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Production and Analysis of Bioresin From Mango (*Mangifera Indica*) Kernel Oil

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ABSTRACT: *The work presents report on production and analysis of bioresin from epoxidized mango kernel oil (EMKO). The bioresin (acrylated epoxidized mango kernel oil) or AEMKO was produced from epoxidized mango kernel oil via acrylation chemical reaction route. The FTIR spectrum analysis of epoxidized mango kernel oil (EMKO) and acrylated epoxidized mango kernel oil (AEMKO) produced gave the degree of acrylation (DOA) as 46%. The Viscosity of AEMKO (resin) was determined at room temperature (25 °C) to be 387cP while the density at 25^{o}C was 1.2 g/cm$^{3.}$ The glass transition temperature (Tg) of the bioresin was determined to be 95^{o}C. Production cost analysis of the bioresin was done and found to be ₦8, 804.35 per litre. The high cost was due to high costs of the chemicals, labour and overhead charges involved at my local level. At commercial level, those components of the costs would definitely reduce to the level compatible with synthetic (polyester) resin (N2, 500 per litre) currently sold by some markers in Nigeria. However, the overall results of the work demonstrated that bioresin can be successfully synthesized from mango kernel oil with properties compatible with ASTM standards. The commercial production of the bioresin will go a long way in mitigating some of the challenges associated with total use of fossil fuel currently use for production of bulk of synthetic resins for composite manufacturing activities.*

KEYWORDS: Acrylation, Bioresin, Composite material, Epoxidized oil, Mango kernel oil, Synthetic resin.

I. Introduction

Many of the modern technologies nowadays require light materials with unusual combinations of properties that cannot or easily be met by the conventional materials such as metals and their alloys, one of these materials that is relatively new in many countries is Fibre reinforced polymer (FRP) material commonly called composite material. It is one of the emerging materials widely used in the fields of Air, Land and Sea transportation among others for production of light materials that have high strength to weight ratio, [1].

Composite material generally consists of two major constituents, reinforcement (fibre) and matrix (commonly called resin). Resin, which is the central focus of this work, is defined by [2] as a viscous and transparent liquid either from organic or inorganic source that will transform (cured and hardened) into solid when treated with suitable catalyst, accelerator with or without heat. Those from inorganic sources (such as crude oil or chemicals) are commonly called synthetic resins while those from organic sources (such as plant or animal) are called bioresin or renewable resins.

Resin (polymer matrix) that is widely used for composite material production has several functions; it is a binder that holds the reinforcement in place, it transfers external loads to the reinforcement and protect them from adverse environmental effects, [1]. According to [3], the matrix redistributes the load to surrounding fibers when an individual fiber fractures and laterally supports the fibers to prevent buckling in compression among others.

The major source of synthetic resins is crude oil and the global demand for crude oil is increasing year by year despite the discovery of more oil fields in some countries including Nigeria. The finite nature of the major source (crude oil) of synthetic resins and increasing global use of composite materials for wide range of engineering applications call for concerted efforts to source for alternative material that is renewable and sustainable for composite resins, [1]

Despite the numerous engineering applications of composite materials, it was captured in the reports of [4] and [5] respectively, that the bulk of the resins and many other polymers matrices used for composite activities across the globe are produced from non renewable Petrochemicals substances like Xylene, ethylene, propylene; benzene and vinyl chloride by polymerization processes. In addition, some health related illnesses such as respiratory tract infections and some skin infections have been reported to be associated with production process of these synthetic resins.

In addition to the above challenges of synthetic resins, it was reported by [6] that the bulk of synthetic resins used for wide range of engineering activities in Nigeria are imported materials. Despite the adverse effects of the importations on our economy, the materials are not readily available for use and are only found in few specialize places in some of the commercial cities.

Concerted efforts have been intensified by researchers across the globe to mitigate the challenges posse by continuous and linear use of synthetic resins (polymer matrices) for composite activities. This lead to two major processes by which vegetable or plant oil can be modified or functionalized to produce bioresin. These are epoxidation and acrylation chemical reactions, [6].

