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## New Alkyd Resins from Underutilized Indigenous Seed Oils: Synthesis and Characterization

Aliru O. Mustapha

Department of Chemistry and Industrial Chemistry, Faculty of Pure and Applied Sciences, Kwara State University Malete, Kwara State, Nigeria, aliru.mustapha@kwasu.edu.ng

Hakeem B. Ayoku Department of Chemistry and Industrial Chemistry, Faculty of Pure and Applied Sciences, Kwara State University Malete, Kwara State, Nigeria

Halimah A. Amao Department of Chemistry and Industrial Chemistry, Faculty of Pure and Applied Sciences, Kwara State University Malete, Kwara State, Nigeria

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### Keywords

alkyd resin; sesame oil, jatropha oil; sweet almond oil; physicochemical

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### **RESEARCH PAPER**

### New Alkyd Resins From Underutilized Indigenous Seed Oils: Synthesis and Characterization

Aliru O. Mustapha\*, Hakeem B. Ayoku, Halimah A. Amao

Department of Chemistry and Industrial Chemistry, Faculty of Pure and Applied Sciences, Kwara State University Malete, Kwara State, Nigeria

#### Abstract

The underutilization of sesame (*Sesamum indicum L.*), sweet almond (*Prunus amygdalus*), and jatropha (*Jatropha curcas*) seeds indicates a significant resource waste. Nine different varieties of 25% (short), 40% (medium), and 60% (long) alkyd resins were produced from the cross-section of drying, semi-drying, and non-drying oils. A large amount of conversion occurred, with the extent of the reaction (Pav) decreasing as time progressed and the corresponding increase in alkyds' average degree of polymerization (Dp) showing synthesized alkyds. Surface drying from the set-to-dry and dry-through periods climaxed at 2 h for all the alkyds. Except for alkali, synthesized alkyd resins demonstrated outstanding resistance to service media as surface coatings, but the sesame resins gave excellent film qualities equivalent to those of a commercial and standard oil-based paint.

Keywords: Alkyd resin, Sesame oil, Jatropha oil, Sweet almond oil, Physicochemical

### 1. Introduction

T he ever-increasing cost of adopting petroleumbased monomers in the production of surface coatings calls for the shift to more environmentally friendly and sustainable alternatives [1]. Besides, there is a potential danger from the depletion of petroleum resources. The scarcity of petroleum products, combined with finite petroleum reserves and volatile prices, makes future availability and supply uncertain, as studied by Choe et al. [2].

As a result, producing alkyds requires a significant amount of oil. In order to produce alkyd resins from local oilseeds with qualities similar to those currently imported for paint formulation, process variables must be optimized. Unsaturated fatty acid-based alkyd resins from non-edible oilseeds may exhibit low inherent viscosity, making them suitable for use in high-solid alkyd paints and promoting economic growth and reducing capital flight as a result of the importation of alkyd resin. A synthetic polymer with useful reactive, film-forming, or adhesive capabilities is known as an industrial resin, as reported in few studies [3,4]. Alkyd resins are useful and environmentally friendly adhesives for surface coating.

According to Mohammed et al. [5], the cost of fundamental raw materials used in the production of alkyd resin has risen over time. As a result, the cost of finished coatings such as paints, varnishes, and other such things skyrocketed. The search for alternative raw materials for the manufacture of alkyd resin has increased in the surface coating business in the face of intense competition from the use of low-cost synthetic resins.

The previous works of Mustapha et al. [6,7] discovered the development of renewable resources from underutilized seed oils that could serve as viable substitutes for petroleum-based alkyd resin products. This has reawakened interest in non-edible vegetable oils since they are not consumed by people but could be used for similar purpose as reported in related studies [8,9].

The majority of alkyd resins used to produce paints, varnishes, and other surface coatings are

\* Corresponding author. E-mail address: aliru.mustapha@kwasu.edu.ng (A.O. Mustapha).

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imported. Finding a local substitute for imported resins will likely lower both environmental and manufacturing costs as reported by Basha et al. [10]. The plants can thrive on agronomically inferior land, making the alkyd production cost lower than that of other energy crops. The ability to make alkyd resin from non-edible seed oils will expand the oil's use as an environmentally beneficial material in a variety of applications, as the oils are one of the least expensive and most renewable resources.

This current work looks at making nine varieties of alkyd resins from the combination of short, medium, and long-drying, semi-drying, and non-drying oils; analyzing their physicochemical properties; and comparing them to commercially available resins. It was to further test if alkyd resins created from locally available materials such as sesame (*Sesamum indicum L*), sweet almond (*Prunus amygdalus*), and jatropha (*Jatropha curcas*) seeds could meet recommended property standards of other imported alkyd resins.

### 2. Materials and reagents

The following apparatus and equipment were acquired: an Oswald viscometer; a reflux condenser equipped with a three-neck flask; a pH meter, Jenway model 3320; suction filtration apparatus; and a FTIR spectrophotometer. Reagents such as phthalic anhydride, glycerol, xylene, marble chips (CaCO<sub>3</sub>), and oxygen scavengers (antioxidants) [9].

