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Hura crepitans L. Seed Oil-Based Medium Oil Alkyd Resin/Calcined Clay Reinforced Bio-Composites: Synthesis, Characterization and Molecular Weight Determination

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Abstract

Research on producing high-quality bio-based products from locally sourced, environmentally friendly materials for the surface coating industry has been revitalized by the Sustainable Development Goals and the concept of green chemistry, promoting both environmental and economic sustainability. This research aims to synthesize bio-composites of alkyd resin from locally sourced *Hura crepitans* L. seed oil (HCSO) and calcined clay (CC) as reinforcing agents for use as binders in surface coating products. Three samples of 50% oil-length HCSO solvent-borne alkyd resin bio-composites with different weight percentages of CC, HOA-1 (2 wt.% CC), HOA-2 (3 wt.% CC), and HOA-3 (5 wt.% CC), were prepared via an in-situ two-stage alcoholysis-polyesterification process. In contrast, the ordinary alkyd resin HOA-Z was prepared without CC to serve as a control. The effect of CC loading on the appearance, surface morphology, number-average molecular weight, thermal stability, and intercalation with the alkyd sample was investigated. The bio-based products were characterized using standard methods. The colour of the final products ranges from dark brown to light brown. The viscosity of the bio-composites decreases from 17430.0 to 737.5 mPa.s. A morphological study showed the homogeneous dispersion of the CC in the alkyd resin. The molecular weight decreases with an increase in wt.% of CC, while the flammability property increases with increased CC loading. The tensile strength increases with the addition of CC from 18 MPa (HOA-Z) to 44 MPa (HOA-3). Processed clay minerals can serve as a better reinforcement agent for the preparation of

polymer bio-composites.

Keywords

Composites, *In-Situ* Alcoholysis, Rast Method, Resin, Polyesterification, Monoglyceride

1. Introduction

The current trend in environmental and climate change the world over suggests that greener technologies and methodologies are the panacea for environmental sustainability, sustainable development, and a sustainable economy [1]. Also, in the pursuit of sustainable development and advancements in more sustainable sourcing of materials in all sectors go hand in hand with nationwide set goals of net-zero greenhouse gas emissions by 2050 [2] [3]. This has led to a paradigm shift in the source of raw materials from petroleum-based to bio-based feedstocks in the development of cost-effective manufacturing technology, thereby reducing the price-performance aspect of the material [4]. The bio-based raw materials are basically bio-renewable, eco-friendly, and agricultural resources such as vegetable oil (VO), starch, and protein. The use of these materials as monomers for polymer synthesis will not only enhance sustainable development but also maintain and sustain the integrity of the environment [5]. Agricultural products are sustainable and biodegradable materials that have many applications in the production of bio-based products. For example, proteins, starch, and sugar cane are used in bio-based aerogel preparation [6]; vegetable oils are used in preparation of binders for surface coatings [5] [7]-[11]; membranes for water desalination [12]; creams, cosmetics, and soaps [13] [14]; margarine and butter [15]; biodiesel and biolubricant [16]-[18]; grape pomace are used as additives in biopolymer formulations to obtain materials with antioxidant and antibacterial properties [19] etc.

However, in keeping with the Millennium Development Goals (MDGs) and Sustainable Development Goals (SDGs), this research was carefully designed to promote green chemistry by using renewable raw materials (*Hura crepitans* L. seed oil, glycerol, etc). Provides an eco-friendly alternative to petroleum-based alkyds. Enhances properties using abundant natural clay, reducing cost and environmental impact. As well as contributing to material sustainability and circular economy goals in polymer science and materials engineering.

A composite is a material with two or more distinct constituents or phases that have different physical or chemical properties and are constructed into a complex architecture at micro-, meso-, or macro-scale levels [20], resulting in either improved performance or a new engineering material that a single constituent material cannot provide. Nanocomposites exhibit markedly improved mechanical, thermal, optical, and physicochemical properties when compared to pure polymers or conventional composites [21]. Biopolymer composites can be prepared

via solution mixing, melt blending, and *in situ* polymerization [22].

One such local material that can be used as a reinforcing agent in composite materials is clay, which also plays a vital role in vegetable oil product processing by improving the colour of the products due to its efficient bleaching power. The use of acid-activated clay for bleaching palm oil has been reported [23]. Clay can be considered a natural, fine-grained mineral with plastic behaviour at appropriate water contents that will harden when dried or fired [24]. The presence of clay can improve the mechanical, thermal, barrier, and fire-retardant properties of the polymer [24]. The attention of scientists and researchers has been focused on bio-based nanocomposites because these materials have significant social and environmental advantages [25] [26]. The addition of modified clay to solvent-borne alkyd resins will result in a bio-composite material with unique properties such as high-temperature stability, oxidation resistance, erosion resistance, corrosion resistance, wear resistance, high hardness, improved drying time, resistance to chemical attack, and a low creep rate, as well as coupling features related to their light, electrical, magnetic, acoustic, and thermal properties.

Alkyd resins have been defined as the products of condensation between polyol, usually having an average functionality equal to or greater than 3, and a polybasic acid, usually dibasic acid or its anhydride, modified with monofunctional acids, most commonly C18 fatty acids or triglyceride oils [27]. Alkyd resins have also been defined as the products of the polycondensation reaction between polybasic acids and polyhydric alcohol modified with fatty acids or drying oil [28] [29]. It is a conventional binder that has been used in the production of paints, varnishes, and other coating products [30].

Vegetable oil-based alkyd resins have several advantages, including versatility in structure and properties [31], overall low cost [30], ease of application, and being renewable [5] [32]. Some of the challenges faced in vegetable oil alkyd resins include poor alkali resistance, inferior mechanical properties, low hardness and thermal stability, and a long curing time [31]. Some of these weaknesses can be improved by the incorporation of reinforcing materials such as clay to form the corresponding bio-composite. The solution mixing method is the simplest and most widely used method for the preparation of biopolymer composites. This method involved the dispersion of the bio-fillers in a common solvent in which polymers are also soluble.

However, as far as our knowledge is concerned, the synthesis of bio-composites based on medium-oil alkyd resin and calcined clay (CC) has not been reported before. Therefore, this research aimed to develop and characterize a sustainable, bio-based composite material using *Hura crepitans* L. seed oil as a renewable feedstock for alkyd resin synthesis, and reinforce it with calcined clay, evaluating its structural, physicochemical, and molecular properties for potential applications in industrial coating formulations. To obtain greener bio-composites with specific properties, the in-situ alcoholysis method was adopted by mixing the CC with the monoglyceride oil obtained after the alcoholysis stage, before polycondensation

with the polybasic acid. The effects of the CC on the molecular weight, surface morphology, thermal, mechanical, and flame retardancy properties of the resulting bio-composites were evaluated.

2. Experimental

2.1. Materials

Hura crepitans L. seed oil (HCSO) was extracted from the mature and dry seeds of the plants obtained from the following locations in Akwa Ibom State, Nigeria: Shelter Afrique in Uyo Local Government Area, Ete community in Ikot Abasi Local Government Area, and Onna and Obot Akara Local Government Areas. All the chemicals, such as phthalic anhydride, glycerol, calcium trioxocarbonate (IV), and xylene, were of analytical grade and were obtained from commercial sources and used without further purification for the preparation of alkyd resin bio-composites.

