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Characterization of Cissus Populnea Bark Powder-Epoxy Hybrid Matrix for Composites Production

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ABSTRACT: Environmental concerns have motivated researchers to replace synthetic matrices with natural matrices in the production of polymer composites. However, natural matrices demonstrate weak mechanical or thermal properties which limit their different applications. Development of hybrid matrix will improve the thermo-mechanical properties of the natural based matrix. In this work, characterization of hybrid matrix from cissus populnea bark powder and epoxy resin for composite production and effect of addition of hardener to cissus populnea bark powder on thermo-mechanical properties were studied. Cissus populnea was pulverized to powder using grinding machine and sieved into size of 75µm. Samples were produced by mixing Cissus Populnea bark powder with epoxy and hardener as catalyst in various ratios. The blends were subjected to physical, mechanical, FTIR, and thermal analysis. The Results of hybrid matrix of Cissus populnea bark-powder and epoxy resin showed that Cissus Populnea/Epoxy (80:20) had density of 1.57 g/cm^3, water absorption was 17% in 288 hours. The modulus of elasticity (MOE) was 3363.82 MPa. The modulus of rupture (MOR) of 25.54 MPa. The result of Cissus Populnea/Epoxy (70:30) hybrid matrix composite had density of 1.25 g/cm^3, water absorption was 14% in 288 hours. The modulus of elasticity (MOE) of 5885.06 MPa. The modulus of rupture (MOR) of 49.66 MPa. Result of Fourier transforms infrared spectroscopy (FTIR) of hybrid matrix indicate hydrogen bond in the matrix, C-H stretching and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids. The TGA result show that, hybrid matrix was stable up to 200 °C with a decomposition temperature of 510°C corresponding to melting point of the resin. Results of hybrid matrix of Cissus populnea bark-powder and hardener for the various formulations gave density values in the range of 1.01 g/cm^3 to 1.16 g/cm^3. In terms of water absorption, the Cissus Populnea/Hardener matrix was unstable when immersed in water and dissolved within 2 hours. The Hybrid Matrix of Cissus populnea Bark-Powder and Hardener gave low values of MOR and MOE with values ranging from 0.62 – 0.91 MPa and 0.21 – 0.79 MPa respectively.These results implied that Hybrid Matrix of Cissus populnea Bark-Powder and Hardener is not suitable for composite production. However, Hybrid Matrix of Cissus populnea Bark-Powder and epoxy was suitable for composite production. Conclusively, Cissus Populnea/Epoxy hybrid resin in the ratio of 70:30 can be deployed as a hybrid resin for composite production.

Key words: Characterization, Hybrid Matrix, Cissus populnea, Composite and FTIR

I. INTRODUCTION

The demands for industrial advanced materials with enhanced and environmentally friendly material properties is increasing day by day. Due to environmental concern to reduce damage and the uncertainties about petroleum supplies, there is quest by researchers for combination of environmentally friendly resin with epoxy and the likes to improve certain properties of matrix for composite production; as well as minimized overdependence on petroleum products. Hybrid-matrix composites are materials that are made by combining two or more different types of matrices or resins [1]. According to Ramakrishnan et al. [2] polymer matrices are formed when resins which constitute thick, gooey compounds are exposed to heat to harden by a process called curing at room temperature. Morelli et al. [3] were of the opinion that, the goal of bringing two types of matrices on a composite is to keep the advantages of both matrices and relief some disadvantages generally. The concept of hybridization provides flexibility to the design engineer to tailor the material properties according to particular requirements, as the behaviour of the hybrid composites appeared to be, in general, a weighted sum of the contributions of the individual components in which there is a balance between their advantages and disadvantages**.**

The advantages of hybridizing or blending of resins to form a single matrix falls into two categories; blending may improveresin or product performanceby producing materials which may have a full set of the desired properties at lowest cost. extending the engineering resins performance by incorporation of less expensive polymers, improvement of specific properties such as brittleness which has to do with toughening brittle polymers, thus eliminating the need to use low molecular weight additives and Modulus and Dimensional Stability that is blending with more rigid and more heat-resistant resin [4].

Several researchers have carried out studies on the hybridization of two more resins to form matrices for production of bio-composites for various applications; with intent of reducing the harmful of toxic resins and to reduce over dependence on petroleum-based resins [5]. Combination of cissus polpuneal bark powder resin with epoxy will enhance mechanical properties of the hybrid matrix and at the same lowering cost of production as cissus polpuneal bark powder resin is a natural matrix that is obtained by renewable sources, cheap and in abundance, also acts as a filler.