The crude sesame oil, CSEO; sweet almond oil, CSAO; and jatropha oil, CJAO, were extracted from the seeds of sesame (*S. indicum* L); sweet almond (*Prunusamyg dalus*); and jatropha (*Jatropha curcas*), respectively, after collection in various farms and markets in Ilorin, Kwara State, Nigeria. The n-hexane oil extraction was regarded as total oil production and kept in a -20 °C freezer. After removing impurities such as phosphorus compounds and waxes, the refinement of crude oils, which included degumming, alkaline treatment, and bleaching, produced refined sesame oil, RSEO; sweet almond

oil, RSAO; and jatropha oil, RJAO, followed by the production of different alkyd resins [9,10].

The various proportions of materials used in the synthesis of various alkyd resins were derived from refined sesame (*S. indicum L*), sweet almond (*P. amygdalus*), and jatropha (*Jatropha curcas*) seed oils. The nine different types of alkyd resin (short, medium, and long) were created from a cross-selection of these drying, semi-drying, and non-drying oils (Table 1).

### 2.1. Alcoholysis and esterification

With a few minor adjustments, Mustapha et al. [11] and Wicks et al. [12] used the monoglyceride method of synthesis of alkyd resin. After assembling the equipment, the reactor for long alkyd resin (R-SOAR), for example, was charged with 60 g of oil and heated using a heating mantle. The heated oil was first treated with 0.24 g of CaCO<sub>3</sub> (catalyst) and agitated. Next, a determined amount of glycerol was added, and the temperature was raised to 230–250 °C (Table 1). A small amount of the aliquot was obtained after 30 min to test its solubility in methanol. When a sample of the produced mixture became soluble in one to three liters of anhydrous methanol and formed a clear solution, alcoholysis was complete. The reaction temperature was then lowered to 140 °C in order to smoothly introduce the esterification stage. In the esterification procedure, phthalic anhydride and xylene were added in calibrated amounts to the flask containing the monoglyceride mixture.

The mixture was heated using a heating mantle while being constantly stirred. The temperature was raised and kept between 230 and 250 °C. The reaction mixture's acid value was monitored on a regular basis to monitor the reaction. As soon as the mixture's acid value fell to 10 mg KOH/g or below, the reaction was stopped [13].

The material was further refined by swirling it for many hours with tetrahydrofuran (THF) after an initial washing with cold ethanol and diethyl ether.

Table 1. Recipes for making alkyd resins.

Ingredients (g)	R-SOAR			R-SAOA	.R		R-JOAR		
	Long alkyd	Medium alkyd	Short alkyd	Long alkyd	Medium alkyd	Short alkyd	Long alkyd	Medium alkyd	Short alkyd
Seed oil	60	40	25	60	40	25	60	40	25
Glycerol	12.3	8.2	5.1	10.8	7.2	4.5	11.1	7.4	4.6
Phthalic Anhydride	29.6	19.7	12.3	25.9	17.3	10.8	26.7	17.9	11.1
Catalyst	0.24	0.16	0.1	0.24	0.16	0.1	0.24	0.16	0.1
Xylene	5	4	2	5	3	2	5	3	2

The acid values indicated the completion of the alcoholysis reaction, and the solid was finally dried under a vacuum. The techniques of the Association of Officials of Analytical Chemists (AOAC, 1990) were used to examine the physicochemical characteristics of the long alkyd resin [14]. Fourier transform infrared spectroscopy (FTIR) structural analysis was used to monitor the functional group changes as the syntheses progressed. The spectroscopic-grade potassium bromide, KBr, was ground into a powder, pelletized, and scanned. After that, KBr was added to the alkyd sample, and a hydraulic press was used to form pellets. After inserting the pellets, the device was then scanned at 400–400 cm<sup>-1</sup> frequency using Shimadzu equipment.

#### 2.2. Performance characteristics

Physical surface coating qualities of synthetic alkyds, such as hardness, impact resistance, abrasion resistance, flexibility, gloss, adhesion, etc., were measured using appropriate ASTM standards. The drying time was determined by "set-to-touch," "surface dry," and "dry through" stages at regular intervals of time according to ASTM D 1640 (1995). The abrasion tests were performed in accordance with ASTM-D-1242-95 (Tabers' method) [15]. The hardness tests were performed when the resins were applied to glass plates and allowed to dry for one week after application, according to ASTM D 4366 (1997). The cross-hatch adhesion test was performed on the coated steel plates after one week of application according to ASTM D 3359 (1997). The flexibility of the dried films was evaluated using the mandrel test (ASTM D 1737). The ASTM D 2794 falling weight impact technique was used to assess the impact resistance following a week of film application. The gloss test was validated using ASTM D-523 (1989). After the resin coating, each of the films had its sixty-degree gloss measured. The chemical resistance tests were carried out in accordance with ASTM D 1647 (1996), D870 (1997), and D1308 (1998) standards. Investigated were the effects of acid, brine, water, and alkali on the alkyd resin. To guarantee the absence of contaminants,

mild steel sheets  $(3 \text{ cm} \times 3 \text{ cm} \times 1 \text{ mm})$  were cleaned with ethanol. Test samples were applied to them, and replicates were submerged in distinct batches of distilled water, diluted with alkali (0.1 M NaOH), diluted acid (0.1 M H2SO4), and brine (5% w/w NaCl). After 30 min, the metal sheets were taken out and their chemical resistance was tested. Four different solvents (methanol, xylene, ethanol, and nhexane) were used to assess the solubilities of the generated alkyd resins. Conical flasks were filled with 5 g of each sample; 5 ml of each of the solvents were separately added and briskly agitated [6].