2.2. Methods

2.2.1. Preparation of CC

The acid-leached clay and CC samples were prepared from a locally sourced clay mineral following the procedure described in the literature [33].

2.2.2. Extraction of Oil from *Hura crepitans* L. Seeds

The vegetable oil was extracted from the milled sample of mature *Hura crepitans* L. seed through solvent extraction with petroleum ether (60˚C - 80˚C) following the method described in the literature [1] [30]. The golden yellow oil obtained was labeled as HCSO.

2.2.3. Preparation of *Hura crepitans* Seed Oil-Based Alkyd Resin and Alkyd Resin Bio-Composites

Medium-oil (50% oil length) *Hura crepitans* seed oil-based alkyd resin was prepared following the method described in the literature [30]. The alkyd sample was labeled HOA-Z, and it served as the control experiment.

Three-grade medium-oil (50% oil length) alkyd resin bio-composites were prepared using HCSO, CC, phthalic anhydride, and glycerol via an in-situ monoglyceride method. The composites were prepared by varying the concentration of calcined clay as follows: 2 wt.% CC (HOA-1), 3 wt.% (HOA-2), and 5 wt.% CC (HOA-3). The recipe used in the preparation of alkyds and bio-composites is given in **Table 1**. Calcium trioxocarbonate (IV) was used as the catalyst for the synthesis of the alkyd resin and its corresponding bio-composite samples.

The alkyd bio-composite samples were prepared in a 500-ml, three-neck round-bottom flask equipped with a motorized stirrer, a Dean, and a Stark trap attached to a water-cooled condenser and thermometer. Heating was achieved with a heating mantle. The temperature of the reaction was between 230˚C and 250˚C, and xylene was used as the azeotropic solvent [34].

The composites were prepared using a two-stage in-situ alcoholysis polyester-

fication method [34]-[36] with slight modifications. In preparing each of the alkyd composite samples, a calculated quantity of *Hura crepitans* L. seed oil was measured into the reaction flask and heated to a temperature between 220˚C and 250˚C with constant stirring. A calculated amount of technical-grade glycerol and 0.15% $CaCO_3$ (based on oil) as the catalyst was added while maintaining the temperature at 240˚C until the alcoholysis reaction was completed. The amount of various ingredients charged is represented in Table 1. This first stage of alkyd preparation is referred to as the alcoholysis stage. The completion of alcoholysis was monitored by a solubility test of one volume (two drops) of the sample with two volumes of methanol (four drops) on a clear glass slide until a clear (non-colloidal) liquid was observed [5] [37] [38]. The aliquots of the samples were withdrawn from the reaction flask every 10 minutes for the solubility test.

After the formation of monoglyceride oil, a calculated amount of calcined clay was added to the reaction mixture in the reactor while lowering the temperature to 180˚C. When the temperature of the reaction mixture was reduced to 180˚C, a measured quantity of polybasic acid (phthalic anhydride) (see the recipe in Table 1) was added, followed by the addition of xylene (10% by weight of the total ingredients charged) to remove the water of esterification by forming an azeotrope. The temperature was quickly raised to about 230˚C and maintained between 230˚C and 250˚C throughout the duration of the reaction.

Determining the acid values of the aliquots of the reaction mixture at 20-minute intervals monitored the extent of the reaction. 1.0 - 2.0 g aliquots of the reaction mixture were dissolved in a neutralized toluene/ethanol mixture in a 1:1 ratio. The resulting mixture was then titrated to a phenolphthalein endpoint with a standard 0.1 M KOH solution at room temperature. The polyesterification reaction was continued until an acid value below 10 mg KOH/g was obtained.

Table 1. Recipe for the formulation of medium oil alkyd resin bio-composites using *Hura crepitans* L. seed oil (HCSO) and calcined clay.

	Samples			
Ingredients	HOA-Z	HOA-1	HOA-2	HOA-3
HCSO (g)	100.00	100.00	100.00	100.00
CC (g)	-	1.98	5.83	9.52
Glycerol (g)	31.00	31.00	31.00	31.00
Phthalic anhydride (g)	71.00	71.00	71.00	71.00
$CaCO_3$ (g)	0.04	0.04	0.04	0.04

2.3. Instruments and Measurements

2.3.1. Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR of samples HOA-Z, HOA-1, HOA-2, and HOA-3 was recorded in the Nicolet Impact 410 FTIR spectrophotometer, USA, following the procedure described in the literature [33] [39]. Small amounts of the alkyd resin, HOA-Z, and alkyd bio-composites HOA-1, HOA-2, and HOA-3, respectively, were thoroughly

ground with potassium bromide, and tablets were prepared by compression under vacuum. The tablet was inserted into the lens of the spectrophotometer to record the FTIR spectrum of each sample.

2.3.2. X-Ray Diffractometry (XRD)

The degree of intercalation of CC in the bio-composite was determined based on the method described by Gogoi *et al.* [40].

2.3.3. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

The surface morphology of the alkyd resin and the alkyd/calcined clay bio-composites was determined based on the method described by Isaac *et al.* [33]. The elemental composition of the samples was determined via energy-dispersive spectroscopy.

2.3.4. Thermogravimetric Analysis (TGA)

To study the thermal degradation of the neat polymer and the corresponding composites, thermogravimetric analysis was carried out based on the method described by Gogoi *et al.* [31].

2.3.5. Physicochemical Properties Test

The physicochemical properties of biopolymers, such as acid value in mgKOH/g, colour, solid content (%), volatile matter (%), and specific gravity (at 40˚C), were determined following the method described in Isaac *et al.* [5]. The viscosity was determined using a digital viscometer model (NDJ-85) at ambient temperature.

2.3.6. Molecular Weight Determination of the Polymer Bio-Composites

The Rast method, as described by Isaac and Nsi [29], was used to determine the molecular weight of the polymer bio-composites.

2.3.7. Limiting Oxygen Index

The minimum concentrations of oxygen, expressed as a percentage, in a flowing mixture of oxygen and nitrogen that will initially support the combustion of a material, known as the limiting oxygen index [40], were measured according to ASTM D 2863. The specimens were cut into 100 mm × 6 mm × 3 mm (longitudinal, tangential, and radial) and placed vertically in the flammability tester. The volumes of nitrogen gas and oxygen gas were initially set at maximum and minimum levels. Thereafter, the volume of nitrogen gas was decreased, and that of oxygen gas was increased gradually. However, the total volume of nitrogen gas and oxygen gas mixture was kept fixed at 18 cc during the experiment. The ratio of N_2 and O_2 at which the sample continued to burn was recorded for at least 30 seconds. The limiting oxygen index was evaluated using Equation (1).

$$\text{Limiting oxygen index} = \frac{\text{volume of oxygen}}{\text{volume of oxygen} + \text{volume of nitrogen}} \times 100 \quad (1)$$

2.3.8. Mechanical Properties

The mechanical properties of the medium-oil alkyd sample HOA-Z and the

corresponding bio-composites (HOA-1, HOA-2, and HOA-3) films, such as tensile strength, Young's modulus, and tensile strain at break, were evaluated in triplicate using a universal testing machine (UTM, Swick, Z010) at ambient temperature following the procedure described in the literature [40].

