Extraction, Synthesis and Characterization of an Alkyd Resin from *Sesamum indicum* Seed Oil

Kalu M. Kalu[1]* , Michael Emmanuel[2], Emmanuel K. Chinedu[3], Ayodele Akinterinwa[3], Uhiene Titus[3], Rome Kenneth[4], Naibi A. Haruna[5], Buba A. Aliyu[3]

[1]Department of Chemistry, Gombe State University, Gombe, Nigeria
[2]Department of Chemistry, Saint Louis University, St. Louis, USA
[3]Department of Chemistry, Modibbo Adama University, Yola, Adamawa State, Nigeria
[4]Department of Chemistry, University of Ilorin, Ilorin, Nigeria
[5]Department of Chemistry, Adamawa State College of Agriculture, Ganye, Nigeria
Email: *michaelkalu14@gmail.com

Abstract

The synthesis and characterization of *Sesamum indicum* seed oil have been thoroughly examined. The seed oil was extracted and utilized in formulating alkyds with varying oil lengths of 40%, 50%, and 60%. The in-situ process for the synthesis was analyzed, revealing significant properties associated with the different oil lengths, including changes in degree of polymerization, average molecular weight, and, notably, the extent of reaction favoring the 40% oil length alkyd resin. Furthermore, the chemical resistances of the various oil lengths were assessed to determine their susceptibility to acidic, basic, brine, and distilled water media. Encouragingly, all the alkyd resins demonstrated good chemical resistance. Additionally, the 40% oil length alkyd resin exhibited superior moisture uptake, which prompted further characterization using Fourier transform infrared spectroscopy, scanning electron microscopy, thermogravimetric analysis, and differential scanning calorimetry. These techniques were employed to explore the exceptional properties of the 40% oil length alkyd resin. Overall, this research sheds light on the synthesis, characterization, and comparative properties of *Sesamum indicum* seed oil-derived alkyds with varying oil lengths. The findings contribute to a deeper understanding of their unique attributes and potential applications.

Subject Areas

Biological Chemistry

Keywords

Alkyd Resin, Oil Length, Synthesis

1. Introduction

Vegetable oils (VO) are extracted from oil containing seeds, nuts or fruits by various squeezing techniques, solvent extraction or a blend of these [1]; they are employed widely in modern applications like plasticizers, biodiesel, greases, glues, biodegradable bundling materials, printing inks, paints and coatings [2]. They are non-harmful, non-depletable, locally bountiful, stable and biodegradable substances [3]. They yield polymers fit for contending with non-renewable energy source inferred petroleum-based items. Such polymers discover applications in the improvement of paints and coatings, other than their other mechanical applications [4].

Alkyds are oil-altered polyesters, comprising of a polyol (typically glycerol), an anhydride (phthalic acid) and an unsaturated fat produced by transesterification process. The binder in most conventional paints, makes it simple to create a consistent coating that adheres to the surface of the substrate and holds other materials together [5] [6]. Alkyd resin is the result of the poly-condensation of a polybasic acid, such as phthalic anhydride, and a polybasic alcohol (such as glycerol), modified with a monobasic fatty acid, or its triglyceride oil [7]. Polybasic acids are those that have many hydrogen atoms that can be replaced. Phthalic anhydride, glutaric anhydride, maleic anhydride, and succinic anhydride are used in the manufacture of alkyd resins [8] [9]. Sebacic, isophthalic, and fumaric acids are further polybasic acids. The fact that Phthalic anhydride has a relatively low melting point (the pure compound melts at 131˚C), which is below the reaction temperature of 230˚C - 250˚C for the synthesis of alkyd resin, could be attributed to the reason why most alkyd chemists and technologists prefer Phthalic anhydride over other polybasic acids [10]. This suggests that the Phthalic anhydride (PA) crystals will dissolve and melt quickly in the reaction mixture at the reaction temperature, shortening the reaction time. Additionally, it boosts the resin's hardness and water resistance while enhancing the resin's solubility. Molten PA is utilized in large-scale manufacturing, which lowers the cost of packing, shipping, and handling. Isophthalic acid ranks as the second most often utilized acid. Esters of isophthalic acid, which are crucial for external durability, are more resistant to hydrolysis than Phthalic anhydride esters in the pH range of 4 to 8 [10]. On the other hand, Phthalic anhydride esters are more resistant to hydrolysis than isophthalic esters in alkaline circumstances. However, due to its high melting point of 330˚C, which is above the reaction temperature of 230˚C - 250˚C, the use of isophthalic acid in the synthesis of alkyd resin presents an issue of longer reaction time and higher temperature. Although it creates a better-performing product than phthalic anhydride, isophthalic acid is also excessively expensive. Although more expensive, fumaric acid also makes resin with greater impact resistance [11].

Alkyds are delegated short (30% - 42%), medium (43% - 54%), long (55% - 68%) and extremely long (>68%), considering the percent weight part of the oil in the resin. The popularity of alkyd gums as vehicle for coatings is generally be-

Table 1. Formulation of 40% oil length alkyd resin (300 g).

Recipe	Amount charged (g)	Functionality	Molecular weight (g)	Equivalent weight	Equivalent mole	Number of acid equivalent	Number of moles
Sesame seed oil	120.00	1.00	325.97	325.97	0.37	0.37	0.37
Phthalic anhdride	108.91	2.00	148.00	74.00	1.47	1.47	0.74
Glycerol	71.09	3.00	92.00	31.00	2.29	-	0.77
Total	300.00	-	-	-	-	1.84	1.88

Table 2. Formulation of 50% oil length alkyd resin (300 g).

Recipe	Amount charged (g)	Functionality	Molecular weight (g)	Equivalent weight	Equivalent mole	Number of acid equivalent	Number of moles
Sesame seed oil	150.00	1.00	325.97	325.97	0.47	0.47	0.46
Pthalic anhydride	90.59	2.00	148.00	74.00	1.22	1.22	0.61
Glycerol	59.41	3.00	92.00	31.00	1.92	-	0.65
Total	300.00	-	-	-	-	1.69	1.72

Table 3. Formulation of 60% oil length alkyd resin (300 g).