### 3. Results and discussion

The physical and chemical properties of the alkyd samples produced by the reaction of the individual seed oils with phthalic anhydride, glycerol, and glycerol are listed in Table 2. Their physico-chemical attributes were contrasted with those of the alkyd that had been manufactured commercially.

The alkyds are dark brown when compared to the equivalent precursor oil and commercially produced alkyd (which is colorless). The catalyst, high reaction temperatures, and oxidation all contributed to the alkyd's darkening hue. In order to produce castor seed oil alkyd resin, Uzoh and Nwabanne [16] looked at the impact of catalyst type and concentration on functional group conversion. Oil was alcoholized using 0.1% (by weight) of the catalyst (LiOH, CaO, CaCO3, PbO, and NaOH). LiOH was reported as the catalyst that completed the alcoholysis reaction the quickest, followed by PbO, CaCO3, NaOH, and CaO. The use of CaCO3 in this work as the ideal catalyst in the synthesis of alkyd resin may have significant advantages for availability and the economy [15,17]. The test alkyd resins' specific gravity and viscosity at 30 °C were discovered to be comparatively high when compared to comparable values found in the literature (Table 3). This might be due to higher molecular weights during the formation of alkyds at the unsaturation of the oil's reactive double bonds [18,19]. However, at 30 °C, the test alkyd resins' specific gravities (1.060, 0.995, and 1.0825) and viscosities (21.96, 23.825, and 29.795 m<sup>2</sup>/s) were not

Table 2. Sesame, sweet almond, and jatropha alkyd resins' physical and chemical characteristics.

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Parameters/units	LSEOAR	LSAOAR	LJOAR	CPAR
Colour	Brown	Brown	Brown	Colourless.
Specific Gravity (30 °C)	$1.060 \pm 0.014$	$0.995 \pm 0.004$	$1.0825 \pm 0.01$	47.40
Viscosity (30 °C, m <sup>2</sup> /s)	$21.96 \pm 0.64$	$23.825 \pm 0.42$	$29.795 \pm 0.31$	32.34
Saponification Value (mg/KOH/100g)	$226.876 \pm 3.45$	$246.845 \pm 3.91$	$256.29 \pm 5.38$	263.97
Iodine Value (gI <sub>2</sub> /100 g)	$84.065 \pm 1.05$	$70.96 \pm 3.89$	$65.015 \pm 4.41$	78.22
Acid Value (mg/KOH/g)	$6.02 \pm 0.028$	$7.230\pm0.28$	$6.565 \pm 4.11$	6.31

Table 3. Physicochemical properties of alkyd resins produced from a variety of vegetable oils.

Parameters/units	JOAR [22]	SEOAR [23]	CAOAR [13]	LSOAR [24]
Colour	Dark brown	Gardner	Dark Brown	Colourless
Specific Gravity (30 °C)	_	_	_	-
Viscosity (30 °C, m <sup>2</sup> /s)	33.0	1.40	_	90.0
Saponification Value (mg/KOH/100g)	383.0	_	812.04	187.0
Iodine Value (gI <sub>2</sub> /100 g)	_	_	8.883	156.0
Acid Value (mg/KOH/g)	23.0	8.6	7.0	9.81

Note: jathropha oil alkyd resin (JOAR); sesame oil alkyd resin (SEOAR); castor oil alkyd resin (CAOAR) and sunflower oil alkyd.

statistically different from those of the commercially manufactured CPAR alkyd resins (47.40 and 32.34 m<sup>2</sup>/s), respectively [20,21].

The saponification values of SOAR, SAOAR, and JOAR were found to be lower than those of seed oils RSO, RSAO, and RJO (168.37, 165.52, and 171.136 mg KOH/g, respectively) and commercially manufactured alkyds (263.97 mg/KOH/g). It follows from this that seed oils could also play a crucial role in the production of soap and ink [20,21]. The acidity of the alkyd resins created from the RSO, RSAO, and RJO oil samples was 6.02, 7.230, and 6.565 mg KOH/g, respectively, which is equivalent to the acid value of 6.31 mg KOH/g reported in commercially produced alkyd resin [22,23]. The acid levels (5.61-8.6 mg KOH/ g) of the alkyd resins were in line with those [24,25] noted in the literature. The alkyd's chemical resistance was also significantly impacted by the amount of acidity in its composition [26]. Empirical comparisons between the properties of resins in this study and similar others as found in the work of Egbuna and Aninwede [14] are shown in Table 4. In the correlated studies of Ikhuoria et al. [27], they demonstrated that the properties of these resins, such as density, iodine number, saponification number, and acid value, depend on the type of vegetable oil used. The effect of a geographical component (soil type) on the seeds is likely to be responsible for the variations in the values of the oil characteristics.