3. Results and Discussion

3.1. Rheological and Physicochemical Properties of Medium-Oil Alkyd Resin Bio-Composites

Polymer bio-composites have become an indispensable raw material that has attracted great attention due to their superior properties, such as strength, toughness, and fire barrier, far from those of conventional polymers [24]. The presence of one nanoscale phase of reinforcing agent such as nanoclay leads to tremendous interfacial contacts between the polymer and clay, resulting in the improvement of the polymer bulk phase in terms of mechanical, thermal, barrier, durability, chemical stability, flame retardancy, scratch/wear resistance, biodegradability, as well as optical, magnetic, and electrical properties [41]-[44], as well as physicochemical properties such as colour.

Four grades of medium-oil alkyd resins were formulated with HCSO and a calcined clay mixture in a percentage of 0 wt.% CC (HOA-Z), 2 wt.% CC (HOA-1), 3 wt.% CC (HOA-2), and 5 wt.% CC (HOA-3), glycerol, and phthalic anhydride via the in-situ monoglyceride process. The reactions showing the chemistry of polymer synthesis and its bio-composite products are shown in **Figure 1(a)** and **Figure 1(b)**. The finished products are shown in **Figure 2**, while the physicochemical properties of the medium-oil alkyd and the bio-composite samples are given in **Table 2**.

The alcoholysis time was 30 minutes for each sample. The colour of the alkyds varies from dark brown to light brown. This may be due to oxygen penetration into the reaction mixture in the reactor as raw materials were charged into the reactor, and also as a consequence of the influence of the calcined clay in varying quantities in the various samples. It is clear from **Figure 2** that the reinforcing agent (calcined clay) has improved the aesthetic properties of the biopolymer products. The four alkyd samples have a relatively low volatile content (**Table 2**), which invariably correlates to a high percentage of solid content. A similar observation was made by Ekpa and Isaac [23], Isaac *et al.* [32], and Isaac *et al.* [5]. This implies that the products are of high standard and quality, therefore suitable as a surface coating binder. The acid value of the finished alkyds decreased relatively, therefore making them preferred binders for paint production. The viscosity of the samples increases significantly with the addition of 2 wt.% CC and decreases as the concentration of CC increases. The viscosity of Jatropha curcas oil-based alkyd/epoxy/graphene oxide (GO) bio-nanocomposites was observed to increase gradually with increasing GO concentration [31], as opposed to the observation in this research. The significant increase in viscosity of alkyd resin with the addition of a small amount of CC (2 wt.%) may be attributed to the possibility of the clay imparting a non-Newtonian behaviour to the alkyd biopolymer, making its

viscosity change in response to shear stress. On the other hand, the decrease in viscosity with an increase in concentration of CC obtained in this research may be due to the wettability property of clay and the method of preparation of the bio-composites (in-situ alcoholysis) in which CC was added before polymerization. The clay may have absorbed the water that was generated as a by-product of the polyesterification. It is reported that clay minerals exhibit different wettability due to differences in chemical composition, morphology, and physical structure. Kaolinite with small and dispersed crystals tends to be wetted by water, while kaolinite with large crystals may be wetted by oil [45]. Previous studies on the clay used in this research show that it is kaolinite clay with microporous morphology when calcined [33].

It is obvious that the incorporation of CC into the polymer matrix increases the fluidity of the composites. This will help reduce the quantity of solvent needed to dissolve it for paint production. The four samples have a very low specific gravity, making them a good binder for paint production as well.

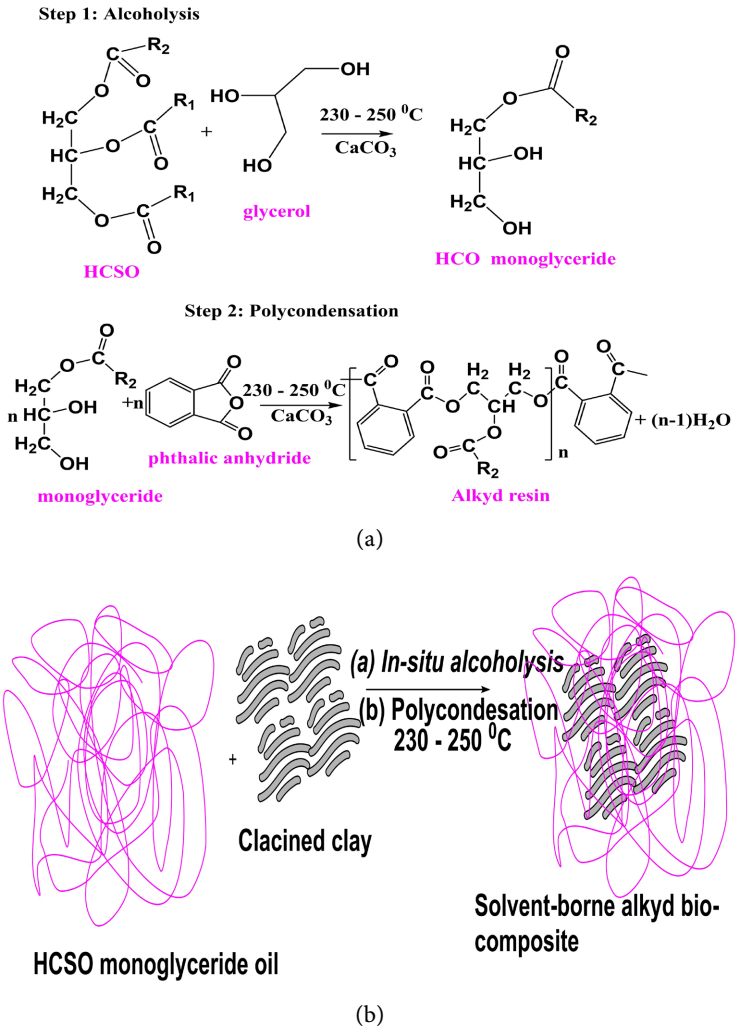


Figure 1. (a) The chemistry of alkyd resin biopolymer synthesis; (b) Solvent-borne alkyd micro-composite biopolymer synthesis.

Figure 2. *Hura crepitans* alkyd and the alkyd bio-composite samples.

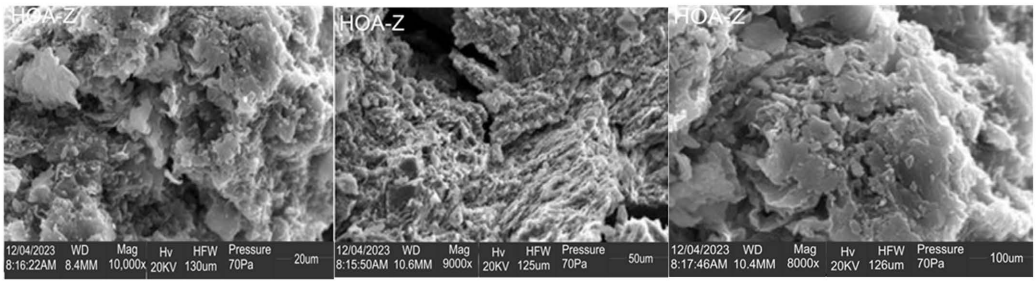
Table 2. Physiochemical properties of the alkyd sample and its bio-composites.