In this work, hybrid matrix from cissus populnea bark powder and epoxy resin has been produced by hand lamination lay-up technique followed by characterization of the hybrid matrix by conducting physical, mechanical, thermal analysis as well as FTIR spectroscopy on the samples. These properties have been evaluated to determine the material's suitability for deployment in engineering applications.

II. MATERIALS AND METHODS

The materials used for this study include; Cissus Populnea bark-powder, Epoxy resin (885) part A, Hardener (995) part B, Distilled water, Mould release agent (Laminating Leather), Hand gloves.
The following experimental procedure were followed in the research.

MATERIALCOLLECTION ANDPREPARATION

Stem barks of Cissus populneawere procured from Makurdi. Cissus populnea were pulverized to powder using grinding machine and sieved into size of 75µm. The Cissus populnea bark powdered material was then stored in air-tight containers for characterization and usage as a binder or resin. Epoxy and hardener were procured from Wuse Market in Abuja.

PRODUCTION OF HYBRID MATRIX FROM CISSUSPOPULNEA BARK POWDER AND EPOXY RESIN

Production of hybrid matrix from CissusPopulnea bark powder and epoxy resin was carried out by hand lay-up method. A wooden mould of size 17cm × 13cm × 0.8cm = 176.8 cm^3 was used for producing the samples. The mould was covered with a lining (lamination leather) as mould release agent. Two samples were initially produced by blending CissusPopulnea bark powder with epoxy resin (with hardener) as shown in Table 1. This was achieved by adding Epoxy resin (epoxy part A and the hardener part B mixed in the ratio of 2:1) to Cissus Populnea bark powder and stirred for 15 minutes to enable effective reaction and was poured into the mould. In addition, three samples were produced by blendingCissusPopulnea bark powder with hardener as shown in Table 2. The CissusPopulnea bark powder became saturated with epoxy resin and a roller was used to ensure good compaction and freedom from porosity for all the five samples produced. The moulds were closed and kept under pressure at room temperature for 24 hours before the samples were removed as shown in Plate 1. The samples were cut for various tests.

Table 1: Production of Hybrid Matrix from CissusPopulnea Bark Powder and Epoxy Resin with Hardener

Sample No.	CissusPopulnea Bark Powder (%)	Epoxy Resin/Hardner (%)
1	80	20
2	70	30

Table 2: Production of Hybrid Matrix CissusPopulnea Bark Powder and Hardener

Sample No.	CissusPopulnea Bark Powder (%)	Hardener (%)
1	80	20
2	70	30
3	60	40

(a) **(b)**

Plate 1: (a) Photograph of Hybrid Matrix from CissusPopulnea Bark Powder and Epoxy Resin (b) Photograph of Hybrid Matrix from Cissus Populnea Bark Powder and Hardener

EVALUATION OF HYBRID MATRIX OF CISSUS POPULNEA BARK-POWDER AND EPOXY RESINS

PHYSICAL TESTS

Density Test; The density of the hybrid matrix was determined according to Archimedes principle at room temperature. The volume of each sample was obtained by immersing the sample in a graduated beaker containing a known level of distilled water. The difference between the final and initial level of water was used to calculate the volume of water displaced. This was taken as the volume of sample while the mass of each sample was obtained using a digital weighing balance. The equation 1, was used to evaluate the density of samples.

$$\rho = \frac{m}{v}(g/cm^3) \qquad (1)$$

Water Absorption Test: The effect of water absorption on the hybrid matrix was investigated in accordance with ASTM D570. The specimen of size 20 × 20 × 5 mm^3 was weighed to an accuracy of 0.1mg using digital weighing balance. Water absorption test was conducted by immersing the composite specimens in distilled water in plastic containers at room temperature for a duration of 288 hours. Once in 24 hours, a specimen was taken out from the water and all surface water was removed with a clean dry cloth and the specimen was reweighed to the nearest 0.1 mg. The specimen was weighed regularly from 24 hours to 288 hours exposure, at intervals of 24 hours. The water absorption was calculated using equation 2.