# 3.1. The acid value (AV), viscosity, extent of the reaction (Re), and degree of polymerization (Dp) of samples in progress over time

Sesame, sweet almond, and jatropha oil alkyd resin in-process samples' AV, viscosity, Re, and Dp



Fig. 1. Change in acid values as a function of reaction time.

changed with time. At each time point, the acid values of the mixture were measured to ensure that the polycondensation process was operating as intended. The samples' increasing acid value was caused by the reaction progressing (Fig. 1). According to Fig. 1, this demonstrated a sharp initial reduction in acid value with a consistent decline, as previously studied [25,27].

In the meantime, a rapid rise in temperature to 230 °C coincides with the previously noted considerable decrease for all alkyd samples caused by an increase in acid value [28]. The molecular weight-related rise in resin viscosity and the evaluation of acid value were employed to control the reaction's progress (Fig. 2). Alkyd resin samples that were still in the reaction process showed similar increase in reaction size and degree of polymerization with reaction time, though at differing rates (Figs. 4 and 5) as previously reported [29,30] (see Fig. 3).

Table 4. Chemical resistance properties of the alkyd resins.

			5							
Medium	LSOAR	MSOAR	SSOAR	LSAOAR	MSAOAR	SSAOAR	LJOAR	MJOAR	SJOAR	Soyabean-based resin [14]
Distilled Water	FA	FA	FA	FA	FA	FA	FA	FA	FA	FA
NaOH 0.1M	FD	FD	FD	FD	FD	FD	FD	FD	FD	FD
H <sub>2</sub> SO <sub>4</sub> 0.1M	FA	FA	FA	FA	FA	FA	FA	FA	FA	FA
5%w/w NaCl	FA	FA	FA	FA	FA	FA	FA	FA	FA	FA

Film Appeared (FA), Film Disappeared (FD).



Fig. 2. Alkyd resin viscosity changes with reaction time.



Fig. 3. Acid value of in-process samples versus reaction extent (%PaV).



Fig. 4. Alkyd resin polymerization degree versus reaction time.

### 3.2. Performance characteristics of the alkyd resins

### 3.2.1. Chemical resistance properties

The film's characteristics result from testing samples of alkyd resin for resistance in various solvent media (Table 4). Alkyd samples were resistant to alkalis but not to water, acids, or solvents. This was expected as the alkali readily dissolves polyesters. The films were found, during a period of immersion, to be very resistant to an aqueous sodium chloride solution and distilled water [31].

### 3.2.2. Coating characteristics of the alkyds

The results are shown in Table 5, as the set-totouch and dry-through periods of the alkyd samples were affected by the type of polybasic acid employed in the alkyd formulation. The drying sequence of

### LSOAR, LSAOAR, and LJOAR resins>

MSOAR, MSAOAR, and MJOAR resins>

SSOAR, SSAOAR, and SJOAR resins correspond to long, medium, and short resins, respectively. This result is consistent with that of [14,20], who found that the drying time increased with the level of unsaturation.

### 3.2.3. Properties of alkyd resins' solubility

The solubility of the nine recently created oilbased alkyd resins is displayed in Table 6. With the exception of n-hexane, the alkyds were not soluble in methanol, ethanol, or xylene. The synthesized alkyd resins showed good overall resistance to xylene and alcohol. All of the created alkyd resins failed the acetone resistance test, indicating that alkyds usually have low acetone resistance. The low acetone resistance of the alkyds may be explained by the presence of ester groups, which are known to be hydrolyzable due to acetone's aprotic character. The impact of alkyd oil length type had no impact on the media resistance of the alkyd resins under study.

### 3.2.4. Alkyd resins' characteristics

Higher oil concentrations in medium and long alkyds initially dry slowly; the thermosetting temperature of long oil alkyds was higher than that of short oil alkyds, making the long oil alkyds retain gloss for a longer period of time than medium and short oil alkyds [32,33]. The outcomes demonstrated the desired adhesion for resins made from the same three oils that produced the identical polyester component that was in charge of adhesion in Table 7. The findings for impact resistance are presented similarly in Table 7, and nine newly created resins exhibit a considerable increase in impact resistance over the commercial resin. Table 7 presents the gloss test results, and the prepared resins' gloss ratings indicate that they contain a component that maintains gloss. Table 7 shows that the cross-link density of the surface influences the film hardness, with hardness values that are close to one another. Results of the flexibility test



Fig. 5. (a) FTIR spectral of RSO. (b) FTIR spectral of RSAO. (c) FTIR spectral of RJO. (d) FTIR spectral of LSOAR. (e) FTIR spectral of MSOAR. (f) FTIR spectral of SSOAR. (g) FTIR spectral of LSAOAR. (h) FTIR spectral of MSAOAR. (i) FTIR spectral of SSAOAR. (j) FTIR spectral of LJOAR. (k) FTIR spectral of MJOAR. (l) FTIR spectral of SJOAR.