	Samples			
Properties	**HOA-Z**	**HOA-1**	**HOA-2**	**HOA-3**
Colour	Dark brown	Brown	Light brown	Light brown
Acid value (mgKOH/g)	13.17 ± 0.291	12.22 ± 0.092	6.03 ± 0.035	3.47 ± 0.028
Solid content (%)	77.76 ± 0.021	83.57 ± 0.035	86.06 ± 0.049	88.98 ± 0.021
Volatile matter (%)	22.45 ± 0.311	16.49 ± 0.035	13.99 ± 0.014	11.06 ± 0.021
Specific gravity (40˚C)	1.04 ± 0.014	1.14 ± 0.085	1.07 ± 0.028	1.06 ± 0.007
Viscosity (mpa. s) (32˚C)	824.0 ± 0.000	17430.0 ± 14.142	9200.0 ± 0.000	737.5 ± 2.121

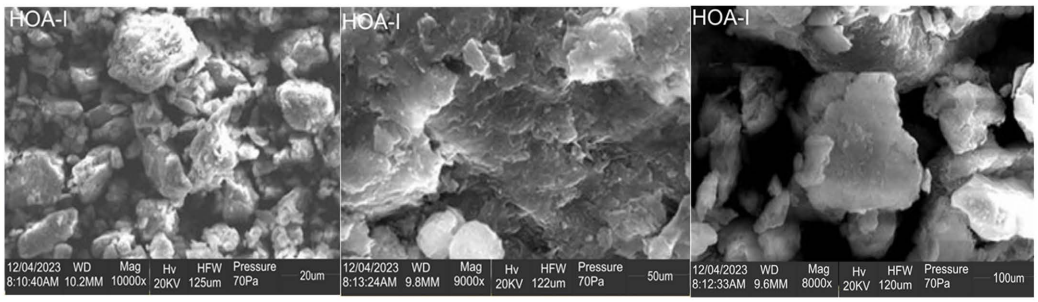
3.2. Surface Morphology of the Polymer and Polymer Bio-Composites

The microstructure of the medium-oil alkyd resin, HOA-Z, and those of the corresponding bio-composites, HOA-1, HOA-2, and HOA-3, have been studied using SEM, and the images are shown in **Figures 3(a)-(d)**. The SEM micrographs of the ordinary alkyd resin HOA-Z (**Figure 3(a)**) suggest a microporous, smooth, and uniform surface with some minor irregularities and granular features. The features show good film formation that is typically desirable in coating applications. Also, there are no significant cracks or voids, indicating that the alkyd biopolymer network is cohesive and well-developed. The granular appearance could suggest partial phase separation or the presence of additives, but not to a degree that could compromise film integrity. This observation could be due to the presence of $CaCO_3$, which was used as a catalyst in alkyd preparation. HOA-Z exhibits moderate homogeneity, with a texture that reflects successful dispersion of

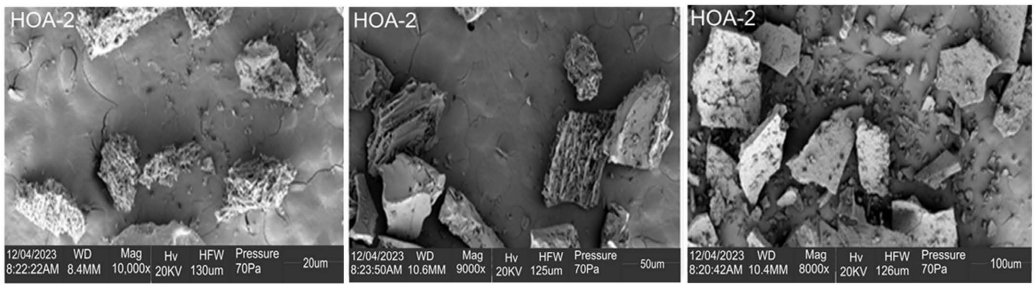
components within the polymer matrix. These properties could result in improved mechanical properties and barrier performance in biopolymer coatings. Other properties, such as smoothness, can contribute to gloss and aesthetic values, while the absence of voids helps with durability and resistance to environmental factors. SEM micrographs of the bio-composites HOA-1 and HOA-2 (**Figure 3(b)**, **Figure** 3(c)) reveal heterogeneous to rough and heterogeneous surfaces, respectively, with distinct aggregates and clusters of CC, indicating partial dispersion of the CC within the alkyd matrix. The calcined clay particles appear embedded in the polymer matrix as sheets of silica, suggesting moderate compatibility between the CC and biopolymer matrix. The irregularities and textural variations introduced into the polymer matrix by the addition of CC are typical when an inorganic reinforcing agent is dispersed in a polymer matrix. This phenomenon increased as the CC increased from 1.98 g (HOA-1) to 5.83 g (HOA-2) (**Table 1**). This morphology may result in improved thermal properties compared to the ordinary alkyd resin HOA-Z due to the presence of the inorganic reinforcing material (calcined clay). The micropores observed in the SEM images of HOA-1 and HOA-2 may be due to air entrapment, solvent evaporation, and imperfect filler matrix bonding. The SEM morphology of HOA-3 (**Figure 3(d)**) shows that the CC is well dispersed in the polymer matrix with compact and continuous morphology. This may suggest strong interfacial adhesion between the inorganic material (CC) and the polymer matrix (alkyd resin). This morphology may enhance barrier performance and mechanical properties. The SEM surface indicates good compatibility, as there is low porosity. The successful dispersion of CC during the preparation of the bio-composites is seen in the presence of fine and even distribution of CC particles. Phase separation of the micro-composite observed in the three composite samples may be because CC is not completely in nano-form, but in micro-form. Polymer composites containing clay have been divided into phase-separated microcomposites, intercalated nanocomposites, and exfoliated nanocomposites [46], as shown in **Figure 4**. The addition of the CC has increased the surface roughness of the alkyd resin, as shown in the SEM results of the bio-composite samples (**Figures 3(b)-(d)**). This could lead to enhanced thermal stability, mechanical interlocking, reduced water and other substance permeability, and adhesion properties, as well as reducing its susceptibility to thermal degradation and increasing the resistance to heat.



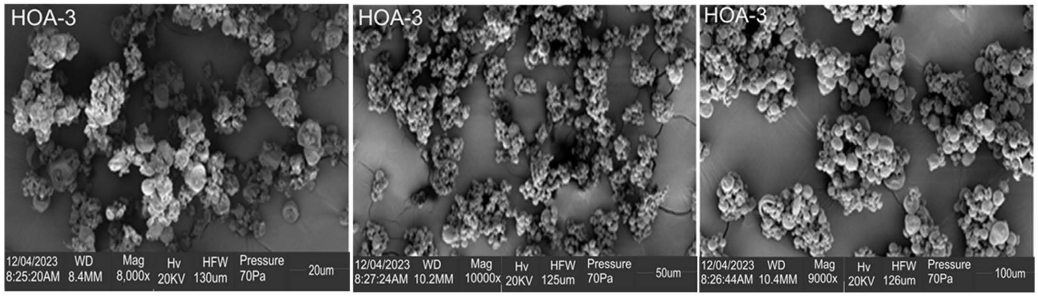
(a) SEM result for alkyd sample HOA-Z



(b) SEM results for bio-composite sample HOA-1



(c) SEM results for bio-composite sample HOA-2



(d) SEM results for bio-composite sample HOA-3

Figure 3. SEM micrographs of HOA-Z, HOA-1, HOA-2, and HOA-3.