Recipe	Amount charged (g)	Functionality	Molecular weight (g)	Equivalent weight	Equivalent mole	Number of acid equivalent	Number of moles
Sesame seed oil	180.00	1.00	325.97	325.97	0.55	0.55	0.55
Pthalic anhydride	72.27	2.00	148.00	74.00	0.98	0.98	0.49
Glycerol	47.73	3.00	92.00	31.00	1.54	-	0.52
Total	300.00	-	-	-	-	1.53	1.56
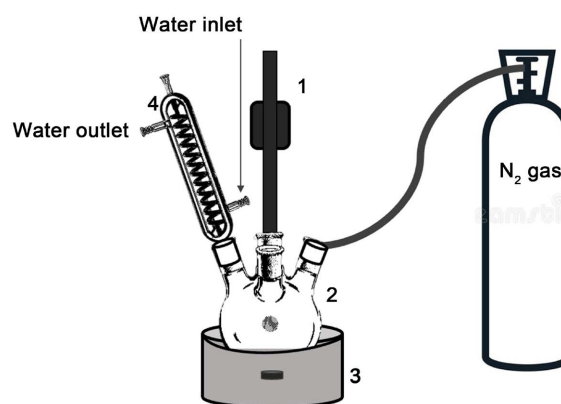
Figure 1. An experimental setup of the alkyd resin synthesis: (1) Mechanical stirrer, (2) 4-neck round bottom flask, (3) Heating mantle, (4) Reflux condenser.

to an elevated temperature of 200˚C and maintained for 2 hours, as the reaction progressed aliquots were constantly been taken at 30 minutes intervals to check for its solubility in 1 to 3 volumes of methanol which clearly indicated the for-

mation of a monoglyceride upon giving a clear solution when observed. The system was then allowed to cool to about 120°C to allow for onset introduction of the trans-esterification stage.

The poly-condensation stage was initiated when the temperature of the monoglyceride stage was brought down to about 120°C and 108.91 g of phthalic anhydride was added to the reaction mixture, followed by the addition of xylene (10% of the total resin weight) to aid eliminate the water of esterification by producing an azeotrope [16] [17]. The temperature was extended to 230°C - 250°C while the reaction progresses. Aliquots were taken from the reaction blend at time periods of 30 minutes in other to determine the drop-in acid value and the volume of water removed was also recorded. The drop-in acid values were determined by titrating two aliquot portions of the mixture withdrawn against 0.1 M KOH solution, a measured amount of phenolphthalein dissolved in a mixture of ethanol and toluene (1:1) served as an indicator [18]. The reaction was halted when the acid value reached a value of less than 10 mg KOH/g. This process was repeated for the 50% and 60% alkyd resin oil lengths respectively. The pathway for the alkyd resin synthesis is shown in Figure 2(a) and Figure 2(b) respectively.

2.2.3. Instrumentation

The analysis was performed on the 40% alkyd resin due to its remarkably low moisture absorption, degree of polymerization, extent of reaction and higher number-average molecular weight formation.

FTIR–8400S, Fourier Transform Infrared spectrophotometer to determine the functional groups present, the morphology of the alkyd resin sample was investigated using a scanning electron microscope (SEM). A JOEL JSM 5600 LV model scanning electron microscope with an accelerating voltage of 15 kilovolts was employed to observe the surface morphology of the alkyd, the resin was coated with gold before analysis to avoid any charge build-up by the electron absorbed by the resin [19].

Thermo-gravimetric analysis (TGA) in an environment of argon at a heating rate of 10°C /min from 30°C to 950°C using the PerkinElmer TGA model, the thermal characteristics of the resin sample were examined.

Using thermogravimetric analysis (TGA), variations in mass with respect to temperature are found. It is typically used to calculate the degradation temperatures, absorbency levels of materials, amounts of inorganic and organic components present in a material, and solvent residue analyses. The sample is hung in a furnace that has a temperature programmer under control using an electrical balance.

Differential scanning calorimetry was carried out using the STAR–SC/DTA F3 JUPITER instrument (NETZSTH, Germany) model within the temperature interval from 20°C to 250°C under nitrogen atmosphere. It is a method of thermal investigation that looks at how temperature affects a material's heat capacity. When a known mass sample is heated or cooled, changes in the heat flow are seen as a result of differences in the sample's heat capacity. This makes it
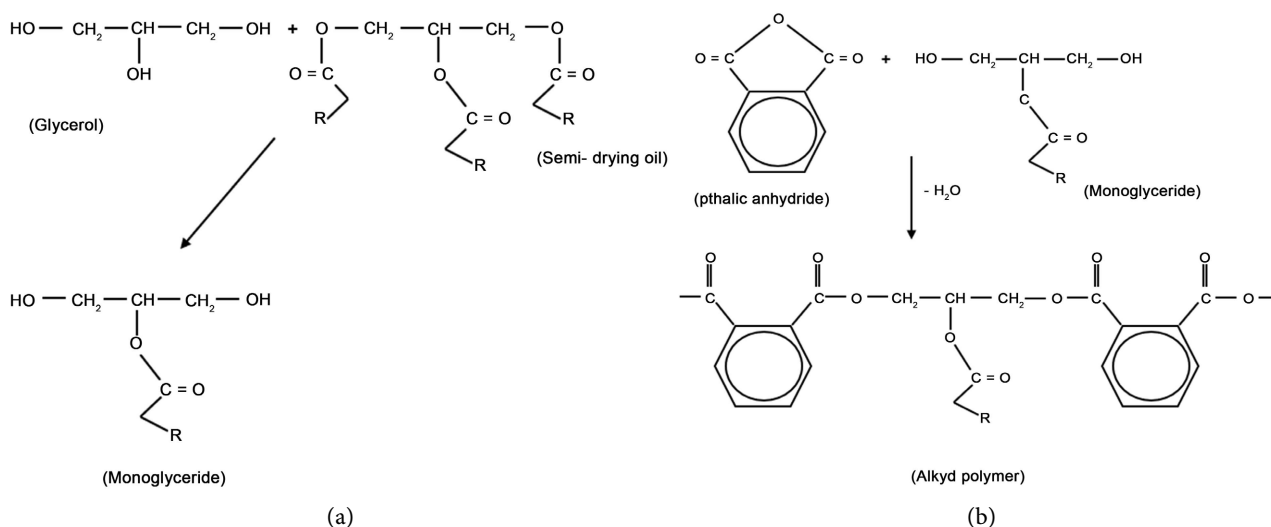
Figure 2. Pathway for the alkyd resin synthesis: Alcoholysis-transesterifications stage (a) and Polycondensation stage (b).