Table 5. Coating prope	erties of alk	yd resin.								
Coating Property	LSOAR	MSOAR	SSOAR	LSAOAR	MSAOAR	SSAOAR	LJOAR	MJOAR	SJOAR	Rubber-based resin [32]
Dry-Through (min)	90	55	45	120	60	30	105	85	60	372
Set to touch (min)	18	14	10	09	06	04	16	10	03	_
Surface-dry (min)	60	42	25	45	30	20	50	35	15	186

### Table 6. Alkyd resins solubility in solvents.

Medium	LSOAR	MSOAR	SSOAR	LSAOAR	MSAOAR	SSAOAR	LJOAR	MJOAR	SJRAR	Castor-based resin <sup>a</sup>	Soya bean-based resin [14]
n-Hexane	+	+	+	+	+	+	+	+	+	±	±
Ethanol	±	±	±	±	±	±	±	±	±	_	-
Methanol	±	±	±	±	±	±	±	±	±	+	+
Xylene	_	_	_	_	_	_	_	_	_	+	+

Soluble (+), sparing soluble (–) and partial soluble ( $\pm$ ).

Table 7. Physical film properties of alkyd resins and commercial alkyd resin.

Properties	LSOAR	MSO AR	SSO AR	LSAO AR	MSAO AR	SSAO AR	LJO AR	MJO AR	SJO AR	Commercial alkyd resins [33]
Adhesion (%)	100	100	100	100	100	100	100	100	100	100
Hardness	Н	Н	2H	Н	Н	2H	Н	Н	2H	3H
Flexibility (mm)	1.5	1.5	2.0	1.5	1.5	2.0	1.5	1.5	2.0	3.0
Gloss, 60°	93.4	92.3	85.4	94.1	91.4	84.6	94.3	92.4	86.6	78.2
Abrasion resistance (l/µm)	14.26	9.90	8.60	11.55	9.12	8.92	12.28	9.80	8.46	_
Impact resistance (cm/kg)	50	50	120	50	50	128	50	50	125	50

are shown in Table 7. Nine samples passed the test with no peeling or cracking visible, and the oil quality [34,35] in the produced alkyd resins may be the cause of this. Table 7 displays the impact resistance data compared to the commercial standard. The impact resistance significantly improves. Table 7 presents the gloss test results, and it was observed that the polyester component that was responsible for gloss retention was present in the gloss values of the prepared resins. The cross-linking density of the film's surface determines the toughness of the film. The coating was made of polymers, and one of their weaknesses was their lackluster resistance to wear. The alkyd's ability to protect a substrate against abrasion, wear, and other mechanical actions were compared with commercial alkyd resins [36] using the ASTM-D-1242-95 standard (Tabers' approach) [15].

### 3.2.5. Analysis of fourier transform infrared spectroscopy

Fig. 5(a–l) of the Fourier transform infrared (FTIR) spectrum show the long, medium, and short alkyd resins produced from RSO, RSAO, and RJO. When figuring out the functional groups of organic compounds contained in any sample, identification of the typical functional groups from the spectral data were achieved using the studies in the literature [36,37].

### 3.2.6. FTIR spectroscopy of RSO, RSAO, and RJO and corresponding alkyd reins

The FTIR spectra and peaks of interest for the refined samples of oil are shown in Fig. 5(a–c) for RSO, RSAO, and RJO in the Appendix. According to the figures, alcohol-induced O–H stretching is represented by the absorption bands at 3541.69, 3576.14, and 3548.37 cm<sup>-1</sup>; aliphatic C–H stretching is represented by the peaks at 2910.69, 2915.14, and 2880.65 cm<sup>-1</sup>; C–O stretching of triglyceride esters is represented by the peaks at 1735.49, 1732.65, and 1742.85 cm<sup>-1</sup>; and aromatic C–C stretching is represented by the peaks at 1614.91 cm1 There were observed O–CH<sub>3</sub> bend values of 1148.62, 1151.53, and 1157.21 cm<sup>-1</sup>, respectively [6,26]. By using FT-IR

