ Ratio = \frac{I_{002}}{I_{002} + I_{101}} * 100 \qquad \text{(III)}$$

where I_{002} is the maximum intensity of the I_{002} lattice reflection and I_{am} is the height of the minimum between the 002 and the 101 peaks. FT-IR spectra of the fibers (raw, alkali treated, dyed) were recorded using Shimadzu FT-IR in KBr matrix with a scan rate of 32 scans per minute with a resolution of 4 cm^{-1} in the wave number region of 400-4000 cm^{-1}. The fiber samples were chopped into smallest particles and ground well. Then it was mixed with KBr and pelletized by pressurization to record the FT-IR spectra under standard conditions.

2.5. Thermal Characteristics

A Perkin Elmer differential scanning calorimeter and Netzsch thermogravimetric analyzer were used to measure the thermal behavior of the fibers. The sample consisting of 2-3 mg of fibers were cut finely with scissors and placed in aluminum pans for DSC analysis & platinum pan for TGA analysis. Both DSC and TGA were performed in nitrogen gas atmosphere flowing at 50 ml/min. The DSC thermograms of the conditioned (25 °C, RH 75%) raw and treated fiber samples were recorded on Perkin Elmer DSC 7 as well as in Netzsch DSC 204 instruments from room temperature to 400 °C and 200 °C respectively at a heating rate of 10 °C/min in nitrogen atmosphere.

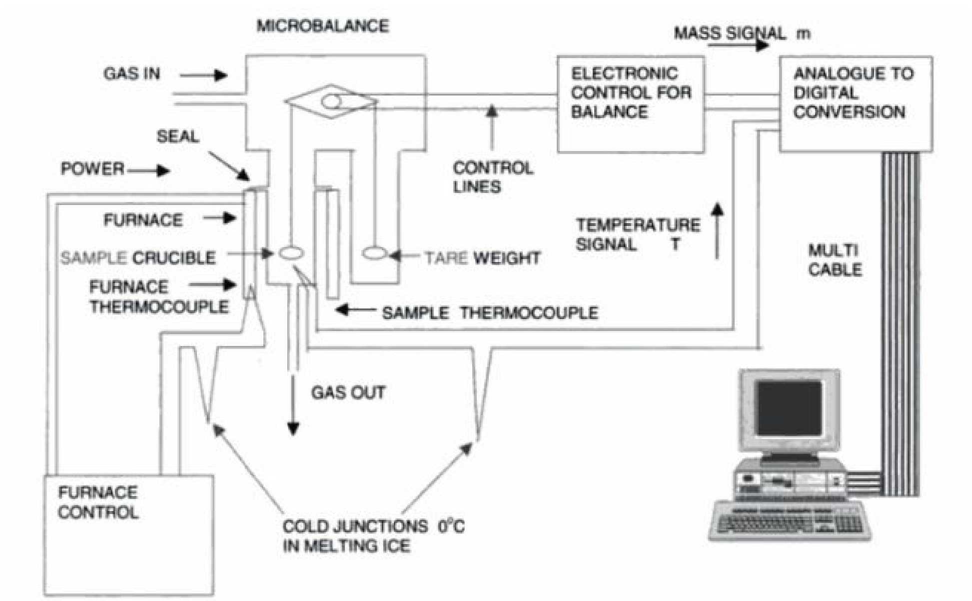


Figure 2. Schematic diagram of thermogravimetric analysis [24]

3. RESULTS AND DISCUSSION

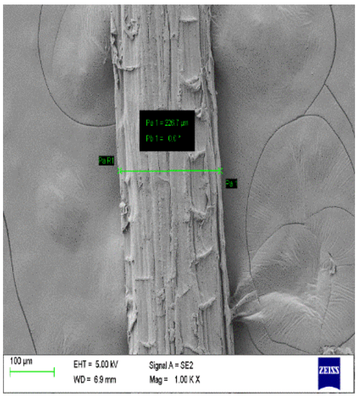
The physical properties of selected fibers such as Milkweed, Sansevieria and Agave are shown in Table 2. The tenacity of the Sansevieria fibers are the highest compared to the three fibers that were selected for the study, which is in the range of the hemp fibers. Agave Americana is a stiff fiber but the tenacity is lower than that of milkweed. Milkweed being a seed hair fiber is finer, whereas Sansevieria has optimum fineness in the range of other bast fibers. Agave Americana fibers are coarser, but if they are fibrillated, the fineness can be improved. The two bast fibers have density comparable to the other bast fibers, while milkweed is lighter than water. The moisture absorption properties of all the three fibers were almost in the same range as that of other bast fibers.