Epoxidation represents one of the most important fundamental reactions involving the triglyceride and specifically involves functionalizing the double bonds (C=C) between the carbon atoms. Epoxidation of plant or vegetable oils leads to the material that is known as epoxidized vegetable oil (EVO). Epoxidized oil serves as raw material for synthesis of many industrial chemicals including bioresins, polymers, lubricants, plasticizers, etc. [7].

According to [8], epoxidized vegetable oil (EVO) polymers by themselves may be suitable as a polymeric matrix for composites if blended with synthetic epoxy resin. Further modifications can be made on the chemical structure to modify functionalities. One such modification involves acrylation of the epoxides formed during epoxidation reaction.

Acrylation is a chemical reaction where epoxidized vegetable oil structure is modified for further functionalities. In this process, epoxy functional triglyceride of the oil is reacted with acrylic acid to incorporate acrylate chemical groups onto the triglyceride thereby attaching vinyl functionalities to its structure. Acrylation reaction helps to convert the unreacted epoxides in the oil to impart further functionalization to the triglyceride thereby improving the polymer performance. After this modification, a polymer in the form of an acrylated epoxidized vegetable oil (AEVO) is obtained. The conversion of EVO to AEVO allows, lower curing temperatures, shorter curing times and improved material properties, [9] and [10].

The aim of this work is to produce and analyze renewable resin from epoxidized mango kernel oil (EMKO) for composite application. Available and sustainable resin from mango kernel oil will serve as alternative resin for composite manufacturing activities. This will go a long way in cushioning the adverse effects of using fossil fuel as raw material for synthetic resins productions and consequently mitigates some of the challenges associated with petrochemical resins.

II. Materials and Methods

2.1 Materials

Table 1: List of materials, chemical compounds, devices and apparatus.

S/No.	Raw material and Chemical compounds	Devices/Apparatus
1	Epoxidized mango kernel oil	Buck infrared spectrophotometer (model 530)
2	Acrylic acid	Digital weighing machine (LA 164. B. Brain)
3	Hydroquinone	Measuring cylinder
4	Toluene	Griffin temperature and adjustable oven
5	Distilled water	Conical flask
6	Anhydrous sodium sulfate	U-tube Viscometer
7		Pipette
8		Burette
9		prolab efflux viscometer,
10		stop watch
11		2-neckround bottom flask
12		Hot plate equipped with magnetic stirrer
13		Magnetic bit
14		Tripod stand
15		Thermometer
16		Graduated dropping funnel
17		Circulating water bath
18		Separating funnel

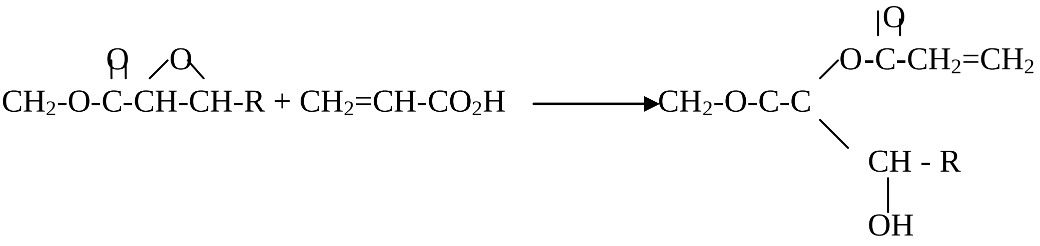
2.2 Methods

2.2.1 Production of acrylated epoxidized mango kernel oil (AEMKO)

Acrylated epoxidized mango kernel oil (AEMKO) was prepared according to ASTM standard reported by [11]. In this study, epoxidized mango kernel oil (EMKO) was reacted with Acrylic acid in a 250 mL round-bottom flask equipped with a magnetic stirrer and a reflux condenser. Hydroquinone which was used as a free radical inhibitor was added to the contents in the flask. The molar ratio of EMKO: acrylic acid was 1:10. The flask was heated to 90°C until the light yellow colour changed to milky colour which indicated complete reaction. The mixture was cooled to room temperature and diluted with toluene before purifying by washing with distilled water. The final product was dehydrated with anhydrous sodium sulfate and the solvent was evaporated using an evaporator. The above procedure was repeated to achieve the quantity of the bioresin targeted.