analysis, the polyesterification reactions were verified with the chemical functional groups of alkyd (LSOAR, MSOAR, SSOAR, LSAOAR, resins MSAOAR, SSAOAR, LJOAR, MJOAR, and SJOAR) shown in Fig. 5(d–l) in the Appendix. The characteristic peaks are discussed in a sequential manner since alkvd resin's structural similarities are evident and all spectra share similar peaks. The broad band shoulder was visible in the mixture's IR spectra at about 3595.63 cm<sup>-1</sup>. Unsaturated fatty acid O-H stretch hydroxyls in the combination were in the following values: 3507.88, 3451.66, 3576.91, 3595.63, 3584.32, 3437.97, 3582.13, 350.65, and 3541.69 cm<sup>-1</sup> in respective order. The C–H aliphatic stretching was represented by the measurements of 2909.09, 2912.81, 2912.62, 2919.69, 2915.64, 2922.59, 2922.59, 2926.45, and 2928.60 cm<sup>-1</sup>. The values of 1746.71, 1739.76, 1736.52, 1730.32, 1742.71, 1734.63, 1728.87, 1730.32, and 1730.18 cm<sup>-1</sup> were for CO stretching (ester). The  $O-CH_3$  bend was visible in the peaks at 1144.55, 1155.12, 1149.63, 1190.35, 1160.39, 1160.49, 1180.02, 1117.99, and 1171.55 cm<sup>-1</sup>. The unsaturated fatty acids' hydroxyl (OH) group was attributed to the wide bands near 3595.63 cm<sup>-1</sup>. Thiocyanate and nitrite compounds' SCN and CN stretches peaked at 2930.93  $\text{cm}^{-1}$  and 2353.23  $\text{cm}^{-1}$ , respectively. The C-O stretch of monoglyceride cyclic ester compounds was compared to the medium bands at values of about 1012.75, 1018.62, 1015.63, 1160.78, 1155.62, 1150.63, 1013.42, 1017.88, and 1021.55 cm<sup>-</sup> The O-CH<sub>2</sub> bend has further substantial peaks of 1370.66, 1372.21, 1385.36, 1374.03, 1372.13, 1381.05, 1388.01, 1374.99, and 1379.56 cm<sup>-1</sup>. Peaks for RCHCHR trans were 972.43, 975.61, 974.16, 970.98, 973.81, 975.89, 970.02, 972.91, and 978.62 cm<sup>-1</sup>, with values for RCHCHR cis being 685.08, 680.62, 683.19, 670.65, 686.16, 690.18, 698.59, 673.52, and 681.92 cm1. The C-Cl bends were detected at 670.20, 660.39, 641.12, 642.66, 631.72, 612.70, 674.20, 652.98, and 671.65 cm<sup>-1</sup>, whereas the C-1 bends were observed at 526.47, 532.11, 538.76, 549.13, 530.16, 499.47, 516.25, and 502.61  $\text{cm}^{-1}$  [38], proving that various vegetable oils were used to manufacture all long oil resins (Tables 8 and 9). Significant variations in alkyd

Table 8. Comparison of the interpretation of refined oils FTIR spectra with a commercial alkyd resin.

Observed vibration	Functional	Refined vegetab	le oils	
band/peaks Frequency (cm- <sup>1</sup> )*	Groups (cm <sup>-1</sup> )	RSO	RSAO	RJO
O–H stretching	3600-3200	3541.69	3576.14	3548.37
C–H aliphatic stretching	2950-2840	2910.69	2915.14	2881.45
C=O stretching (ester)	1750-1720	1735.49	1732.65	1742.85
-CH <sub>3</sub> bending	1465 - 1440	1614.91	1639.91	1635.85
-OCH <sub>3</sub> vibration	1390-742	1453.49	1456.17	145163
C–O bend	1035-1149	1148.62	1151.33	1157.21

Table 9. Comparison of the in	tterpretation of alkyd	resin FTIR spt	ectra with a con	mmercial alky	d resin.						
Observed vibration band/peaks	Functional Groups (cm- <sup>1</sup> )	Sesame m alkyd resii	odified ns		Sweet almo alkyd resins	nd modified		Jathropa n alkyd resir	nodified ns		CPARL*
Frequency (cm-¹)*		LSOAR	MSOAR	SSOAR	LSAOAR	MSAOAR	SSAOAR	LJOAR	MJOAR	SJOAR	
O-H stretching	3600-3200	3507.88	3451.66	3576.91	3595.63	3584.32	3437.97	3582.13	3500.65	3541.69	3500.92
C-H aliphatic stretching	2950 - 2840	2909.09	2912.81	2912.62	2919.69	2515.64	2922.59	2922.59	2926.45	2928.60	2880.78
C=O stretching (ester)	1750 - 1720	1746.71	1739.76	1736.52	1730.32	1742.71	1734.63	1728.87	1730.32	1730.18	1727.31
-CH <sub>3</sub> bending	1465 - 1440	1452.14	1454.69	1456.90	1457.44	1460.64	1455.99	1455.99	1457.44	1460.72	1452.45
-OCH <sub>3</sub> vibration	1390 - 742	1370.66	1372.21	1385.36	1374.03	1372.13	1381.05	1388.01	1374.99	1379.56	Ι
C–O bend	1035 - 1149	1021.75	1018.62	1015.63	1160.78	1155.62	1150.63	1013.42	1017.88	1021.05	1131.29
<b>RCHCHR-trans</b>	980—965	972.43	975.61	974.16	970.98	973.81	975.89	970.02	972.91	978.62	976.25
RCHCHR-cis	730 - 650	685.08	680.62	683.19	670.65	686.16	690.18	698.59	673.52	681.92	681.50
C-CI	730 - 500	570.20	660.37	641.12	642.66	631.72	612.70	674.20	652.98	671.65	720.50
C–I	620 - 490	526.47	532.11	538.76	549.13	546.19	530.16	499.47	516.25	502.41	449.43
Note: *CPAR-Commercial	alkvd resin.										

compositions led to chemical shifts on a few FTIR peaks [39]. This could be due to the varying physicochemical compositions of different vegetable oils.