From Figure 3a to Figure 3c it is clear that the surface morphology of the Agave and Sansevieria fibers are similar to that of the other bast fibers. They have a multi cellular structure and very rough surface morphology with many striations. These rough surfaces are as a result of the frictional contact of the fibers between each other. From Figure 3b it can be seen that the milk weed fibers have a very smooth surface morphology. This smooth and lustrous effect on the fiber surface is observed as a result of the wax content in the fibers.

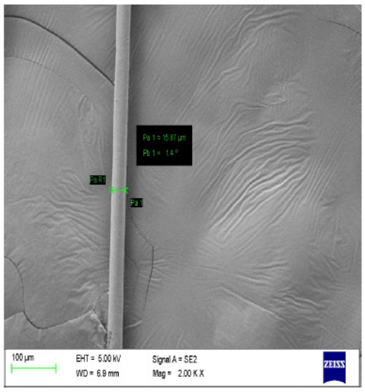
Like cotton, milkweed fiber is a single cell fiber but without convolutions. It is evident from Figure 4b that the milkweed fibers are hollow in nature, which aids in better capillary action and air permeability, while the other fibers are completely solid structures. In case of agave Americana there are few fibrils floating on the surface of the fiber. Both the bast fibers have uneven surface morphology along the width and diameter of the fiber varying across the length of the fibers.

Table 2. Physical properties of fibers

Property	Milkweed	Sansevieria	Agave Americana	Jute	Hemp
Tenacity (g/tex)	16.575	53.58	13.423	30	55
Fineness (tex)	0.11	5.33	26.22	1.8	20
Density (g/cc)	0.9	1.35	1.38	1.45	1.48
Moisture Content (%)	10.4	10.5	8.85	12.5	8



(a)



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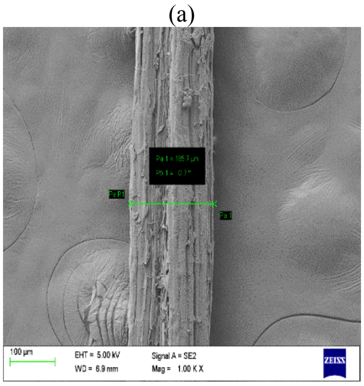
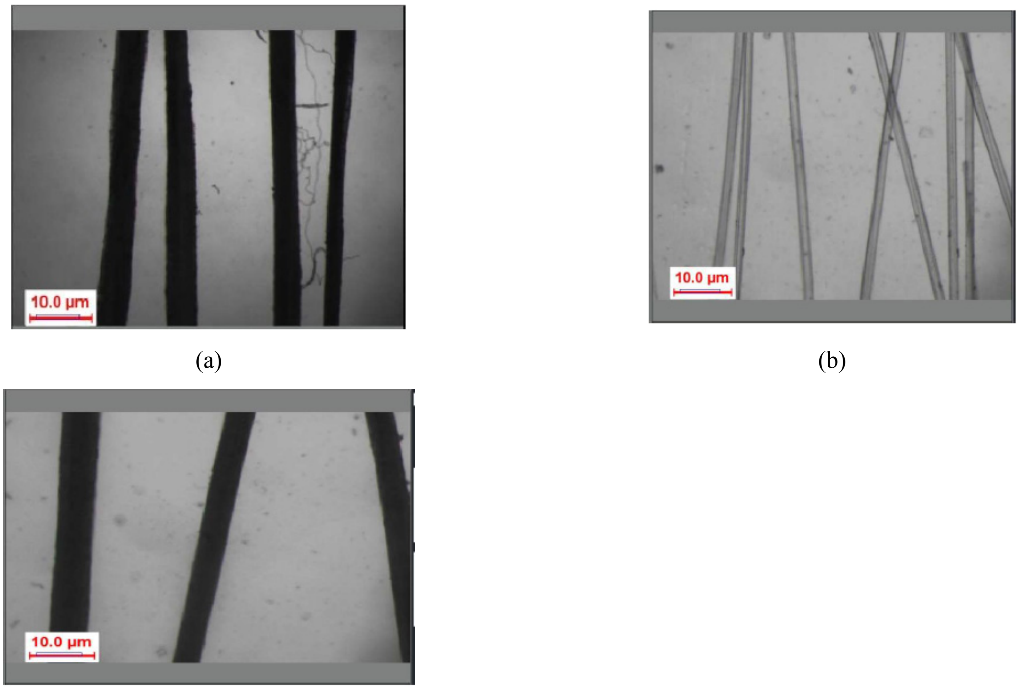