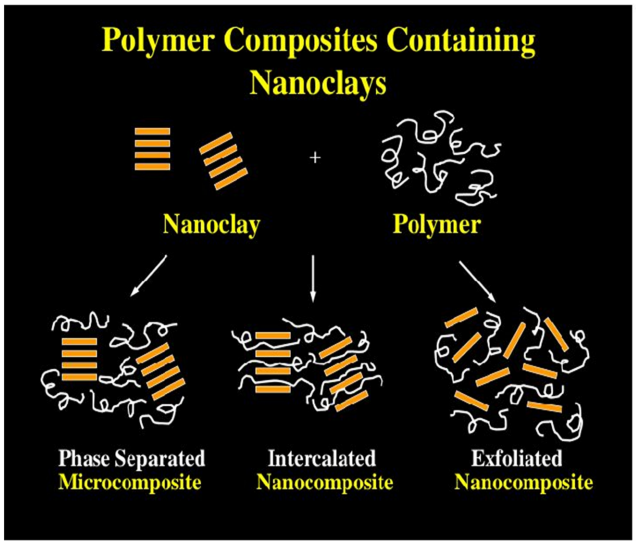


Figure 4. Types of polymer composites containing nanoclay.

3.3. EDS Results of Alkyd Sample and the Bio-Composite Samples

The EDS results presented in Figure 5 show that the products contain environmentally benign elements, with carbon and oxygen having the highest concentrations in all the samples, followed by silicon. Other elements present were magnesium, calcium, sodium, iron, copper, and manganese.

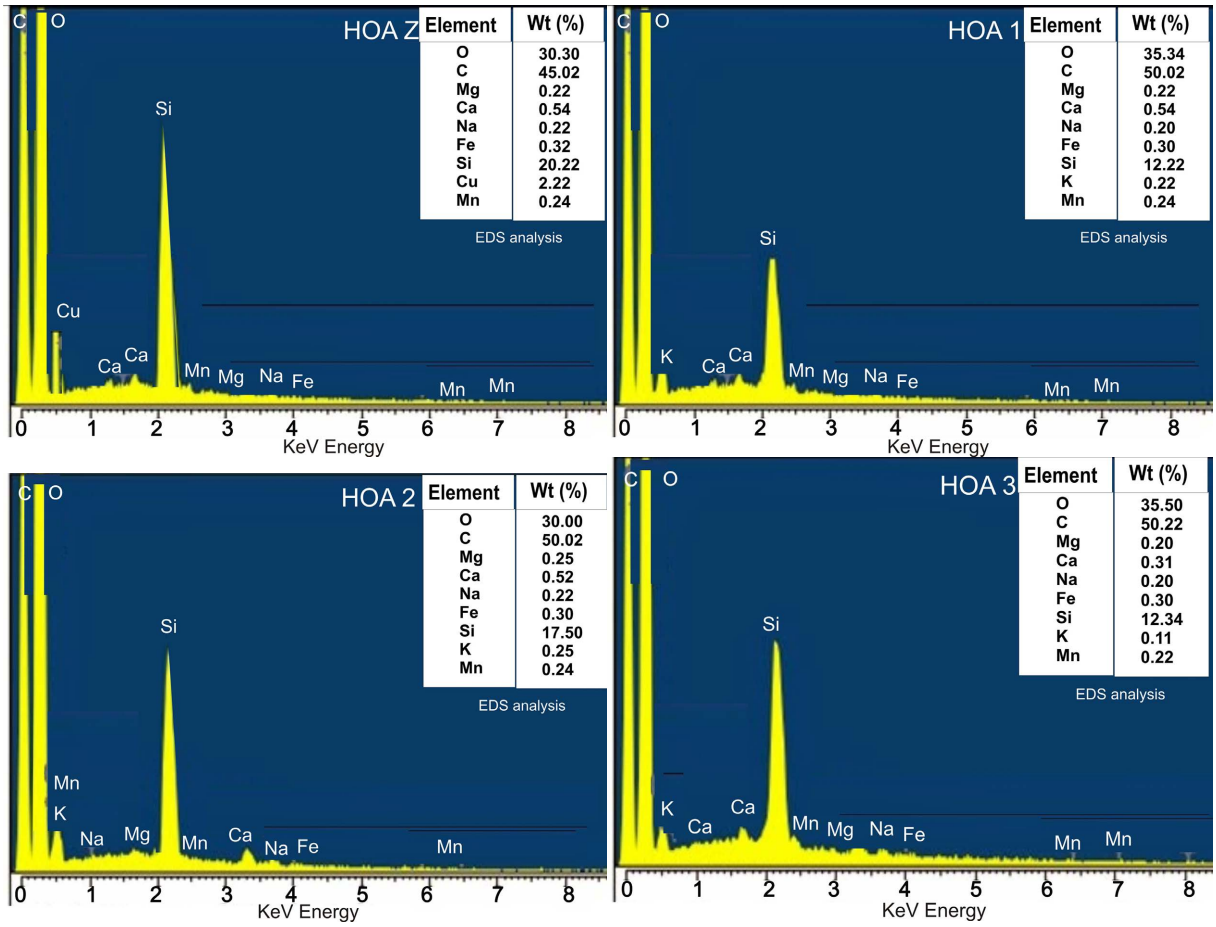


Figure 5. EDS results for alkyd polymer and the bio-composite samples.

3.4. XRD Analysis of the Alkyd Resin and Its Bio-Composites

The structure and interlayer spacing (d spacing) of medium-oil alkyd resin HOA-Z and the corresponding alkyd bio-composites prepared via in-situ alcoholysis (that is, CC was added to the monoglyceride oil before polyesterification) have been detected by X-ray diffraction. Figure 6 shows the XRD patterns for HOA-Z (neat polymer) and the bio-composite polymers HOA-1, HOA-2, and HOA-3 prepared by varying the percentage of CC. The medium oil alkyd biopolymer did not show a diffraction peak in the 2θ range from 2 to 150, suggesting that the medium oil alkyd has no ordered structure within this dimension range, but a weak and broad diffraction peak at 18.640 corresponding to a d-spacing of 0.48 nm was observed. This shows that the medium-alkyd biopolymer is amorphous. Lack of sharp crystalline peaks indicates the absence of inorganic material,

signifying that it is not a composite material. Similar observations with *Jatropha curcas* oil-based alky/epoxy/graphene oxide bio-nanocomposites have been reported [31]. The occurrence of the peak at $2\theta = 20.720$, close to that of the ordinary alkyd sample, confirms the presence of CC in the bio-composite samples. This also suggested that the CC has been able to form a phase-separated microporous structure with the alkyd biopolymer. The intensity of the peak increases with an increase in the wt.% of CC. This may be attributed to an increase in the silica layer as CC content increases. The basal spacing of the bio-composites was calculated to be 0.43 nm, and this was similar to all the bio-composite samples, indicating that there is no change in the gallery spaces of CC layers during the polymerization process. This observation corroborates those made by Gogio *et al.* [40]. On the whole, increasing the CC content transitions the alkyd biopolymer from amorphous (HOA-Z) to semi-crystalline (HOA-3). The improved crystallinity of the biopolymer by the addition of CC may enhance barrier properties and thermal stability, while affecting flexibility and toughness. However, optimum performance may be at HOA-2, where the CC dispersion is adequate without excessive aggregation.