### 4. Conclusion

In this study, the oils from the seeds of sesame (S. indicum L.), sweet almond (P. amygdalus), and jatropha (Jatropha curcas) were extracted, with their extracted oilseed yields observed to be 62.25, 72.50, and 53.50% (w/w), respectively, showing that the oilseeds could be categorized as good sources. It was discovered that the physiochemical properties of the oil and the ASTM standards were in good accord, allowing for the synthesis of alkyd resin with different oil concentrations (short, medium, and long alkyd resins). A cross-selection of drying, semidrying, and non-drying oils based on the iodine value was used to make the selection for the synthesis of the alkyd resins with varied qualities.

The nine classes of alkyd resins were generated using the alcoholysis-polyesterification processes, and the traits of each class were suitably identified. Calculating the reaction volume and the associated average degree of polymerization (Dp) allowed the synthesis of a high molecular weight alkyd to be identified. From three underused seed oils, alkyd resins with 25, 40, and 60% oil content were created. According to the characteristics of the generated alkyd resins, the surface drying time for the 60% oil proportion had the longest length of less than two hours. Except for alkali, the nine manufactured alkyd resins demonstrated resistance to acid, brine, and water. The alkyd resins derived from the three different underutilized seed oils have varying yields and physical film qualities. According to the physical and chemical properties of the oils and the performance evaluations of the alkyd films, they are practical and environmentally benign adhesives for surface coating. Based on the resistance to service media and other physicochemical properties comparable to those of typical oil-based paint, sesamederived alkyd resins, followed by jatropha, have proved to be great materials for conventional oilbased paints. The research was on the development of alkyd resins from indigenous resources by optimizing different input parameters to produce lowcost alkyd resins of different grades with appreciable yields and better qualities compared to others in the literature that had been earlier published and commercial standards. The variety of vegetable oils utilized in the alkyd resin business and the complexity of the new alkyd resins' architectures determine their qualities. The novel alkyd resins created in this work include qualities including high

adhesion, hardness, solubility, impact resistance, abrasion resistance, better gloss quality, high flexibility, and high dry through without the addition of a drying agent that make them useful in many industries like paints and surface coatings.

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#### References

- M.D. Joshi, R.A. Patil, Y.-R. Ma, R.S. Devan, S.S. Hosmani, Reciprocating wear behavior of noncoated and polymer/ composite coated AISI 316L steel: role of surface mechanical attrition treatment, Tribol Trans 64 (5) (2021) 916–935. https://doi:10.1080/10402004.2021.1950884.
- [2] Y.-A. Choe, S.-I. Kim, K.-S. Ju, Synthesis and characterizations of high oil length alkyd resin with dehydrated castor oil and acrylic pimaric acid, Chem Phys Lett 805 (2022) 139934–139939. https://doi:10.1016/j.cplett.2022.139934.
- [3] M. Jonas, C. Ketlogetswe, J. Gandure, Variation of Jatropha curcas seed oil content and fatty acid composition with fruit maturity stage, Heliyon 6 (2020) e03285, 1-6, https://doi:10. 1016/j.heliyon.2020.e03285.
- [4] C.F. Úzoh, N.J. Obodo, O.D. Onukwuli, Exploring the effect of styrene and anhydride ratio on the coating properties of non-dring vegetable oil based alkyd resin, J King Saud Univ Eng Sci 30 (1) (2018) 12–21.
- [5] A.M. Mohammed, M. Noor, H. Abdul, Synthesis and characterization of new alkyd resins (short, medium and long) based on sunflower oil and linoleic acid as binder for paints. From T. Peru-Viana seed, Int J Chem Petrochem Technol 7 (1) (2017) 1–16.
- [6] A.O. Mustapha, S.F. Adisa, S.G. Oladele, Y.T. Afolabi, Synthesis and evaluation of alkyd resins from underutilized (Delonix regia and theventia peruviana) seed oils, Iraqi J Industr Res 9 (1) (2022) 57–74. https://doi:10.53523/ ijoirvol9i1id148.
- [7] S.Y. Adeyinka, Parametric optimization of solvent extraction of Jatropha curcas seed oil using design of experiment and its quality characterization, S Afr J Chem Eng 35 (2021) 60–68.
  [8] A.O. Mustapha, S.G. Oladele, S.F. Adïsa, Y.T. Afolabi,
- [8] A.O. Mustapha, S.G. Oladele, S.F. Adisa, Y.T. Afolabi, Response surface methodology to op-timize the yield of alkyd resin from jatropha (ja-tropha curcas) and sesame (sesamum indicum) seed oils, F?rat Univ Turk J Sci Technol 17 (2) (2022) 475–488. https://doi:10.55525/tjst.1090577.
- [9] D.B. Nde, A.C. Foncha, Optimization methods for the extraction of vegetable oils: a review, Processes 8 (2) (2020) 1-21. https://doi:10.3390/pr8020209.
- [10] J.S. Basha, T. Jafary, R. Vasudevan, J.K. Ba-hadur, M.A. Ajmi, A.A. Neyadi, M.E.M. Sou-dagar, M. Mujtaba, A. Hussain, W. Ahmed, K. Shahapurkar, S.M. Ashrafur Rahman, I.M. Rizwanul Fattah, Potential of utilization of renewable energy technologies in Gulf countries, Sustainability 13 (18) (2021) 1–29. https://doi:10.3390/su131810261.