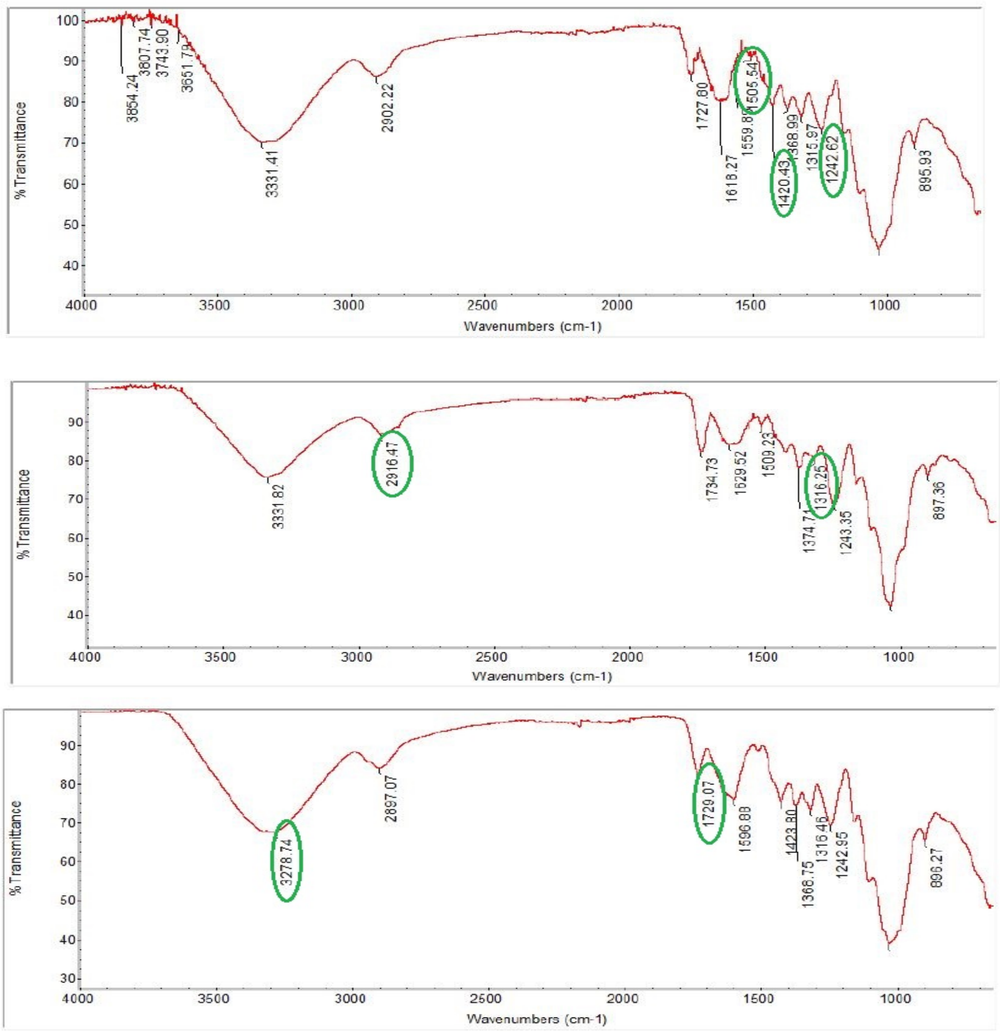
(c)

Figure 3. SEM images of fibers (a) Agave Americana (b) Milkweed (c) Sansevieria



(a) (b) (c)

Figure 4. Longitudinal structure of (a) Agave Americana (b) Milkweed (c) Sansevieria



(a) (b) (c)

Figure 5. FTIR graphs of (a) Agave Americana (b) Milkweed (c) Sansevieria

From Figure 5, it can be observed that both the bast fibers have functional groups like lignin, proteins and hemicelluloses (identified by the presence of certain carbon linkages) that are easily soluble. In case of the milkweed fibers polysaccharides form the major portion due to the presence of O-H and C-H linkage.