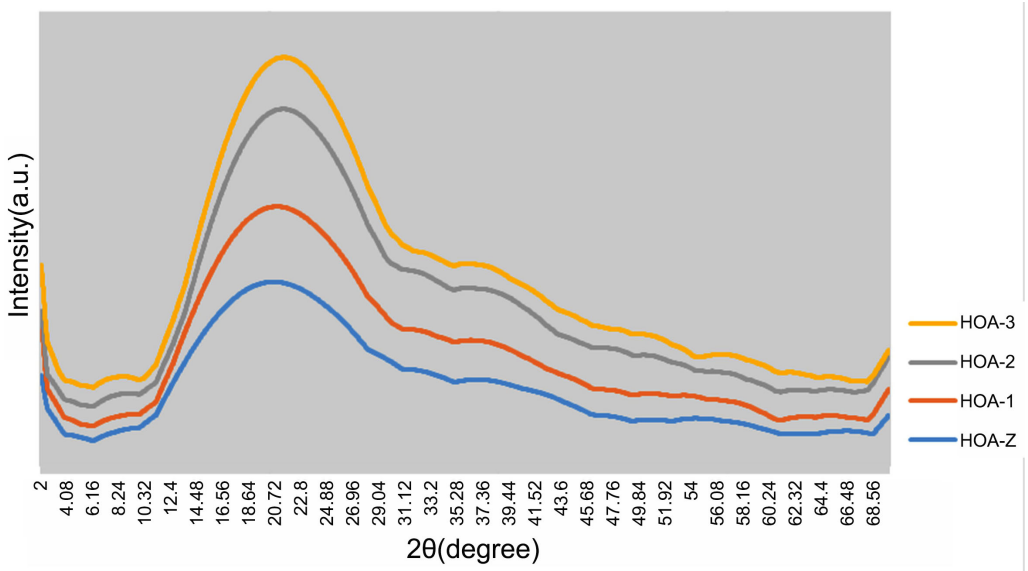


Figure 6. XRD patterns of HOA-Z, HOA-1, HOA-2, and HOA-3.

3.5. Thermogravimetric Analysis of the Alkyd Resin and Its Bio-Composite Samples

The TGA thermogram of the neat polymer (HOA-Z) and the corresponding bio-composites, HOA-1, HOA-2, and HOA-3, are shown in **Figure 7**. The HOA-Z sample shows major weight loss in two stages, the first stage occurring between 150–300˚C, corresponding to volatilization of low molecular weight components and the initial breakdown of ester linkages (**Table 3**). The second stage occurred between 300˚C - 500˚C, indicating the main degradation of the alkyd bio-polymer backbone, ie, oxidative and thermal cleavage of polymer chains. The residual mass

was very low at 600˚C - 700˚C, suggesting the absence of CC. Hence, HOA-Z degrades easily due to the absence of thermally stable CC fillers.

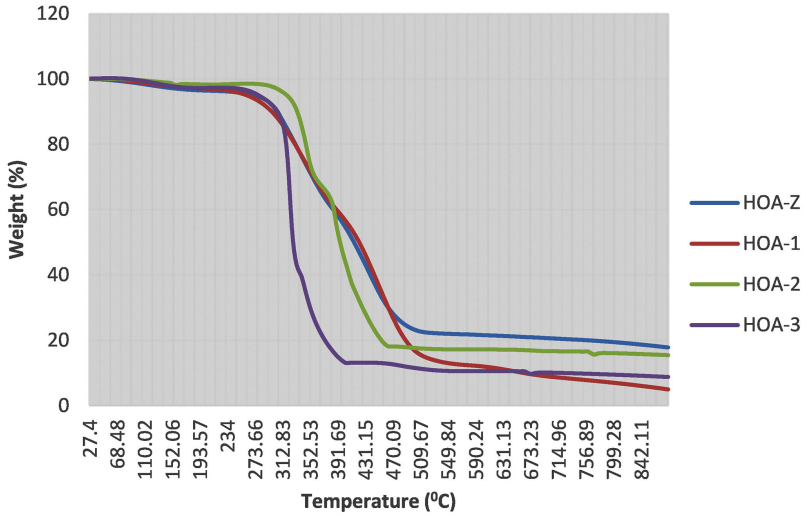


Figure 7. TGA thermogram of the polymer bio-composites.

On the other hand, a slight improvement in thermal stability was observed when 1.98 g of CC (HOA-1) was added to the neat polymer as the inorganic reinforcing agent (**Table 3**). The onset of degradation shifted slightly higher, while the residual mass at higher temperatures is marginally increased, indicating that a small quantity of CC added acts as a heat barrier, delaying degradation slightly. Also, when the CC content was increased to 5.83 g (HOA-2), there was a further shift in the degradation temperature, and more residue was obtained at 600˚C - 700˚C. This implies that an increase in the concentration of CC improves the thermal barrier effects and contributes more to char formation. Calcined clay particles can restrict heat and oxygen diffusion into the alkyd matrix.

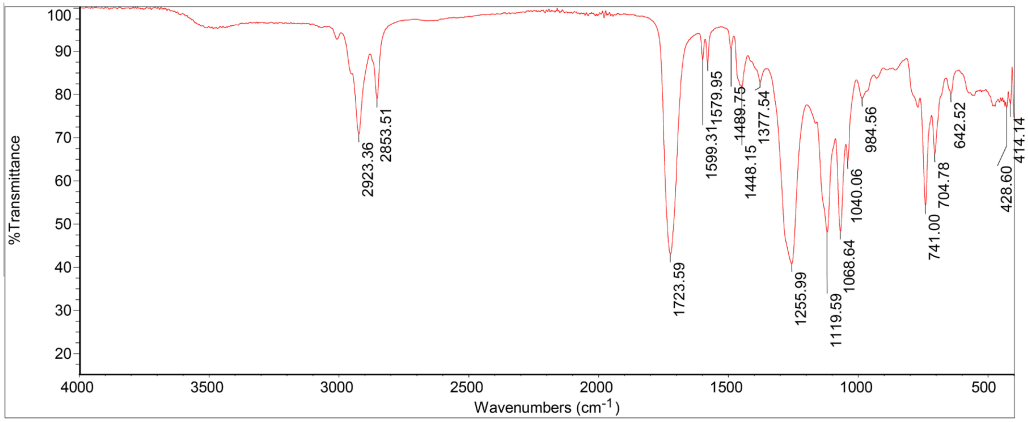
Further increase in the concentration of CC to 9.52 g (HOA-3) leads to the highest onset of degradation among the HOA-Z, HOA-1, and HOA-2 samples. Maximum residues were also obtained at the end of the heating cycle. This signifies a significant enhancement in the thermal stability of the sample. Suggesting that the alkyd bio-composite exhibits stronger interfacial bonding. However, an increase in CC content may limit polymer flexibility, which could affect other properties.