Plate 1 showed the sample of epoxidized mango kernel oil (EMKO) that was reacted with acrylic acid to produce the acrylated epoxidized oil. Plate 2 showed the setup for acrylation reaction in the laboratory while plate 3 showed the acrylated oil (bioresin) produced.

Epoxidized oil + Acrylic acid Acrylated epoxidized oil ……… (Eq. 1)



$$CH_2\text{-}O\text{-}\overset{O}{\overset{\|}{C}}\text{-}\overset{\diagup O \diagdown}{CH\text{-}CH}\text{-}R + CH_2{=}CH\text{-}CO_2H \longrightarrow CH_2\text{-}O\text{-}C\text{-}C\begin{cases} O\text{-}\overset{O}{\overset{\|}{C}}\text{-}CH_2{=}CH_2 \\ \underset{\underset{OH}{|}}{CH}\text{-}R \end{cases}$$

Plate 1: Epoxidized mango kernel oil (EMKO)

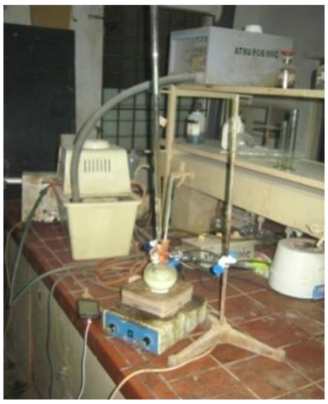


Plate 2. Setup for Acrylation reaction

Plate 3. Bioresin produced from EMKO

2.2.2 Production of Fourier transforms infrared (FTIR) Spectrum

In this study Buck infrared spectrophotometer (model 530) was used to produce the FTIR spectra of the epoxidized and acrylated epoxidized oils respectively. The 4000–500 cm-^{1} infrared spectral regions of the oils are shown in Figures 2 and 3. From the recognized peaks, heights and wave number on the spectra of each oil sample, the functional groups corresponding to each wave number were identified according to [12] and John [13] interpretation charts. The results are found in tables 2 and 3.

2.2.3 Determination of Viscosities of the bioresin

The Viscosity of the acrylated epoxidized oil and the acrylated epoxidized oil mixed with 33% by weight of styrene were determined at temperature of 25 °C according to ASTM method that involved the use of Brookfield viscometer apparatus as described by [14]. The result of the viscosity measurements are found in table 4.

2.2.4 Determination of the density of the bioresin

The density of the bioresin was determined at (27^{o}C) according to ASTM standard obtained from [15]. The result is found in table 4.

2.2.5 Determination of Glass transition temperature of the bioresin

The glass transition temperature of the fully cured bioresin produced was determined using ASTM method that involved the use of Differential Mechanical Thermal Analysis (DMTA) described by [ ]. The result is found in table 4.

2.2.6 Economic Analysis of bioresin from mango kernel oil

The economic analysis of the bioresin is centered on the costs implications of the various activities involved right from the Collection and processing of mango seeds for oil extraction up till the production of the bioresin via acrylation method.

i. Collection and processing of mango seeds for oil extraction

Mango seeds (house the kernels containing oil) are waste materials that are readily available, affordable and sustainable in Nigeria and many other countries. It virtually cost nothing to gather the seeds during the harvesting season after they are thrown away by the farmers, users and the consumers. Processing of the kernels for oil extraction virtually also cost nothing. The labour and over head costs associated with the collection of the seeds were estimated as ₦200.

ii. Extraction of oil from the seed kernels.