possible to see transitions like melt-to-glass transitions (Tg), melting point (Tm), and crystallization degree (Tc) [20].

3. Results and Discussion

3.1. Physicochemical Properties of Sesame Oil

The physiochemical properties of the *Sesamum* seed oil are shown in Table 4. It has a specific gravity of 0.92, less dense than water. The iodine value is 122.33 g I_2/100 g which implies that SISO is a semi-drying oil. The iodine value is an important parameter employed in ascertaining the suitability of oil for alkyd synthesis. It can be concluded that the SISO is a semi-drying oil whose iodine value is in the range of 120 - 150 g I_2/100 g [21]. Semi-drying oils have various applications such as in the synthesis of alkyd resins for the paint industry and in the manufacturing of soap, whereas the non-drying oils are majorly employed as plasticizers [22].

The SISO had a peroxide value of 6.15 mEq/kg which suggests that the oil is not prone to oxidative rancidity [23].

The percentage yield is calculated to be 19.48% w/w. However, the low percentage oil yield may not be unconnected to the fact that the hot water floatation method is less efficient when compared to the solvent extraction method.

The saponification value obtained for the SISO is 172.13 mg KOH/g and this suggests that the SISO may be suitable for the manufacture of soap due to its high molecular weight fatty acid [24].

The acid value of the SISO was found to be 6.86 mg KOH/g which suggests low lipolytic activity of enzymes in the sesame seed which also denotes its stability [25] and thus enhances its industrial application in the manufacture of paints and varnishes at low acid values [26]. Acid value is used to measure the level of deterioration of oil. It has been reported that high acid value of oil could be due to hydrolytic reaction during processing of the oil or as a result of enzymatic

Table 4. Physicochemical properties of *Sesamum indicum* seed oil (SISO).

Properties	SISO
Iodine value (g I_2/100 g)	122.33
Peroxide value (mEq/kg)	6.15
Free fatty acid (%)	6.22
Saponification value (mg KOH/g)	172.13
Acid value (mg KOH/g)	6.86
Viscosity (mPa·s, 30°C)	65.29
Refractive index (30°C)	1.468
Specific gravity (g/cm³, 30°C)	0.92
Color	Brownish yellow
Percentage oil yield (w/w)	19.48

action in the oil-bearing seed [27]. The refractive index obtained for SISO is 1.468 and is within the range of 1.460 - 1.540, which suggests that it can serve as a test for purity and a means for identification [28].

3.2. FT-IR Characterization of the Extracted SISO

Fourier transforms infra-red spectrum of the extracted seed oil was obtained using FT-IR–8400S in other to determine the functional groups present as shown in Figure 3. Since the extracted SISO and the synthesized alkyd were composed of essential esters; it was observed that their FT-IR spectra were found at similar frequencies of absorption; the O-H carboxylic acid stretch which was observed at the 2635.00/cm in the extracted oil as seen at the 2682.59/cm in the alkyd spectrum, the -CH_2 alkane stretching which was observed at the 2932.17/cm in the extracted SISO was observed at the 2933.51/cm in the alkyd spectrum, the C=O stretching of an ester which was observed at the 1743.83/cm in the extracted SISO was observed at the 1728.60/cm in the alkyd spectrum, another similarity was noted in their C=C aromatic stretch which was found at 1454.59/cm in the extracted SISO and occurring at 1585.93/cm in the alkyd spectrum.

However, the synthesized alkyd differed from the extracted SISO possessing a strong, broad O-H stretch of alcohol at the 3454.21/cm which could be attributed to the presence of an intermolecular bond between the extracted SISO and glycerol, it was also observed that there was a strong, broad C=O stretching of a tertiary alcohol which was absent in the extracted SISO spectrum at the 1192.01/cm wave number.

3.3. In-Situ Characterization of the Alkyd Resin

The extracted SISO was used to formulate three grades of alkyd resin of oil lengths 40%, 50% and 60% respectively, each comprising varying concentrations of glycerol, phthalic anhydride and the extracted oil as enumerated in Tables 1-3. A two-staged alcoholysis-polyesterification process as described by Oladipo [6] was adopted. The in-situ process was reported as follows:
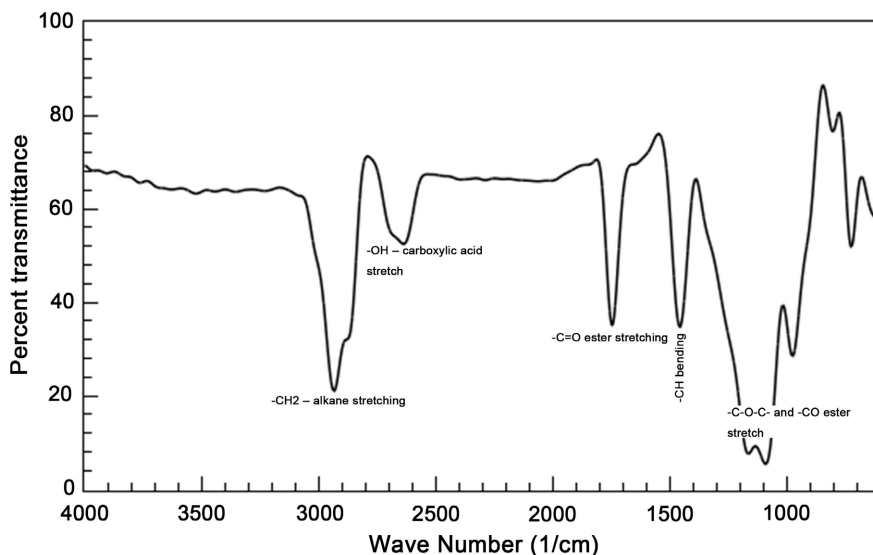
Figure 3. FT-IR Spectrum of *Sesamum indicum* seed oil, revealing all the functional groups present in the seed oil.