Table 3. Summary of the thermal degradation of the neat polymer and the composite samples.

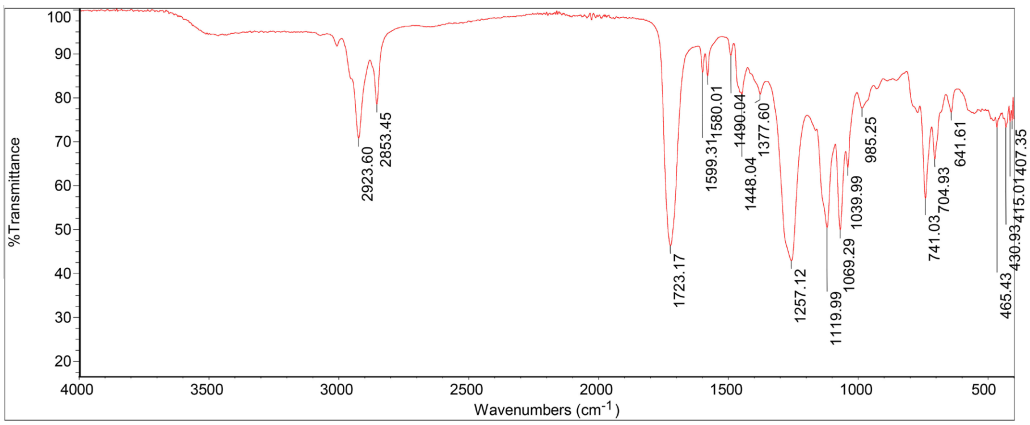
Sample	Onset temperature	Degradation stages	Residue (%)	Stability trend
HOA-Z	Lowest	2	Very low	Baseline
HOA-1	Slight	2	Slight	Slight improve
HOA-2	Moderate	2	Noticeable	Clear improve
HOA-3	Highest	2	Highest	Most stable

3.6. FT-IR Results of the Medium-Oil Alkyd Resin and Its Bio-Composite Polymers

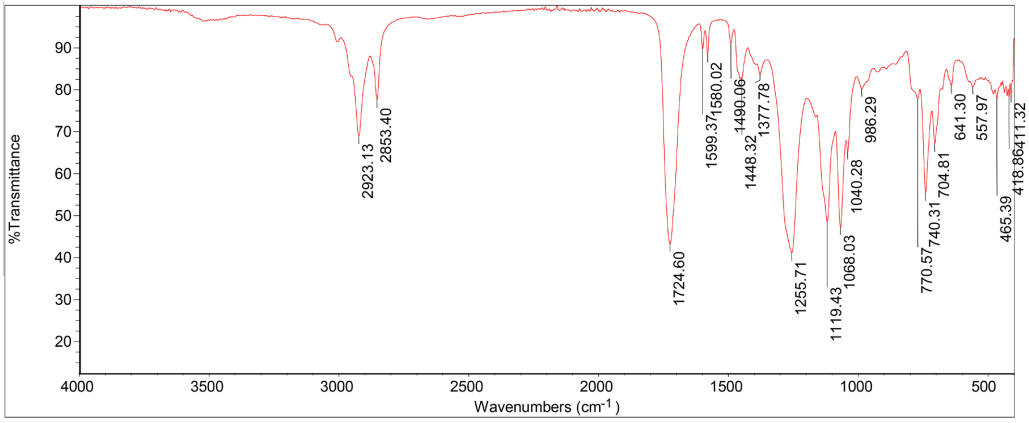
The FT-IR results for the medium alkyd sample HOA-Z and those of the medium oil alkyd bio-composite polymers (HOA-1, HOA-2, and HOA-3) are shown in Figures 8(a)-(d). The absorption bands at 1723.59 cm^{-1} (HOA-Z), 1723.17 cm^{-1} (HOA-1), 1724.60 cm^{-1} (HOA-2), and 1723.47 cm^{-1} (HOA-3) represent the –COO– (ester group) functional group characteristic of alkyd resin. The slight shift in the absorption regions of the bio-composite samples compared to the ordinary alkyd sample may be due to the interaction of CC with the alkyd matrix. The R-CO-O-R^1 of ester corresponds to absorption bands at 1068.64 cm^{-1} for HOA-Z, 1069.29 cm^{-1} for HOA-1, 1068.03 cm^{-1} for HOA-2, and 1067.64 cm^{-1} for HOA-3. The absorption bands at 465.43 cm^{-1} (HOA-1), 465.39 cm^{-1} (HOA-2), and 464.51 cm^{-1} (HOA-3) shifted in descending order as the wt.% of CC increased, which were only observed in the bio-composite samples. The bands represent the Al-O bond of the CC. Previous reports show that calcined clay has about 19.414% Al_2O_3 [33]. The –OH stretching (broad) absorption band at 3450 cm^{-1} for the HOA-Z sample may be from polyol or polybasic acid, suggesting a polymer with terminal –OH groups. The –OH stretching at 3450 cm^{-1} for the composite samples may be enhanced by the surface hydroxyl group of the clay mineral.



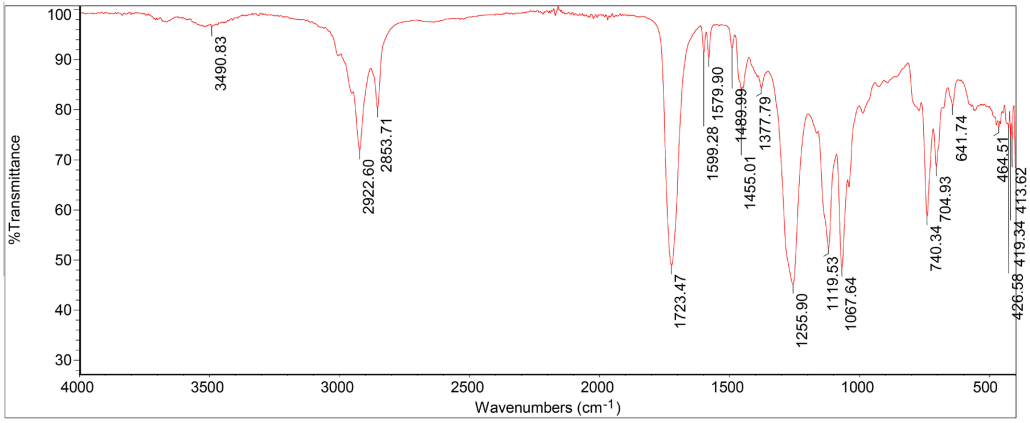
(a) FT-IR result of HOA-Z



(b) FT-IR result of sample HOA-1



(c) FT-IR result of sample HOA-2



(d) FT-IR result of sample HOA-3

Figure 8. FT-IR results of the neat polymer and the bio-composite samples.