The solvent used for the oil extraction was N-hexane and it costs N18, 000 for 4 litres used to extract 3.2 litres of the oil. The labour and over head costs were estimated as ₦1, 000 and ₦500 respectively.

iii. Conversation of the raw oil to epoxidized oil.

The cost of conversion of 3.2 litres of the raw oil to epoxidized oil was estimated as ₦5, 000. This covers the materials (chemicals) estimated as ₦3, 500 and labour and over costs estimated as ₦1, 500.

iv. Conversation of epoxidized oil to Resin

The cost of conversion of 3.2 litres of the epoxidized oil to acrylated oil and later to resin was estimated as ₦14, 000. This covers the materials (chemicals) estimated as ₦12, 000 while the labour and over head costs were estimated as ₦2, 000.

Treatment of acrylated oil to resin involves addition of Styrene to the acrylated oil to reduce the viscosity to the level suitable for composite processing. The viscosity reduction leads to increase volume from 3.2 to 4.6 litres. The chemical, labour and over head costs were estimated as N1000, N500 and N300 respectively. The summary of total expenses incurred in the production of the bioresin is shown in table 5.

III. Results

The results of the Spectra of EMKO and AEMKO are found on figures 1 and 2 while tables 2 and 3 showed the results of the interpretations of the FTIR spectra of the figures. Table 4 showed the result of the viscosity measurements of AEMKO and treated AEMKO with 33% styrene, density and the glass transition temperature of the bioresin. Table 5 showed the summary of the determined properties of the resin and comparism with ASTM standards.