Percentage Water Absorption

$$= \frac{\textbf{Wet weight} - \textbf{Dry weight}}{\textbf{Dry weight}} \times \frac{\textbf{100}}{\textbf{1}} \quad (2)$$

MECHANICAL TEST

Flexural Test

The flexural test was performed on the hybrid matrix using 100 kN capacity universal materials testing machine This was done as per ASTM D-790 using the 3-point bending fixture, utilizing centre loading on a simple supported beam. The dimension of the sample was 100 mm × 30 mm × 8 mm^3, the gauge length of sample was 80 mm. The sample was placed on two supports and was loaded by means of a loading nose midway between the supports. The maximum axial

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fibre stresses occurred on a line under the loading nose. Three samples were tested and the average values of the modulus of rupture and modulus of elasticity were calculated using equation 3 and 4 respectively.

Modulus of Rupture

$$MOR = \frac{3pl}{2bt^2}\ (MPa) \qquad (3)$$

Modulus of Elasticity

$$(MOE) = \frac{pl^3}{4bt^3Y}\ (MPa) \qquad (4)$$

Where, p is max. load applied on test specimen (N)
l is gauge length (mm)
b is the width of specimen (mm)
t is thickness of specimen (mm)
Y is the deflection.

2.3.3 Spectroscopy analysis

Fourier Transform Infra-red (FTIR) Spectroscopy

The FTIR spectroscopy was carried out on the hybrid matrix in accordance to ASTM 168-20. FTIR machine equipped with a focal plane array (FPA) detector at the mid-Infrared beamline was employed. A homogenous sample free of contaminants was loaded in a sample holder of the machine under vacuum pressure. The vacuum pressure causes the removal of moisture. IR spectrum bands were obtained when laser of infrared was projected onto the sample with a sample scan of 64, a resolution of 16 and spectral range of 4000-650 cm^{-1}. The IR radiations impacts on the atomic vibrations of the molecules in the sample, resulting in the specific absorption and/or transmission of energy. The molecular components and structures contained in the specimen were displayed as absorption bands.

THERMAL ANALYSIS

THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermo-gravimetric analysis (TGA) was done on the hybrid matrix. The machine consists of an analytical balance supporting a platinum crucible for the specimen, situated in an electric furnace. The sample was heated with a heating rate of 10ºC/min over the temperature range from 30.00°C to 910.00°C. The weight loss of the sample was measured in a nitrogen atmosphere with temperature and time as a function continuously in a confined space to decompose the composite. The machine was connected to a computer monitor and the weight loss curve was displayed on the screen.

III. RESULTS AND DISCUSSION

RESULTS OF THE DENSITY HYBRID MATRIX OF CISSUSPOPULNEA BARK-POWDER AND EPOXY RESIN

The results of the densities of hybrid matrix of Cissus populnea bark-powder and epoxy resin as well as Hybrid Matrix of Cissus populnea bark-powder and hardener for the various hybrid matrix are presented in Tables 3 and 4. The densities are in the range of 1.01 g/cm^3 to 1.57 g/cm^3. The density result illustrates that CissusPopulnea/Epoxy (80:20) has maximum density value of 1.57 g/cm^3. It is also observed that as the amount of Cissus Populnea powder decreased in the formulations corresponding density decreased.

Table 3: Density of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin

Sample	%	Density (g/cm^3)
Cissus Populnea/Epoxy	80:20	1.57
Cissus Populnea/Epoxy	70:30	1.25

Table 4:Density of Hybrid Matrix of Cissus populnea Bark-Powder – Epoxy/Hardener

Sample	%	Density (g/cm^3)
Cissus Populnea/Hardener	90:10	1.16
Cissus Populnea/Hardener	80:20	1.12
Cissus Populnea/Hardener	70:30	1.01

WATER ABSORPTION IN HYBRID MATRIX OFCISSUS POPULNEA BARK-POWDER AND EPOXY RESIN

The percentage of water absorption in the hybrid matrix of Cissus populnea Bark-Powder and EpoxyResin was calculated by weight difference between the samples immersed in water and the dry samples. The variation of water absorption with the formulations at room temperature is shown in the Table 5. Water absorption was 17% in Cissus

Populnea/Epoxy (80:20) formulation in 288 hrs. Suggesting it can be deployed for outdoor apllications. In Cissus Populnea/Epoxy (70:30) water absorption was 14% in 288 hrs While the Cissus Populnea/Hardener matrix was unstable when immersed in water and dissolved within 2 hours in water. Indicating that it ca not be deployed for outdoor application.