The crystallinity values of the fibers are calculated by the intensity of light absorption at different wavelengths. The Sansevieria fibers have the highest crystallinity values which can be observed from Figure 6. This may be due to the relative fineness of the fiber and the chemical groups present in it. In case of the milkweed fibers, the crystallinity values are comparable with the other seed fibers like cotton. From Figure 6a it can be inferred that in case of Agave Americana, the crystalline regions are scattered across the different lattices.

Table 3. Assignment of FT-IR peaks and their relative sources

Wave Number (cm^{-1})	Vibration	Source
1242.62	C-O Aryl Group	Lignin
1420.93	C=C Stretching in Aromatic Groups	Lignin, Hemi cellulose
1505.00	C=C Aromatic symmetrical stretching	Lignin
1727.80	C=O unconjugated	Semi cellulose
3278.74	O-H Stretching	Polysaccharides
2885.00	C-H Symmetrical Stretching	Wax
1335.00	C-O Aromatic Ring	Cellulose

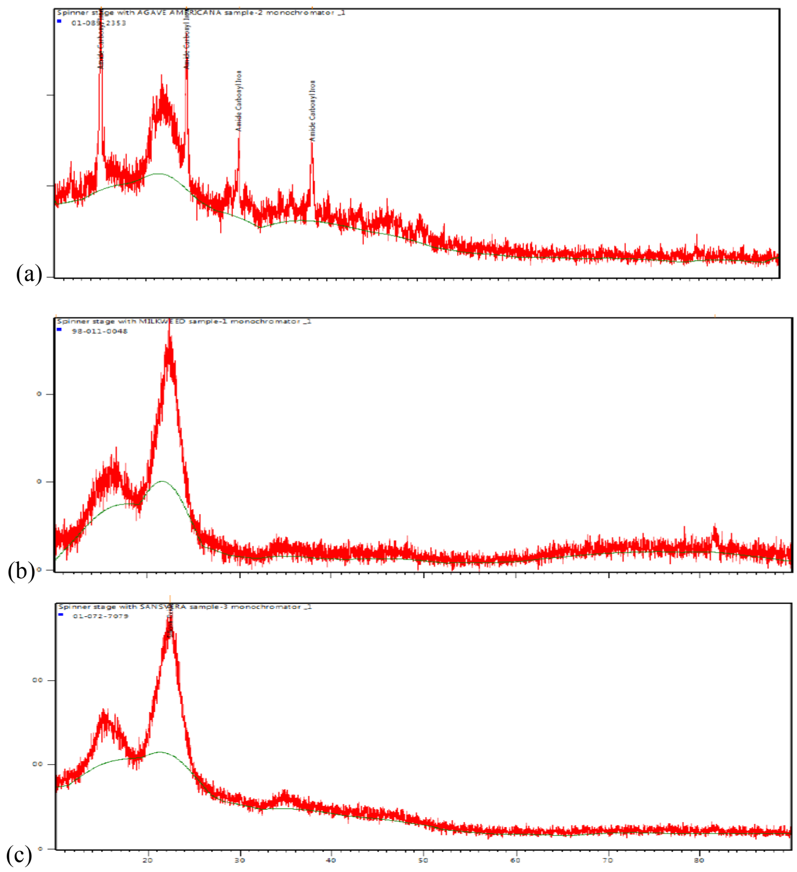


Figure 6. X-Ray diffraction graphs (a) Agave Americana (b) Milkweed (c) Sansevieria

Table 4. Crystallinity of different fibers

Fibers	Crystallinity Index	Crystallinity Ratio (%)
Agave Americana	54.0	68.5
Milkweed	41.3	63.0
Sansevieria	64.8	73.9

From Figure 7, the initial weight loss due to evaporation of water in sample was around 10.5%, 10% and 9% respectively for milkweed, Sansevieria and Agave Americana. From Table 4, it can be observed that the degradation for the Agave Americana starts first at 230 °C which may be due to the reason of low moisture regain values compared to the other fibers. For milkweed and sansvieria, the degradation starts at 260 °C and 280 °C respectively after the evaporation of 10% moisture.