3.3.1. Acid Value

The graph below shows the result of the variation of acid value with reaction time of the various alkyd resin oil lengths; group [29] reported that acid values less than 15 are suitable for paint application which signifies a reasonable amount of conversion.

It was observed that there was a corresponding decrease in acid values at 40%, 50% and 60% oil lengths as the reaction progressed with time as seen in **Figure 4(a)**, there is also a significant decrease in the acid values at the earlier stages and then followed by a gradual decrease which could be attributed to the variations in reactivities of the primary and secondary hydroxyl groups of the glycerol with the phthalic anhydride carboxyl group [12].

It was reported by Nway and Mya [29] that the primary hydroxyl groups of the monoglyceride react faster than those of the secondary hydroxyl groups of the monoglyceride with the added phthalic anhydride which also accounts for the decrease in the acid values observed, and this is due to the increased concentration of these functional groups present in the glycerol and the phthalic anhydride at the first phase of the reaction which is characteristic of a step-growth polymerization [30].

3.3.2. Volume of Water Evolved

It was observed that as the oil length increased, the total volume of water evolved also increased, at the onset of the synthesis, the rate of water removal was high, and this trend is followed by a gradual removal rate at longer periods of reaction from 30 to 180 minutes till no water of esterification is observed. This reveals the formation of alkyd resin since all the available reactive species have reacted at this stage which can also be related to the acid value changes highlighted in **Figure 4(b)**.
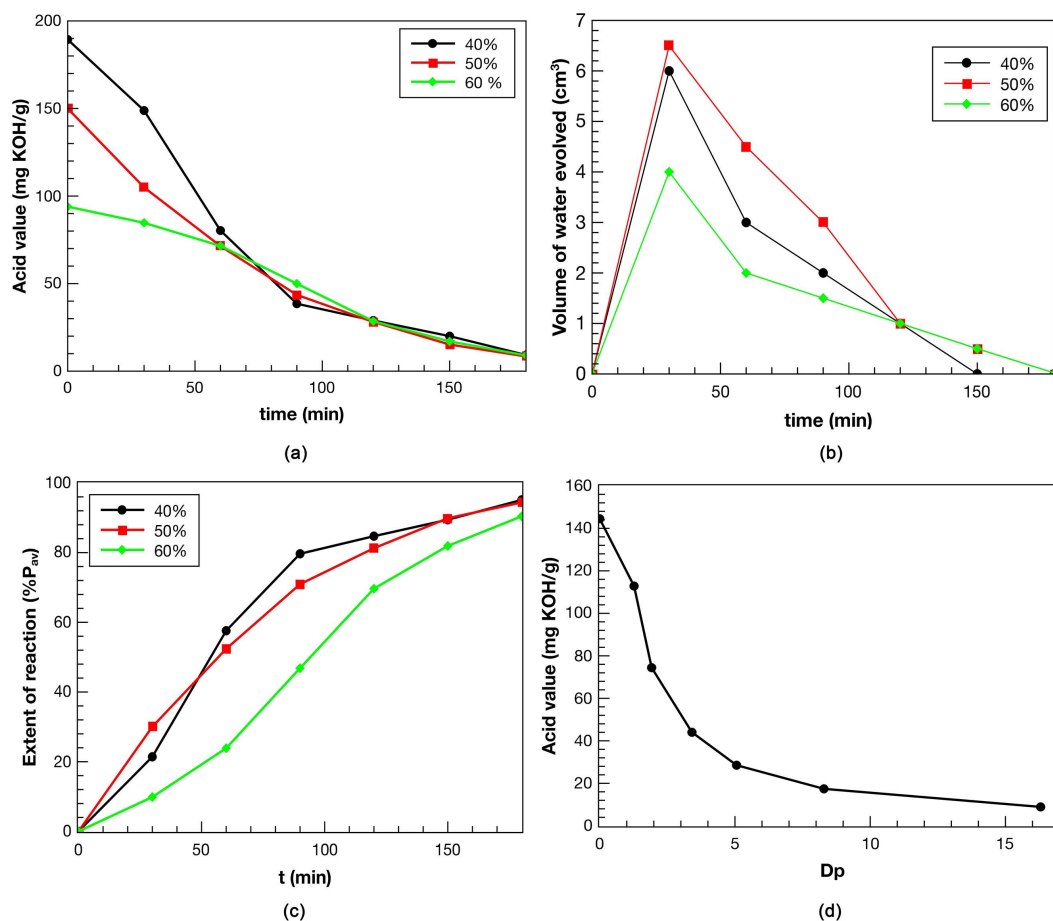
Figure 4. In-situ characterization of alkyd synthesis: acid value (a), volume of water evolved (b), extent of reaction (c), and average acid value vs degree of polymerization (D_p) (d).

3.3.3. Extent of Reaction

The extent or fraction of the reaction is defined as the fraction of the hydroxyl or carboxyl functional group that has taken part in the reaction at a given time [24], **Figure 4(c)** shows that the extent of the reaction otherwise known as the esterification conversion percentage (%P_{av}) increased with increase in reaction. It can also be observed that this trend is faster in the 40% alkyd resin synthesis than in the 50% and 60% synthesis processes.