Table 5: Water Absorption in Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy Resin

Sample	%/ Hours	24	48	72	96	120	144	168	192	216	240	264	288
Cissus Populnea/Epoxy	80:20	5	5	10	10	12	12	15	15	17	17	17	17
Cissus Populnea/Epoxy	70:30	3	3	9	9	9	11	11	11	14	14	14	14
Cissus Populnea/Hardener	90:10	Unstable and dissolved in water within 24 hours											
Cissus Populnea/Hardener	80:20												
Cissus Populnea/Hardener	70:30												

FLEXURAL PROPERTIES OF HYBRID MATRIX OF CISSUS POPULNEA BARK-POWDER AND EPOXY RESIN AS WELL ASHYBRID MATRIX OF CISSUS POPULNEA BARK-POWDER AND HARDENER

The values of modulus of elasticity (MOE) and modulus of rupture (MOR) of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resins as well as Hybrid Matrix of Cissus populnea Bark-Powder and Hardener specimens are shown in Table 6. MOE is in the range of 0.62 - 5885.06 MPa. Cissus Populnea/Epoxy (80:20) had MOE value of 3363.82 MPa while the highest value of 5885.06 MPa was obtained for Cissus Populnea/Epoxy (70:30). Hybrid Matrix of Cissus populnea Bark-Powder and Hardener gave low values of MOE in all the three samples due to weak Cissus populnea Bark-Powder – hardener adhesion with MOE increasing as the percentage of hardener increased with a maximum MOE of 0.91 MPa.

The MOR is in the range of 0.21 – 49.66 MPa with Cissus Populnea/Epoxy (80:20) having MOR of 25.54 MPa, Cissus Populnea/Epoxy (70:30) having maximum MOR of 49.66 MPa. The MOR values in the Hybrid Matrix of Cissus populnea Bark-Powder and Hardener were low with Cissus Populnea/Hardener (90:10) giving the lowest value of 0.21 MPa with the MOR increasing as the hardener increased with a maximum of 0.79 MPa in Cissus populnea Bark-Powder and Hardener (70:30). The implication is that, Cissus Populnea/Epoxy have sufficient strength that can be deployed for structural applications while Cissus populnea Bark-Powder and Hardener is not suitable for structural applications.

Table 10: Flexural Properties of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin as well as Hybrid Matrix of Cissus populnea Bark-Powder and Hardener

Sample	%	Deflection (mm)	Load (N)	MOE (MPa)	MOR (MPa)
Cissus Populnea/Epoxy	80:20	1.8	125	3363.82	25.54
Cissus Populnea/Epoxy	70:30	2.0	243	5885.06	49.66
Cissus Populnea/Hardener	90:10	9.3	2	0.62	0.21
Cissus Populnea/Hardener	80:20	7.8	3	0.87	0.55
Cissus Populnea/Hardener	70:30	8.1	5	0.91	0.79

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FOURIER TRANSFORM INFRA-RED SPECTROSCOPY OF HYBRIBMATRIX OF CISSUS POPULNEA BARK POWDER AND EPOXY RESIN

The result of Fourier transforms infrared spectroscopy (FTIR) spectral of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin is presented in Figure 1**.** The infrared spectrum of the hybrid matrix showed peaks numbering fifteen (15) as seen in the Figure. This suggest that the analyzed Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resins is a complex molecule.

The peaks showing wavenumber of 3354.60405 cm^{-1} falls between 2500-4000 cm^{-1}; there was a broad absorption band, informing there was hydrogen bond in the material. No peaks were observed between 3000 and 3200 cm^{-1}, indicating that cissus populnea bark powder resin has no aromatic structure. There were three peaks detected at 2922.23286, 2855.14078 and 2318.40413 cm^{-1}. The peaks observed at 2922.23286 cm^{-1} and 2855.14078 cm^{-1} for Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resins is attributed to the C-H stretching (vibration of the bond between carbon and hydrogen in a molecule) and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids. According to Ipilakyaaet al. (2024) the presence of carboxylic acids in natural fibres influence biodegradability, hydrophilicity and adhesion.

Regarding the double bond region (1500-2000 cm^{-1}), there are peaks at 1744.39411, 1610.20995 and 1505.84449 cm^{-1}. This according to Tile et al. (2024) informs some carbonyl double bond, which can be from ketones, aldehydes, esters, or carboxyl. Other peaks noted are; 1461.11643, 1356.75097, 1297.11357, 1237.47616, 1103.29200, 1028.74524, 827.46900 and 730.55822 and cm^{-1} corresponding to functional groups of alkanes, aromatic amine, amine, alkene, epoxy and oxirane rings, and 1,2,3-trisubstituted respectively. This implies that cissus populnea bark powder resin can be used as a resin in composite production.