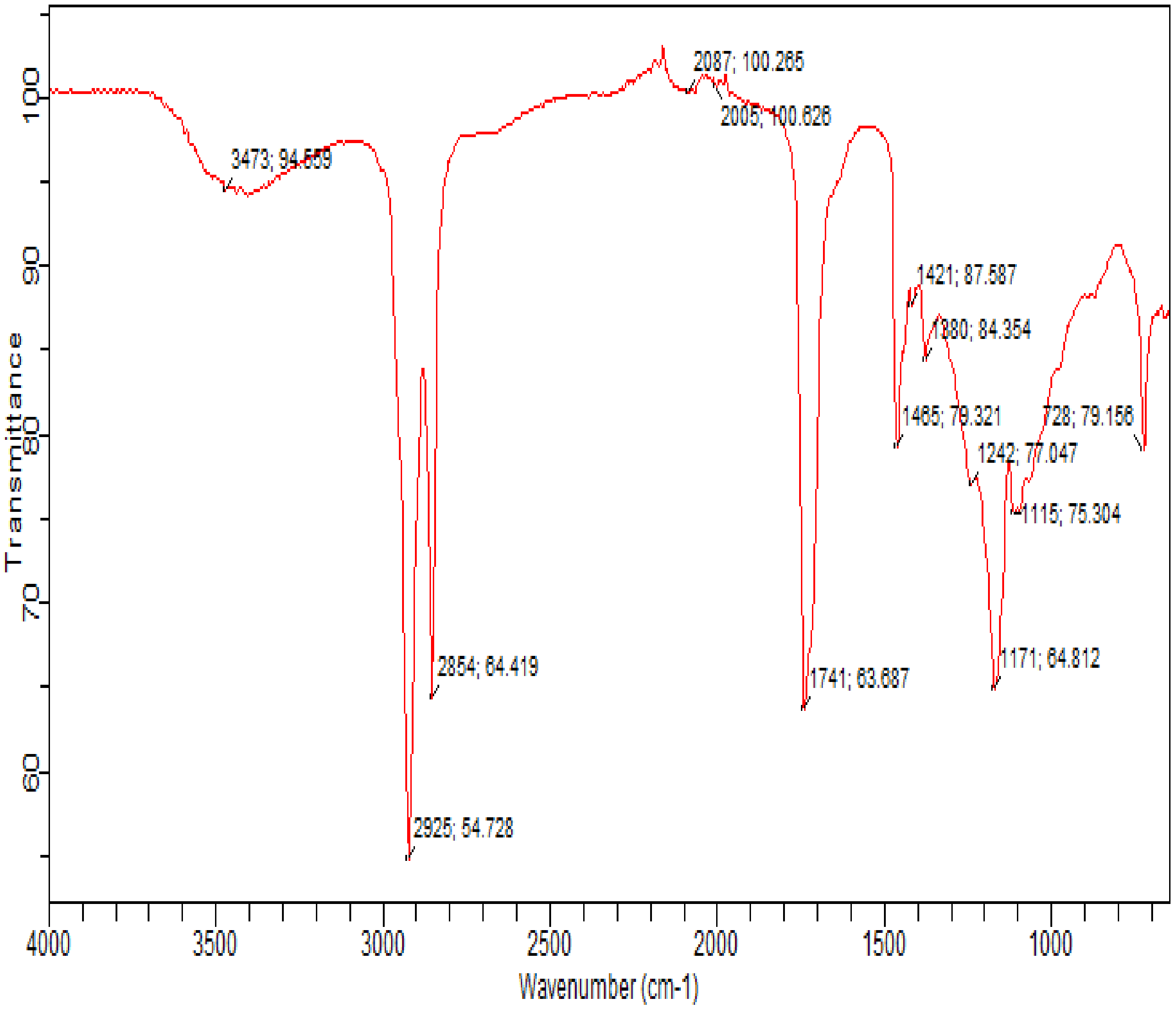


Figure 1. Spectrum of epoxidized mango kernel oil (EMKO)

Figure 2. Spectrum of acrylated epoxidized mango kernel oil (AMKO)