3.3.4. Average Acid Value versus Average Degree of Polymerization (D_p)

The average acid value decreases with an increase in the average degree of polymerization for the SISO alkyd resin samples as seen in **Figure 4(d)** which was expected owing to the formation of high molecular weight alkyd resin polymer.

3.3.5. Degree of Polymerization (D_p)

The variation in degree of polymerization with reaction times is shown in **Figure 5(a)**. There is a linear behavior at the onset of the reaction progress from 0 to 60 minutes before the deviation from linearity which marks the occurrence of branching along the polymer chain [31], nevertheless a second order kinetics was observed at ≥ 9 D_p region, equivalent to >70% conversion, at this period,
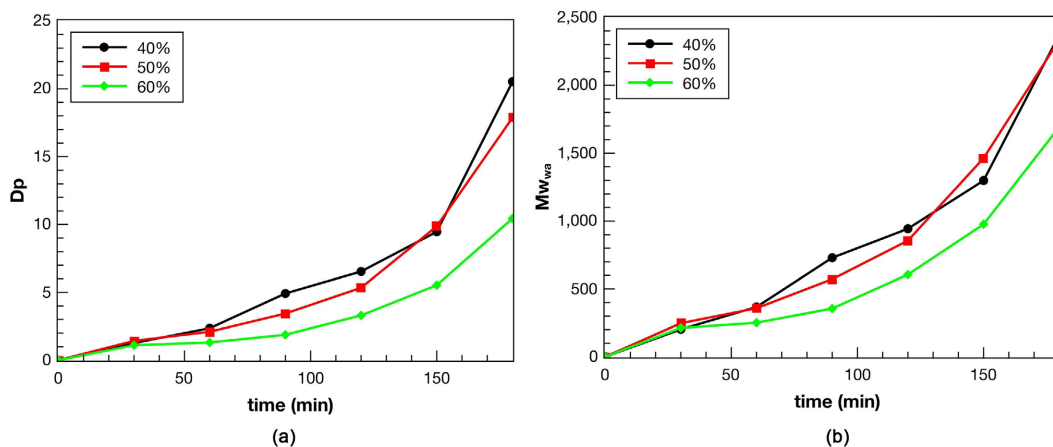
Figure 5. In-situ characterization of alkyd synthesis: degree of polymerization (D_P) (a), and weight-average molecular weight (b).

a straight-line plot was obtained. This portion could also be used to mark changes in physical characteristics of the reaction process (such as changes in viscosities) [32]. It can also be observed that the 40% alkyd had the highest degree of polymerization followed by the 50% and 60% in that order and this corresponds to the reaction times of the primary and secondary hydroxyl groups which results in the yield of linear and three-dimensional molecules respectively [12].

3.3.6. Weight-Average Molecular Weight

The average molecular weight (M_{av}) with respect to the acid value at different stages of the reaction is calculated using the equation:

$$M_{av} = W/(k - P_{av})\ell A \quad [18]$$

where W is the total weight of all the reactants; k is the ratio of the total moles of all reactants to the total equivalent of the acid (N_m/E_a); ℓA is the total equivalent of the acid and P_{av} is the extent of reaction.

It is deduced that no reasonable increase in the molecular size of the reaction mixture occurred during the early stages of the reaction. However, as shown in **Figure 5(b)**, there is a significant increase as the reaction progresses. The low rate of polymerization is responsible for the low molecular weight observed at the start of the reaction. An increase in polymerization rate led to an increase in weight-average molecular weight.

This pattern continued until the gelation point, when structural changes in the polymer size of the molecule in solution begin. The weight-average molecular weight of the alkyd decreased as the length of the oil increases. This is in line with previous findings by Aigbodion and Oladipo [6] [12].

It has also been suggested that increasing the oil length increases the fatty acids available for the reaction, resulting in a higher chance of chain growth termination, resulting in lower molecular weight polymers [33] and this can be seen evidently in the 40% alkyd resin having the highest weight-average molecular weight followed by the 50% and 60% alkyd with respect to their oil lengths.

3.4. Physicochemical Properties of the Alkyd Resins

The physicochemical properties have been summarized as shown in Table 5, it revealed that the saponification values increased with an increase in the oil length of the resins indicating that the level of ester linkages increased as the quantity of the oil in the resins increases for 40%, 50% and 60% alkyd resin oil lengths was 227.5, 254.5 and 258.4 mg KOH/100 g respectively, their saponification values were expected to be higher than the crude *Sesamum* oil since the alkyd were essentially polyesters [12].

The characterization of the alkyd gave iodine values of 70.2, 74.1 and 80.2 g I_2/100 g. The iodine values increased with an increase in the oil length from 40% to 60% in the alkyd resins which can be attributed to the level of unsaturation increasing as the quantity of the oil used in the resins was increased. The level of unsaturation indicated by the iodine value influences the drying properties. The iodine value of the alkyd resin was observed to have decreased considerably when compared to that of the crude seed oil. This could be a result of dimerization and polymerization reactions at the reactive double bonds of unsaturation of the oil during the alkyd synthesis.

The various viscosities are 123.22 mPa.s, 115.6 mPa.s and 110.34 mPa.s respectively for the 40%, 50% and 60% alkyd resins. The 40% alkyd resin sample had the lowest moisture absorption when compared to the 50% and 60% alkyd resins: 0.22%, 0.35% and 0.41% respectively.

The color of the alkyd is dark brown. The darkening in color of the alkyd could be attributed to high temperatures of reaction, oxidation and the use of a catalyst [31]. The densities of the various alkyd resin oil lengths at 30˚C 0.953, 0.947 and 0.938 (g/cm^3) were seen to be high compared to their corresponding values obtained in the seed oil. This could also be a result of polymerization reactions at the reactive double bonds of the unsaturated oil leading to increased molecular weights and viscosities during the alkyd synthesis.