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) OF HYBRID MATRIX OF CISSUS POPULNEA BARK-POWDER AND EPOXY RESINS

Thermogravimetric analysis (TGA) and Differential thermal analysis (DTA) curves of the Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin is presented in Figures 2 and 3 respectively. The result show that, Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin was stable upto 200 ^{o}C. It can be observed that the sample started losing weight at about 200 ^{o}C. There was a drop-in weight from 100 to 90 % as temperature increased from 200 – 500 ^{o}C. This can be ascribed to the release of absorbed moisture in the hybrid matrix. The decomposition temperature of the sample ranges from 500-650^{o}C where a sudden drop in the weight of the sample is observed with a loss of 40 % of the total weight of sample. This could be related to the degradation of substances such as hemicelluloses and celluloses in the Cissus populnea Bark-Powder. The decomposition temperature is usually taken at the point of sudden drop in weight of sample on the TGA curve which is at 510^{o}C (Jagadeeswaran and Chandrasekaran, 2023). As illustrated in Figure 2, the thermal stability decreased with increase in temperature and time. This result is in agreement with the works of Isabela et al. (2012) on Thermal Characterization of Jute Fibres by TGA and DSC.

The DTA curve of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin (Figure 3) observed to peaked with negative peaks signalling an endothermic process. For an endothermic process, more heat flows into the sample and the peak at 510°C corresponds to melting point of the resin

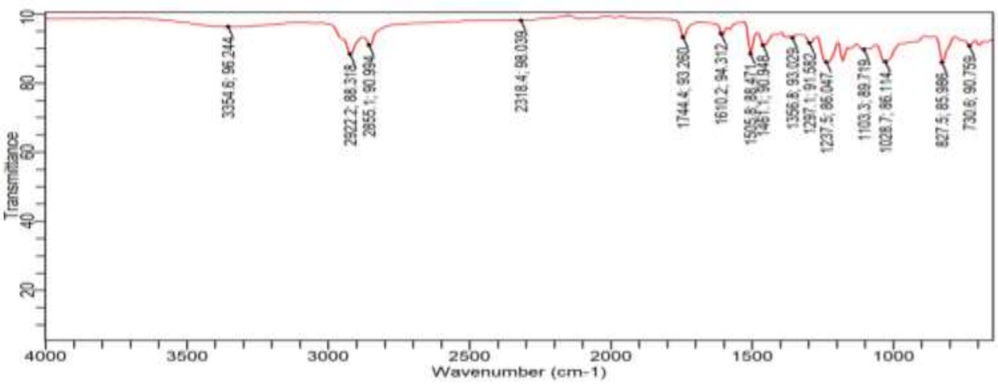

Figure 1: FTIR Spectral of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy Resin