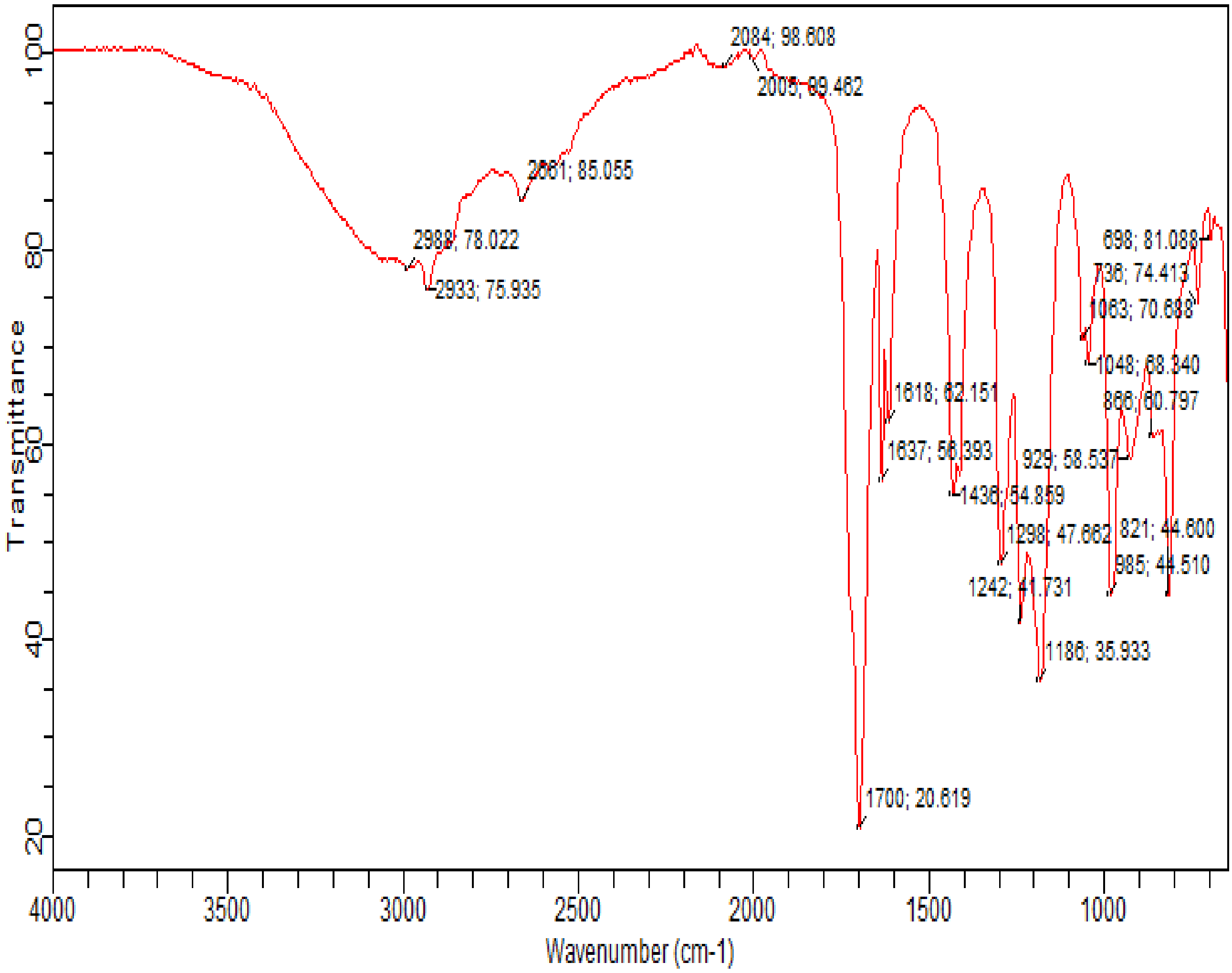


Table 2. Peak identification of FTIR spectra of Epoxidized mango kernel oil (EMKO) and interpretations

Frequency range (cm^{-1})	Wave number (cm^{-1})	Peak Height Percent transmittance (%)	Peak Assignment (Bond)
730 – 665	728	79.156	C = C
1124 – 1089	1115	75.304	C – O
1200 – 1020	1175	64.812	C - OH
1250 – 1050	1242	77.047	C – O - C
1600 = 1400	1421	87.587	C = C
1600 – 1400	1465	79.321	C = C
1745 -1715	1741	63.687	C= O
2950 - 2840	2854	64.419	C - H
2950 - 2840	2925	54.720	C - H

Table 3. Peak identification of FTIR spectra of Acrylated mango kernel oil (AEMKO) and interpretations

Frequency range (cm^{-1})	Wave number (cm^{-1})	Peak Height Percent transmittance (%)	Peak Assignment (Bond)
730 – 665	698	81.088	C = C
840 – 790	736	74.413	C = C
840 – 790	821	44.600	C = C
840 - 915	866	60.797	C = C
915 – 985	929	58.537	C = C
915 – 985	985	44.510	C = C
1050 – 1048	1048	68.340	CO – O = CO
1085 – 1050	1063	70.688	C = O
1250 -1050	1242	41.731	C – O - C
1480 – 1440	1436	54.859	CH2
1680 – 1600	1618	62.151	C = C
1680 – 1600	1637	56.393	C – C
1700 – 1500	1700	20.619	C = O
2830 – 2695	2661	85.055	C – H
2950 - 2695	2933	75.935	C - H

Table 4: Summary of determined properties of the bioresin

S/NO.	Parameters	Determined value
1	Iodine value (IV) of renewable oil suitable for bioresin synthesis	60.7mg iodine/100g oil
2	Degree of Epoxidation (DOE)	69.4%
3	Degree of Acrylation (DOA)	46%
4	Viscosity	387Cp
5	Density	$1.2g/cm^3$
6	Glass transition temperature (Tg)	95^oC

Table 5: Summary of total expenses incurred in the production of bioresin from mango kernel oil.