3.5. Evaluation of Chemical Resistance of the Alkyd Resin Films in Different Solvent Media

Four media were used to test the alkyd resin film's resistance: 0.1M KOH solution, distilled water, 5% w/v NaCl solution and 0.1M H_2SO_4. After been submerged in the various media for 7 and 18 hours respectively, it was observed that the various oil lengths of alkyd resins were not affected by brine and distilled water, but they were affected by the acidic and basic media to some extent. The 40%, 50% and 60% oil length alkyd films were fairly resistant to the acidic medium but prone to undergoing alkaline hydrolysis as shown in Table 6.

3.6. Instrumental Analysis

Thermogravimetric analysis, Differential scanning calorimetry, Fourier-transform infra-red spectrophotometry and scanning electron microscopy were employed to investigate the suitability of the 40% alkyd resin owing to its exceptional

Table 5. Physicochemical properties of *Sesamum indicum* seed oil modified alkyd resin.

Property	Alkyd resin grades		
	I (40% A.R)	II (50% A.R)	III (60% A.R)
Refractive index (30˚C)	1.4744	1.4760	1.4763
Colour	Dark brown	Dark brown	Dark brown
Iodine value (g I_2/100 g)	70.2	74.1	80.2
Saponification value (mg KOH/100 g)	227.5	254.5	258.4
Density (g/cm³)	0.953	0.947	0.938
Free fatty acid value	0.679	1.167	1.856
Moisture uptake (%)	0.22	0.35	0.41
Turbidity (NTU)	1076	1074	1075
Acid value mg KOH/g	1.35	2.32	3.69
Viscosity (m.Pa.s)	123.22	115.60	110.34

Table 6. Chemical resistance of the alkyd resin films in different solvent media.

Media	Time (Hrs)	Effect on film		
		40%	50%	60%
Distilled water	7	N	N	N
	18	N	N	N
0.1M NaOH	7	W	W	W
	18	R	R	R
0.1M H_2SO_4	7	N	N	N
	18	S	S	S
5% w/v NaCl	7	N	N	N
	18	N	N	N

Key: N = No effect, W = Whitening, S = Shrinkage, R = Removal of film.

degree of polymerization, moisture uptake and higher molecular weight polymer species formation.

3.6.1. Thermogravimetric Analysis of the 40% Alkyd Resin

The alkyd resin thermal degradation can be divided into three phases: drying, main pyrolysis and carbonization, the first phase, between 100˚C and 150˚C, results in a weight loss of around 4% which could be attributed to moisture vaporization and the possible emission of volatile organic compounds. The degradation begins around 300˚C and spreads across a wider temperature range. The second stage, between 300˚C and 500˚C, could be attributed to the removal of some chemical species that could have been identified using a combined TG-MS, and the third stage, above 500˚C, reflects total decomposition to char.

The results such as Initial decomposition temperature (T_i), final decomposi-

tion temperature (T_f), the temperature of 50% weight loss ($T_{50\%}$), and percent residue at 500˚C is shown in Figure 6. The temperatures of 50% weight loss of the 40% alkyd resin as a standard indication for thermal stability of polymers was ≤440˚C which indicates they could meet temperature resistant requirements, which can be used in surface coating application. The percent weight residue of the 40% alkyd resin is 10% at 500˚C [34].

3.6.2. Scanning Electron Microscopic Analysis of the 40% Alkyd Resin

The morphology of the alkyd resin sample was investigated using a scanning electron microscope (SEM). A JOEL JSM 5600 LV model scanning electron microscope with an accelerating voltage of 15 kV was employed to observe the surface morphology of the 40% alkyd resin sample at different magnifications as seen in Figure 7(a) and Figure 7(b), whilst Figure 8 displays a 3D interfacial plot of the polymer matrix of the 40% alkyd resin sample (Figure 7(c)).

The SEM micrograph highlighted regions that are homogenous and void of particles and regions that are reflective of particles unevenly distributed along the alkyd film, the magnification shows that the homogenous surface could be attributed to the formation of a polymer matrix that is strongly held forming the polymer backbone which can also be ascribed to the rigidity of the alkyd film, the SEM micrograph also revealed some areas of weakness in the form of an underlying crack which could be corrected if the alkyd was to be co-polymerized or blended.

3.6.3. Differential Scanning Calorimetric Analysis of the 40% Alkyd Resin

The temperature at which the polymer changes from the glassy state to the rubbery state in the amorphous region as seen in Figure 8, indicates that the value of the glass transition (T_g) of the 40% alkyd resin is 34.99˚C and this value lies between the −93.5˚C to 226.85˚C range for most synthetic polymers [35] at temperatures above Tg, polymer molecules absorb energy to form ordered arrangements and undergo crystallization. This is an exothermic process and
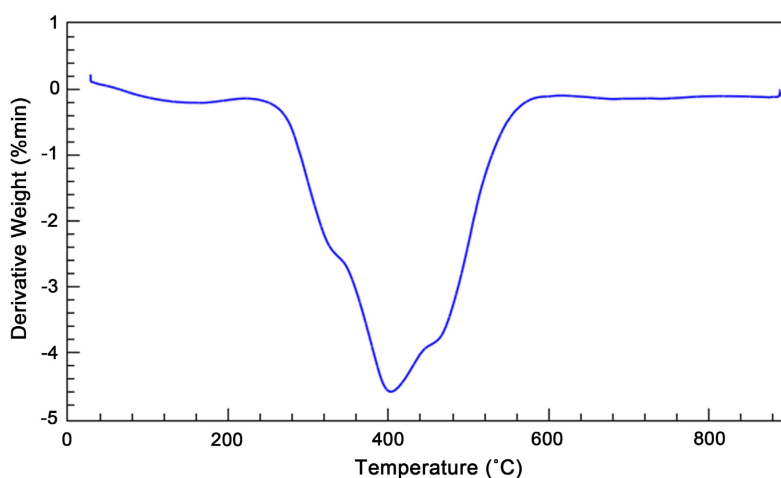