3.7. Number-Average Molecular Weight of the Polymer Bio-Composites

Number-average molecular weight of the composite materials was determined in duplicate based on the Rast method described in Isaac and Nsi [29] using Equation (2).

$$\overline{M_{det}} = \frac{k \times y \times 1000}{(T_2 - T_1) \times z} \tag{2}$$

where $\overline{M_{det}}$ is the number – average molecular weight of the biopolymer composite, k is the molecular depression constant of camphor and it is equals 39.7 [47], y is the weight (g) of the composite material, T_2 is the melting point of pure camphor, determined in this research as 176˚C, and z is the weight (g) of pure camphor. The result obtained is as follows: HOA-Z (1157.90 ± 233.91), HOA-1 (827.09 ± 233.93), HOA-2 (590.78 ± 100.26), and HOA-3 (289.48 ± 58.48). The number-average molecular weight of these composite materials was correlated with the viscosity. The result shows a very weakly negative correlation, r (4) = −0.1231, p = 0.877. There was no significant difference at $p < 0.05$. The molecular weight of the biopolymer composite decreases as the percentage of CC increases,

such that the ordinary medium oil alkyd sample HOA-Z had the highest value of 1157.90 ± 233.91, while the bio-composite with the highest amount of CC (9.52 g), HOA-3, had the lowest molecular weight of 289.48 ± 58.48. This trend may be attributed to the ability of the calcined clay to interrupt the composite chain growth, thereby reducing the degree of polymerization as the concentration of calcined clay increases, since the method for the preparation of these composite materials was an *in-situ* two-stage alcoholysis-polyesterification process. Isaac and Nsi [48] determined the average molecular weight of medium oil alkyd resin prepared from cottonseed and melon seed oils as 583.57 and 696.25, respectively. These values corroborate the results obtained for the bio-composites in this research.

3.8. Mechanical Properties of the Bio-Composite Films

The tensile strength and elongation at different CC loading for the neat polymer and the bio-composite samples are shown in Table 4. The tensile strength increases significantly with the addition of CC from 18 MPa (HOA-Z) to 44 MPa (HOA-3) when 5% of CC is added. The reinforcement effect may be attributed to the rigid, inorganic nature of the calcined clay that enhances the load-bearing capacity of the bio-composite. Maximum strength was observed at the highest CC loading (HOA-3), *i.e.*, 5% CC addition, suggesting strong interfacial adhesion and good dispersion of the CC with the polymer matrix.

Elongation at break decreases with increased CC loading from 62% (HOA-Z) to 19% (HOA-3), also suggesting a significant loss in ductility. This reflects the increased stiffness and restricted polymer chain mobility as the rigid particles hinder deformation. Therefore, as the CC content increases, the composite biopolymer becomes more brittle.

However, increasing CC content up to 5% (HOA-3) results in a composite with reduced flexibility. An optimum performance might be obtained at 3% CC loading (HOA-2), which offers significant strength improvement (31 MPa) while retaining moderate elongation of 36%.

Table 4. Mechanical properties of the alkyd bio-composite films.

Filler loading (wt, %)	Tensile strength (MPa)	Elongation (%)
0	18	62
2	27	40
3	31	36
5	44	19

3.9. Limiting Oxygen Index

Table 5 shows the flame behaviour, smoke/fume evolved, and char characteristics for the ordinary alkyd resin and its bio-composites prepared with 2%, 3%, and 5% calcined clay. The flame characteristic of the neat polymer (candle flame) shows poor flame retardancy with an LOI value of 17.5%. The smoke is typical of organic

compounds with hydrocarbon chains that require less oxygen for the formation of flammable volatiles and propagation of flame during thermal decomposition. The char is a little contributing to the high flammability. A similar observation was made by Gogoi *et al.* [40]. But the addition of 2 wt.% CC enhances the LOI value of 27.5% and increases steadily with increasing CC loading, and finally it reaches the maximum (42%) when CC loading is 5 wt.%. The flammability property of the composite has improved significantly with the addition of the CC reinforcing agent. This may be due to the barrier property and thermal stability of CC to heat and oxygen transport, which in turn delays the ignition of the bio-composite [40] [44].

Table 5. Limiting oxygen indices (LOI) and flaming characteristics of the alkyd and bio-composites.

CC content (wt%)	LOI (%)	Flame description	Smoke/fumes	Characteristics
0	17.5 ± 2.12	burns readily in air	dense smoke/irritant fumes	little
2	27.5 ± 0.71	self-extinguishing in ambient air	reduced smoke/fumes	thin and cohesive
3	32.3 ± 1.77	small and localized	decreased smoke/fumes	denser and more stable
5	42.0 ± 1.41	small and localized	less dense	thick and cohesive

4. Conclusion

Bio-composites polymers based on *Hura crepitans* L seed oil alkyd resin biopolymer reinforced with 2 - 5 wt.% of calcined clay were prepared by *in-situ* alcoholysis polyesterification method. The viscosity of the bio-composites decreases from 17430.0 to 737.5 mPa.s as the amount of CC increases. The molecular weight of the biopolymer composite decreases as the percentage of CC increases, such that the ordinary medium oil alkyd sample HOA-Z had the highest value of 1157.90 ± 233.91. In contrast, the bio-composite with the highest amount of CC (9.52 g), HOA-3, had the lowest molecular weight of 289.48 ± 58.48. The molecular weight was weakly negatively correlated with viscosity ($r(4) = -0.1231$, $p = 0.877$) and there was no significant difference at $p < 0.05$. The absorption bands at 1723.59 cm^{-1} (HOA-Z), 1723.17 cm^{-1} (HOA-1), 1724.60 cm^{-1} (HOA-2), and 1723.47 cm^{-1} (HOA-3) represent the –COO– (ester group) functional group characteristic of alkyd resin. The XRD results of the neat polymer and the composites show that the medium–alkyd biopolymer is amorphous, while the bio-composites are semi-crystalline. The SEM micrograph of the medium-alkyd shows no significant cracks or voids, which indicates that the neat polymer network is cohesive and well developed. The SEM micrograph of the bio-composites reveals a heterogeneous to rough heterogeneous surface, respectively, with distinct aggregates and clusters of CC, indicating partial dispersion of the CC within the alkyd matrix. The thermal, mechanical, and flame-retardant properties of the bio-composites are found to be improved due to the incorporation of CC. The tensile strength increases significantly with the addition of CC from 18 MPa (HOA-Z) to 44 MPa (HOA-3) when 5% of CC is added. Elongation at break decreases with increased

CC loading from 62% (HOA-Z) to 19% (HOA-3), also suggesting a significant loss in ductility. The flame characteristic of the neat polymer (candle flame) shows poor flame retardancy with an LOI value of 17.5%. The LOI value of the bio-composite increased significantly with increasing CC loading.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Credit Authorship Contribution Statement

Ibanga O. Isaac: Project administration, Conceptualization, Supervision, Formal analysis, Methodology, Visualization, Writing: original draft.

Itoro E. Willie: Formal analysis, Software, Writing: review & editing.

Itohowo O. Akpan: Investigation, Writing: review & editing, Conceptualization, Data curation.

Issa A. Abdulazeez: Resources, Software, Visualization, Validation.

Mmeyene P. Raphael: Resources, Software, Validation.

Ubong I. Etukudo: Methodology, Formal analysis, Visualization.

Emah O. Akpan: Writing: original draft, Validation, Investigation.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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