S/No.	Criteria	Material cost (₦)	Labour cost (₦)	Over head cost (₦)
1	Collection and processing of mango seeds for oil extraction	--------	……	200
2	Extraction of oil from the seed kernels.	18,000	1,000	500
3	Conversation of the raw oil to epoxidized oil	3,500	1,000	500
4	Conversation of the epoxidized oil to Resin	12,000	1,500	500
5	Treatment of acrylated epoxidized oil with 33% styrene so as to reduce viscosity to acceptable level	1,000	500	300
	Total	34, 500	4,000	2, 000

The cost of one (1) litre of the resin = $\frac{Material\ \ cost + Labour\ \ cost + Over\ head\ cost}{4.6}$

$$= \frac{₦40{,}500}{4.6} = ₦8,\ 804.35$$

The high cost of production is due to the high costs of chemicals and the labours involved. At commercial level, these components of costs would definitely reduce to the level compatible with synthetic (polyester) resin (N2, 500 per litre) currently sold by some markers in Nigeria.

IV. Discussion

Epoxidized mango kernel oil (EMKO) which serves as a raw material for bioresin production by acrylation method was previously produced with 69.4% DOE as explained by [6]. The acrylation reaction conducted on EMKO in this study is a further modification on the chemical structure where the epoxy functional triglyceride of the epoxidized oil was made to react with acrylic acid so as incorporates acrylate chemical groups onto the triglyceride to yield vinyl functionalities structure.

Although epoxidized vegetable oil (EVO) may be suitable as a polymeric matrix for composites applications if blended with synthetic epoxies, but acrylation of the epoxidized oil according to According to [7] and [8], gives better properties (such as lower curing temperatures, shorter curing times and improved properties) on the resin.

According to [16], vinylesters are processed the same way as unsaturated polyesters despite the difference in the chemical structure. The crosslinkng reaction of vinylester is identical to the free-radical crosslinkng of unsaturated polyesters and the same crosslinkng agents, initiators, acceleretors and inhibitors used for polyesters are also used for vinylesters. This is underscores the reason why acrylation reaction is carried out on the epoxidized oil.

The FTIR spectrum of the epoxidized and the acrylated oils respectively were determined using Buck infrared spectrophotometer (model 530). The FTIR spectrum were used to monitor the epoxidation and acrylation reactions by analyzing the peaks pertinent to C=C consumption and epoxide and acrylate formation. Figure 1 presents the spectra of EMKO while figure 2 shows the spectra of AEMKO. On each of the spectrum, significant areas at specific wave numbers, peak heights and the possible functional groups were documented as shown in tables 2 and 3. The shape of the plot (wave number against transmittance) of the EMKO is different from that of the AEMKO which indicated that some transformations took place during the reaction of the EMKO with acrylic acid.

Considering the spectrum of the EMKO (i.e. figure 1), there are many peaks where the carbon double bond functional groups that were not completely reacted during the epoxidation reaction resurfaced (i.e. wave number 728,1421, 1465 cm^{-1}). However, peak of interest occurs at 1242 cm^{-1} which reflects epoxidized functional group. The carbon double and the epoxidized functional groups were responsible for acrylation reaction yield.

Similarly Figure 2 presents the FTIR spectra associated with the acrylation reaction of the epoxidized mango kernel oil (EMKO). The spectrum shows different functional groups including the carbon double that could not react completely during the acrylation process. However, the peak of interest occurs at wave number1241 cm^{-1} and is associated with the transformation of epoxy group to acrylate group, [17] and [18]. Before the acrylation reaction, the peak height was 77.047% on the epoxidized oil spectrum and after the acrylation reaction, the peak reduced to 41.731%. This indicated 35.32% transformation and gave rise to 46% DOA on the basis of 100% peak height. Other peaks of interest occur at wave number 2005 and 2085 cm^{-1}. According to [19], these peaks and others associated with carbon double are attributed to the formation of acrylic functional groups in the acrylated epoxidized oil. Comparing the peak height of wave number 2005 cm^{-1} on epoxidized oil and acrylated oil spectra, only 30.8% transformation took place. This gave rise to 40% DOA. The low transformation figures may be unconnected with effective management of the acrylation chemical reactions and the low unsaturated nature of the mango kernel oil as indicated by the iodine value (60.7 mg iodine/100 g oil) determined and explained by [20].