Figure 6. TGA curve of the 40% alkyd resin.
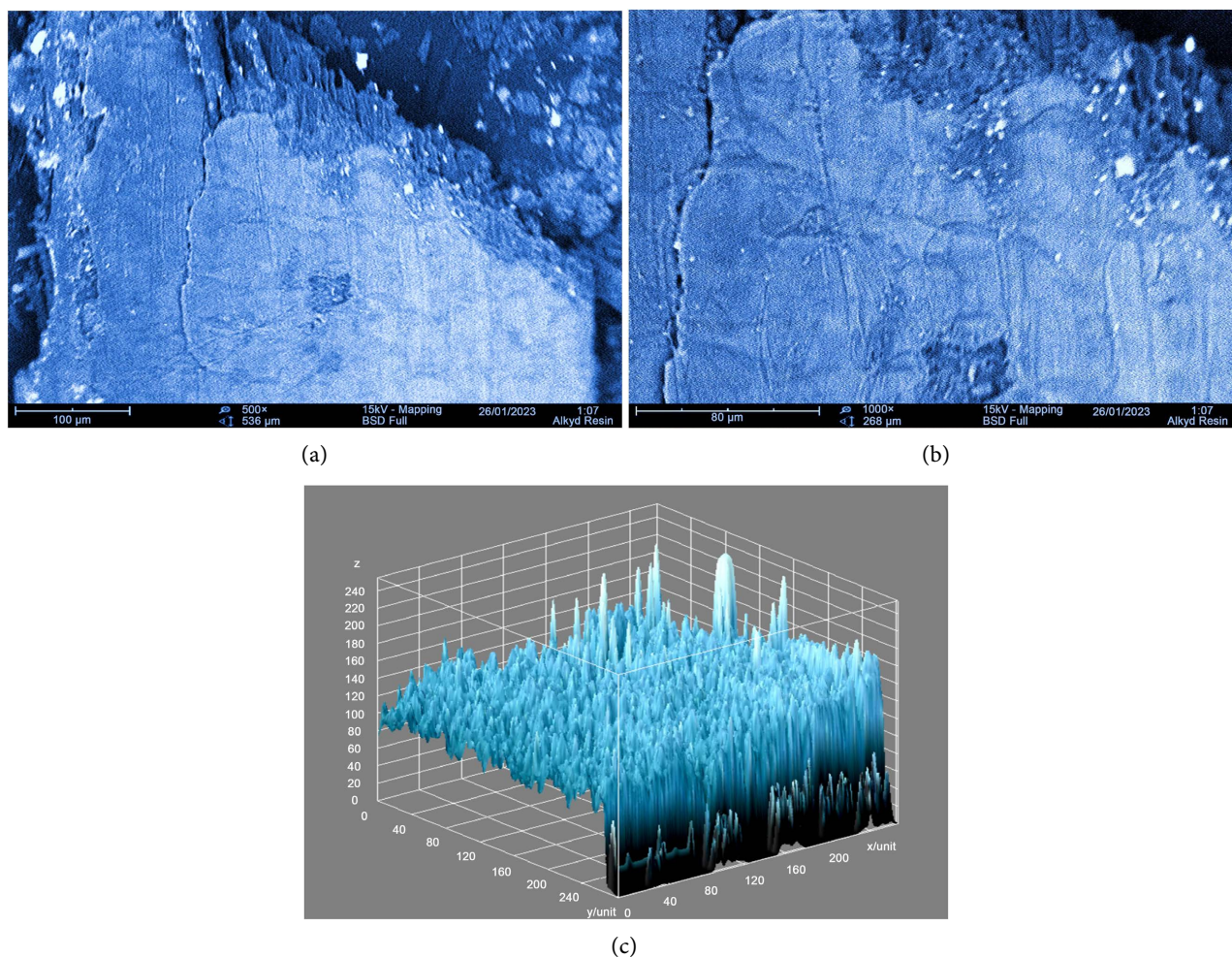
(a)

(b)

(c)

Figure 7. Scanning electron microscope micrograph showing surface morphology of 40% alkyd resin (a), its magnification (b), and 3D interfacial plot of the polymer matrix (c).
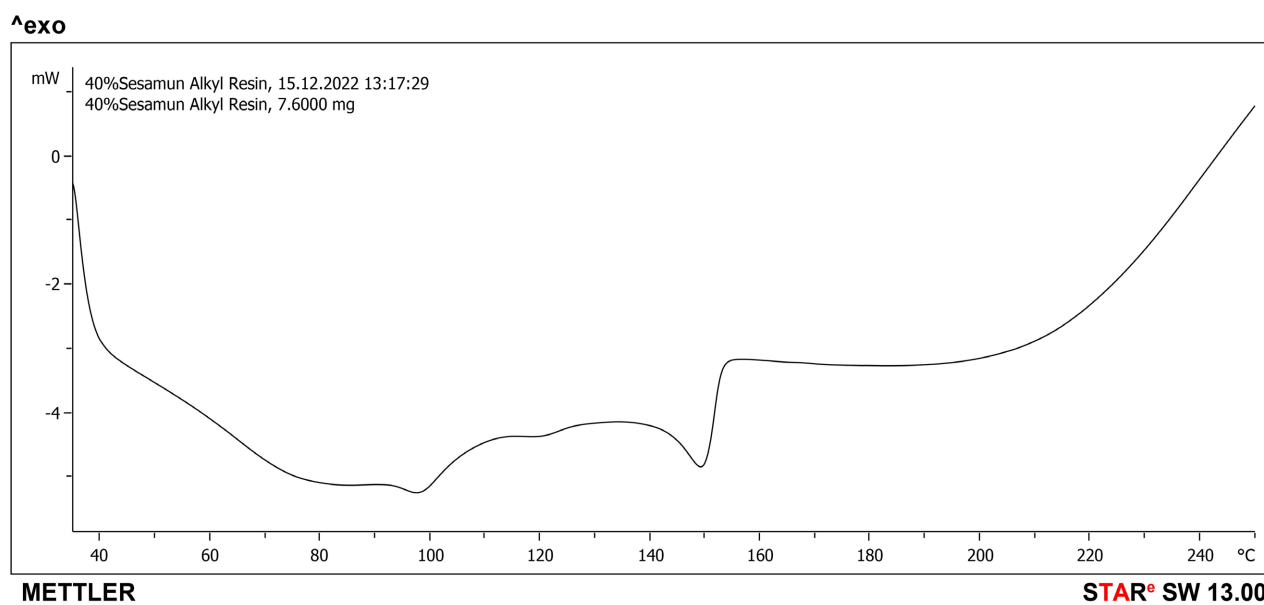