The presence of polysaccharides, wax and cellulose in the milkweed fibers results in very low residual content, while for the two bast fibers a residual content of around 25% is found due to the presence of lignins and proteins. In case of the milkweed fibers, the loss in weight is sudden and continuous where as in case of the bast fibers, degradation starts at a point and then it remains stable over a short period and then continues to degrade.

From Figure 8, it can be observed that the Agave Americana fibers show exothermic peak around 80 °C, milkweed shows around 60 °C and Sansevieria fibers, around 364 °C. In case of Sansevieria, at around 90 °C, there is a small occurrence of a peak, but there is a higher rate of reaction at 364 °C, indicating the presence of a substance that is volatile at higher temperatures.

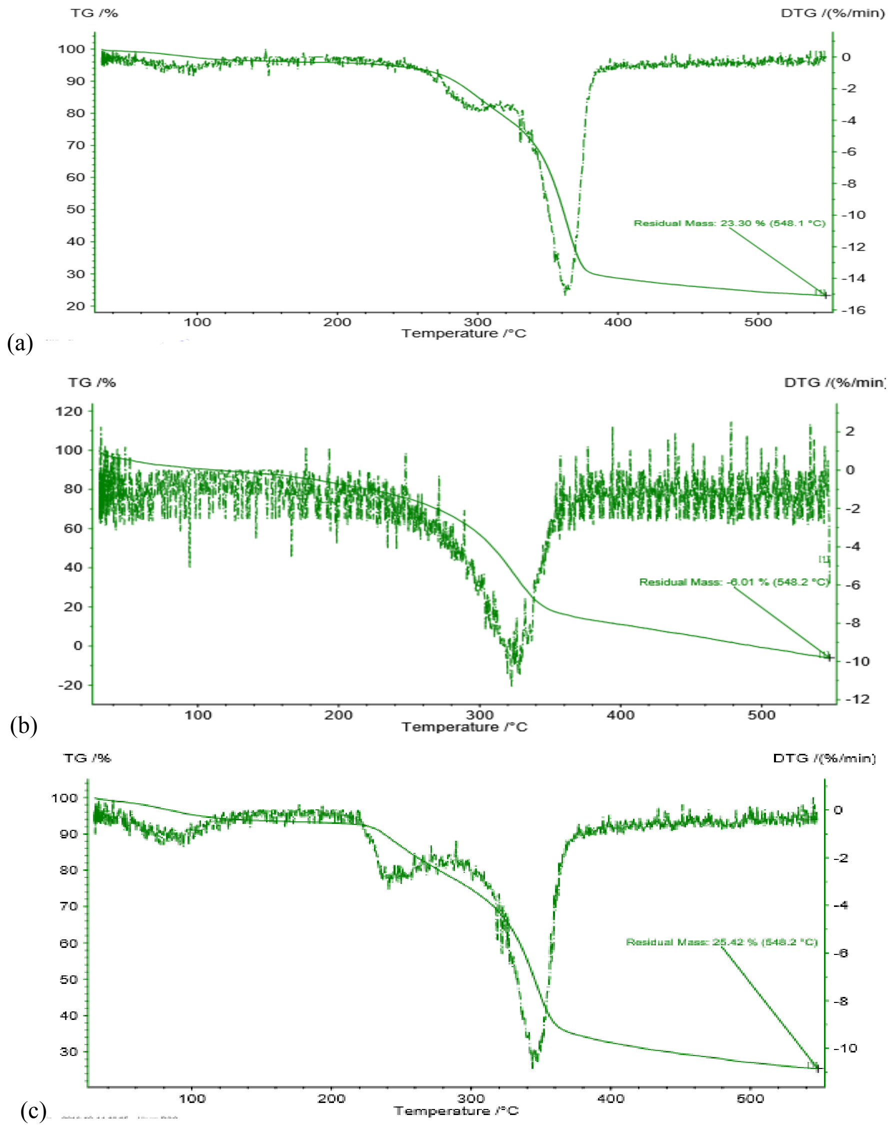


Figure 7. TGA and DTG graphs of fibers (a) Agave (b) Milkweed (c) Sansevieria

Table 5. Thermal degradation values of fibers

Fibers	Degradation Temperature, T_D (°C)	Maximum Degradation Temperature, T_{Dmax} (°C)
Agave Americana	230	360
Milkweed	260	340
Sansevieria	280	375