The viscosity of resin generally has to be such that can easily be used for production without any stress. The viscosity of AEMKO without treatment with styrene was 1632 cP. This figure was too high for composite processing techniques. Going by report of [7], there is need to decrease such very high viscosity by adding about 33% by weight of styrene to the AEMKO in order to improve processability for manufacturing composite parts by traditional composite processing techniques. The 33% by weight of styrene added to the AEMKO decreased the viscosity to 387 cP. Apart from decrease in viscosity of AEMKO, the addition of styrene also increases the volume of the bioresin from 3.2 to 4.6 litres.

The density of resin is one of the physical properties that enable one to know the heaviness (dense nature) of the resin. It gives idea about the mass of the resin with respect to the volume (space) occupied. The density of the final product determined was 1.2g/cm^3.

The glass transition temperature (Tg) is a thermodynamic property that gives idea about the working temperature of the fully cured resin. The (Tg) was determined to be 95^{o}C. This means that at 95^{o}C, the polymer begins to turns from a hard and brittle material to a ductile material.

Production cost analysis of the bioresin was conducted and gave one litre of it to be ₦8, 804.35. The high cost of production is due to the high costs of chemicals and the labours involved at my local level in Nigeria.

V. Conclusion

The following conclusions were made based on the outcome of the work:

1. The acrylation chemical reaction was conducted using acrylic acid. The degree of acrylation (DOA) estimated from FTIR spectrum of the epoxidized oil and the bioresin analysis was 46%. Although the value is low, it however meets the ASTM standard of 33 – 94%. The low value of the DOA is a reflection of the low level of unsaturation of MKO as dictated by its iodine value (60.7g iodine/100g of oil) and of course effective management and control of acrylation process.

2. The analysis of the infrared (IR) spectrum of the epoxidized oil and the bioresin also indicated that the oils contain other compounds and bonds apart from the C=C that form the bases of unsaturation level for the conversion of the raw oil into epoxidized oil and the epoxy functional groups that provides the plate form for conversion into acrylate groups of the bioresin. These functional groups contributed in the overall reaction in the epoxidation and acrylation processes respectively.

3. The dynamic viscosity (1632 cP) of the AEMKO produced was too high for composite production activities. This necessitated the addition of 33% by weight of styrene to the AEMKO thereby decreasing the viscosity to 387 cP which is within the ASTM standard range of viscosity (100 to 500 cP at 20^{o}C) of resin for composite processing. In addition to the decrease in viscosity, the 33% by weight of styrene mixed with AEMKO also increases the volume of the resin from 3.2 to 4.6 litres.

4. One of the physical properties of the resin that enable one to know the heaviness (dense nature) of the resin, density was determined to be 1.2g/cm^3 and falls within the ASTM standard range of 1.0 – 1.5g/cm^3. The glass transition temperature (Tg) which is a thermodynamic property that gives idea about the working temperature of the fully cured resin was also determined to be 95^{o}C. This value meets the ASTM standard range of 80^{o}C – 110^{o}C for bioresins and 80^{o}C – 280^{o}C for synthetic resins.

5. Economic analysis of the bioresin estimated one litre of the resin to be ₦8, 804.35.
The high cost of production is due to the high costs of chemicals and the labours involved. At commercial level, these components of costs would definitely reduce to the level compatible with synthetic (polyester) resin (N2, 500 per litre) currently sold by some markers.
However, when the bioresin produced is compared with those from soya bean or palm fruit oils or other eatable oils developed by some researchers, the bioresin from mango kernel oil has an added advantage in that the oil is not eatable and more so the source (mango seed kernel) is a waste material that is readily available, affordable and sustainable in Nigeria and many other countries.

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