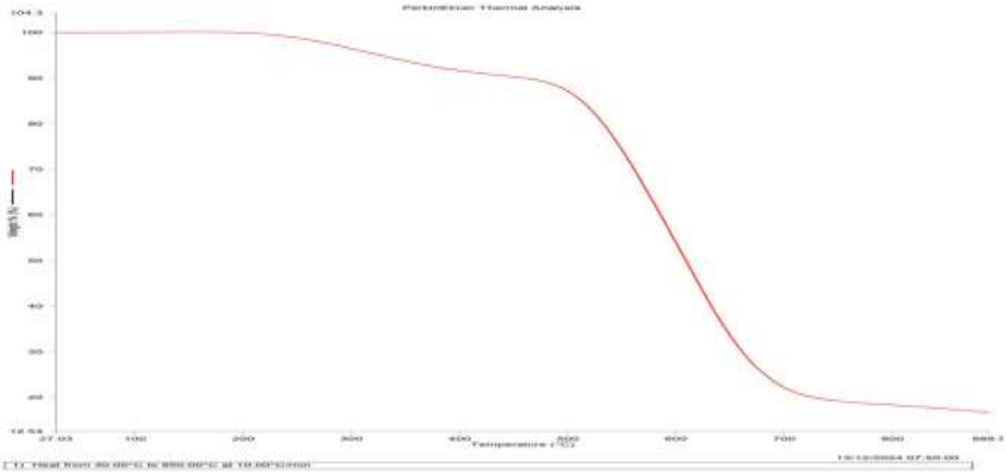

Figure 2: TGA of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy Resin

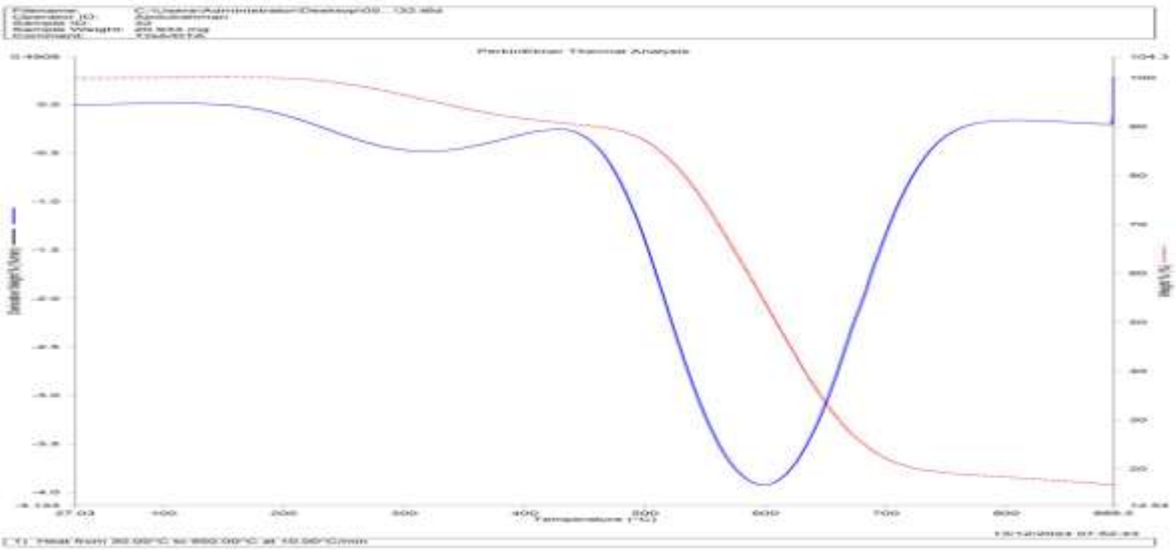

Figure 3: DTA of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy Resin

IV. CONCLUSION

The following conclusions can be drawn from the results of the characterization of hybrid matrix from cissus populnea bark powder and epoxy resin for composite production.The physical properties of Cissus Populnea/Epoxy (70:30) had density of 1.25 g/cm^3. Water absorption was 14% in 288 hours. Addition of 30 % epoxy resin to Cissus Populnea bark-powder resulted to significant drop in water absorption. Cissus Populnea/Epoxy (70:30) gave the highest MOE of 5885.06 MPa and maximum MOR of 49.66 MPa. This indicate that, the addition of 30 % epoxy resin to Cissus populnea Bark-Powder has been confirmed to enhance flexural properties. The Cissus Populnea/Epoxy (70:30) gave optimum properties and was subjected to Fourier transforms infrared spectroscopy (FTIR) and Thermogravimetric analysis (TGA). Result of Fourier transforms infrared spectroscopy (FTIR) of Hybrid Matrix of Cissus populnea Bark-Powder and Epoxy resin showed peaks in single bond region indicating a broad absorption band, informing there was hydrogen bond in the material. Also, some peaks which are attributed to the C-H stretching (vibration of the bond between carbon and hydrogen in a molecule) and O-H stretching bond structure that contains a functional group of alkanes (cellulose and lignin) and carboxylic acids were observed. In addition, peaks corresponding to functional groups of alkanes, aromatic amine, amine, alkene, epoxy and oxirane rings, and 1,2,3-trisubstituted respectively were confirmed. The TGA result show that, hybrid matrix of Cissus populnea Bark-Powder and Epoxy resin was stable up to 200 °C.

The results of hybrid matrix of Cissus populnea bark-powder and hardener for the various compositions gave density values in the range of 1.01 g/cm^3 to 1.16 g/cm^3. In terms of water absorption, the matrix was unstable when immersed in water and dissolved within 2 hours which indicates, it cannot be employed for outdoor applications. The Hybrid Matrix gave low values of MOR and MOE. This is a further indication that, the addition of hardener to Cissus Populnea matrix lacks the mechanical strength to be employed for structural applications.

These results suggest that, hybrid matrix of Cissus populnea Bark-Powder and Epoxy resin has good physical and thermo-mechanical, properties. Based on the results obtained from this study, it is recommended that, Cissus Populnea/Epoxy (70:30) be deployed as a hybrid resin for composite production

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