Figure 8. Differential thermal analysis of the 40% alkyd resin.

requires that less heat is needed to maintain a constant heating rate of the sample with the reference and as a result, it is observed as the lowest point of the dip. At higher temperatures, crystallinity is distorted reaching the melting temperature (Tm), since melting is an endothermic process, the heat is increased to maintain the heating rate of the sample constant with the reference resulting in a discontinuity in the plot, and it is considered as the temperature at the peak.

3.6.4. Fourier Transform Infra-Red Spectrophotometric Analysis of the 40% Alkyd Resin

FT-IR – 8400S Fourier Transform Infrared spectrophotometer was used to detect the functional groups of the ester links present from the poly-condensation reaction between phthalic acid and glycerol modified with sesame seed oil.

It can be observed from Figure 9 that the weak broad peaks at 3454.21 suggest the presence of a carboxylic –OH stretch present in the SISO modified alkyd resin samples. The medium peaks found at 2933.51 cm^{-1} suggest the presence of an alkane C-H stretching in the SISO modified alkyd resin. The less intense peaks at 2682.59 cm^{-1} infer the presence of an asymmetric stretch of the carbonyl groups present in the SISO modified alkyd resin. The peak at 2290.18 cm^{-1} could be attributed to the C=O vibration of carboxylic and carbonyl compounds present. The medium peaks found at 1454.38 cm^{-1} indicate the presence of a –CH bending of the methyl groups. The medium peaks at 1742.55 cm^{-1} show the presence of a carbonyl group which could be attributed to an aromatic ester C-O stretch present in the SISO modified alkyd resin.

The weak band at 1585.93 cm^{-1} indicates the presence of a C=C stretching for aromatics and some in-plane and out-plane stretching vibrations were recorded at 726.15 cm^{-1} which identifies the presence of the –CH$_2$ rocking mode.

4. Conclusions

The suitability of Sesame seed oil in the preparation of alkyd resin was investigated. The physicochemical characterization of the oil indicated it is semi-drying based on the iodine value of 122 g I$_2$/100 g which connotes its suitability for alkyd resin synthesis. Three grades of alkyd resin were formulated via the alcoholysis-polyesterification method, and the kinetics of the reaction show it is of the second order which was evident from the plot of the degree of polymerization with reaction time. Some features such as the decrease in the iodine values of the alkyd resin grades indicated the extent to which polymerization had taken place across the unsaturated bonds of the alkyd matrix, resulting in the higher saponification values which is the average molecular weight of all free-fatty acids present in the alkyd resin grades.

The variations in acid value, change in volume of water evolved, extent of reaction, degree of polymerization and weight average molecular weight with reaction time and variations in average acid value with degree of polymerization were studied for the in-situ alkyd resin synthesis. The extent of reaction and the corresponding degree of polymerization determined revealed the formation of
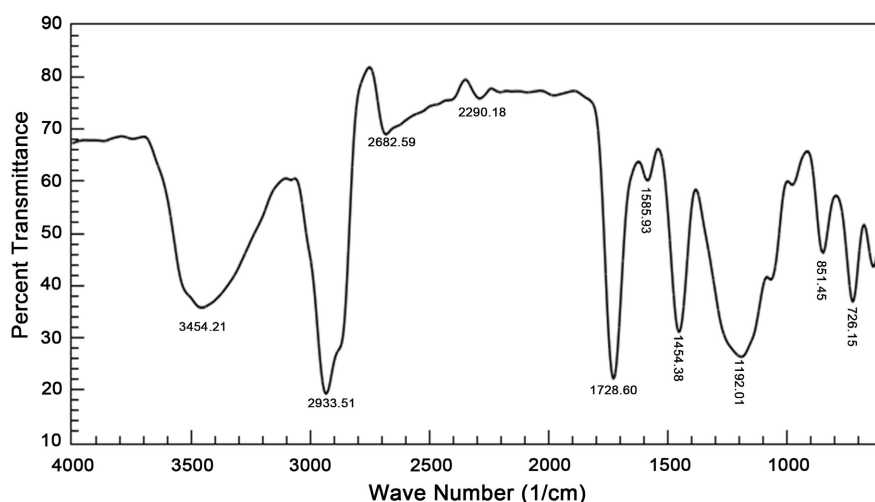
Figure 9. FT-IR spectra of the 40% alkyd resin sample.

high molecular weight alkyd which decreased with an increase in the oil length owing to a higher chance of chain growth termination as a result of the increase in the fatty acids in 50% and 60% alkyds. The alkyd resins exhibited good resistance to acid, brine and water, but offered fair resistance to alkali which is linked to the susceptibility of undergoing alkaline hydrolysis. The 40% alkyd resin oil length exhibited better moisture uptake percent, higher degree of polymerization, higher viscosity and higher molecular weight alkyd formation which suggests the possibility of drying within a short period of time if applied in the formulation of surface coating.

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 paper.

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