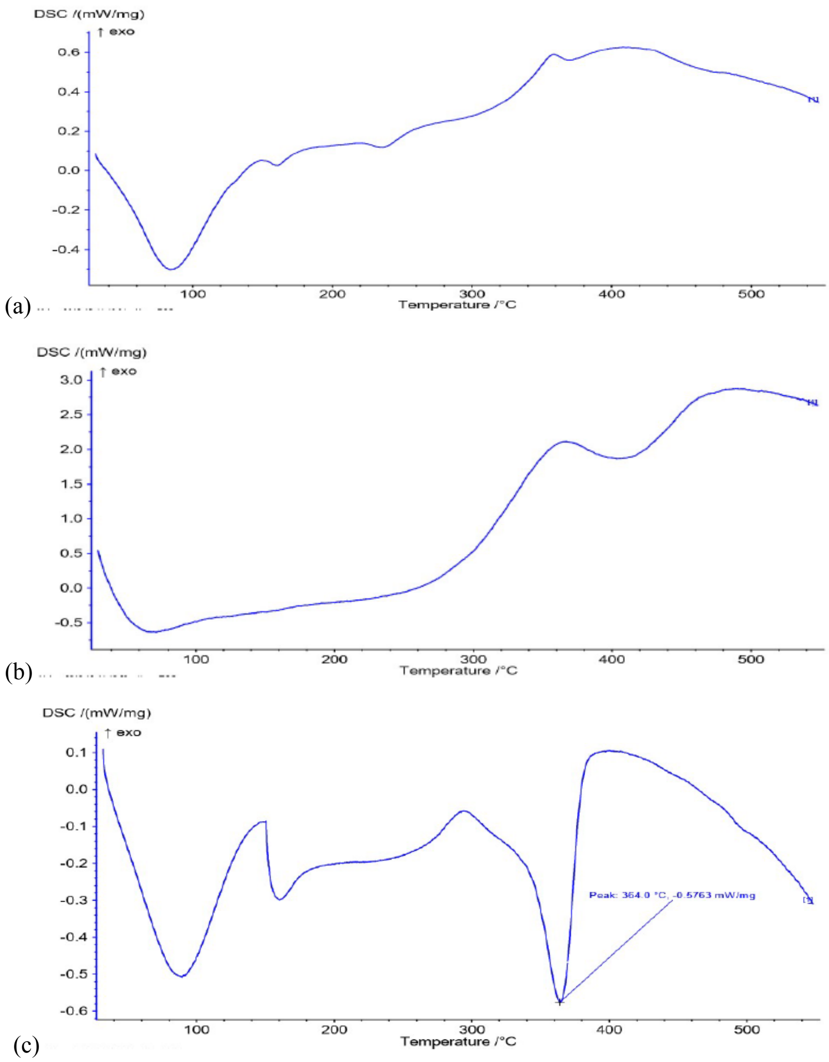


Figure 8. DSC curves of (a) Agave (b) Milkweed (c) Sansevieria

4. CONCLUSION

In this paper natural fibers namely – milkweed fiber, agave Americana fiber and sansevieria roxburghiana fibers have been studied experimentally to determine the physical, chemical, morphological and thermal properties by FTIR, SEM, PLM, X-Ray Diffraction, DSC and TGA analysis methods. Sansevieria fiber was observed to have the highest tenacity of 53.58 g/tex and maximum degradation temperature of 375 ^oC. Milkweed fibers were found to have the lowest density of 0.9 g/cc and possessed hollow structure with smooth surface as observed from SEM and PLM techniques. Agave Americana fibers were found to have a crystallinity index of 54% and the highest was recorded for sansevieria fibers having 64.8%. Bast fibers possessed functional groups like lignin, proteins and hemicellulose whereas milkweed fibers possessed polysaccharides as a major constituent. Agave fibers were found to have greater residual mass of 25.42% at 548.2 ^oC as observed from TGA graphs.

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