- [11] A.O. Mustapha, T.A. Amodu, R.A. Adepoju, Effects of degumming waste cooking oil on the physicochemical and fuel properties of biodiesel, J Appl Sci Environ Manag 24 (5) (2020) 749–753. https://doi:10.4314/jasem.v24i5.3.
- [12] Z.W. Wicks Jr., F.N. Jones, S.P. Peppas, Organic coatings: science and technology volume 1: film formation, components and appearance, Dry, Technol 11 (6) (1993) 1477–1497, https://doi.org/10.1080/07373939308916913.
- [13] A.O. Mustapha, F.A. Saliu, G.O. Simeon, Y.T. Afolabi, Synthesis and evaluation of alkyd resins from underutilized (delonix regia and theventia peruviana) seed oils, Iraqi J Ind Res 9 (1) (2022) 57–74, https://doi.org/10.53523/ ijoirVol9I1ID148.
- [14] S.O. Egbuna, C.S. Aninwede, Comparative analysis of alkyd resins produced from castor and soybean seed oils, Int J Novel Res Eng Sci 5 (1) (2018) 24–32.
- [15] R. Rodriguez, S. Vargas, E. Rubio, S. Pacheco, M. Estevez, Abrasion properties of alkyd- and acrylic-based polymerceramic nano-hybrid coatings on wood surfaces, Mater Res Innovat 10 (2) (2006) 193–206, https://doi.org/10.1179/mri. 2006.10.2.193.
- [16] Official methods of analysis: 15th edition, Trends Anal Chem
   9 (4) (1990) 87098-87099. https://doi:10.1016/0165-9936(90) 87098-7.
- [17] C.F. Uzoh, J.T. Nwabanne, Investigating the effect of catalyst type and concentration on the functional group conversion in castor seed oil alkyd resin production, Adv Chem Eng Sci 6 (2) (2016) 190–200. https://doi:10.4236/aces.2016.62020.
- [18] P. Das, N. Sharma, A. Puzari, D.K. Kakati, N. Devi, Synthesis and characterization of neem (Azadirachta indica) seed oilbased alkyd resins for efficient anticorrosive coating application, Polym Bull 78 (1) (2021) 457–479.
- [19] O. Daniel, T. Claudia, H. Antonella, F. Santiago, Low viscosity alkyd resins based on trimethylo-propane and Peruvian oil, J Polym Res 28 (6) (2021) 1–10.
- [20] R.R. Salata, B. Pellegrene, M.D. Soucek, Synthesis and properties of high solids triethox-ysilane-modified alkyd coatings, Prog Org Coating 133 (2019) 340–349. https://doi: 10.1016/j.porgcoat.2019.04.037.
- [21] U.E. Igbo, M.A. Osinubi, R.O. Ishola, V.I. Isiba, E.U. Akubuaze, Effect of varietal difference on the physicochemical properties and fatty acid profile of kenaf seed oil, Res J Inter Study 4 (26) (2016) 2908–2913.
- [22] Y.H. Choi, R. Verpoorte, Green solvents for the extraction of bioactive compounds from natural products using ionic liquids and deep eutectic solvents, Curr Opin Food Sci 26 (2019) 87–93, https://doi.org/10.1016/j.cofs.2019.04.003.
- [23] M. Boruaĥ, P. Gogoi, B. Adhikari, S.K. Dolui, Preparation and characterization of Jatropha Curcas oil based alkyd resin suitable for surface coating, Prog Org Coating 74 (3) (2012) 596–602. https://doi:10.1016/j.porgcoat.2012.02.007.
- [24] U.E. Igbo, C.C. Igwe, E. Akubueze, O.R. Ishiola, A.O. Odusote, A.O. Oyewole, Utilisation of beniseed oil for the production of alkyd resin, IOSR J Appl Chem 7 (5) (2014) 104–106. https://doi:10.9790/5736-0753104106.
- [25] M. Mutar, A. Hassan, Synthesis and characterization of new alkyd resins (short, medium and long) based on sunflower oil and linoleic acid as binder for paints, Int J Chem Petrochem Technol 7 (2017) 1–16.
- [26] I.H. Ifijen, M. Maliki, I.J. Odiachi, O.N. Aghedo, E.B. Ohiocheoya, Review on solvents based alkyd resins and water borne alkyd resins: impacts of modification on their coating properties, Chem Afr 5 (2) (2022) 211–225. https://doi:10. 1007/s42250-022-00318-3.
- [27] E.U. Ikhuoria, A.I. Aigbodion, S.O. Omorogbe, Correlative studies on the properties of rubberseed and soyabean oilbased alkyd resins and their blends, J Coating Technol Res 18 (2) (2021) 459–467.
- [28] F.A. Kareem, S.T. Yussuf, F. Akinwunmi, A.A. Ibikunle, Comparative evaluation of alkyd resins of soybean and Castor seed oils, J Chem Soc Nig 44 (5) (2019) 914–921.
- [29] F. Chemat, M. Abert-Vian, A.S. Fabiano-Tixier, J. Strube, L. Uhlenbrock, V. Gunjevic, G. Cravotto, Green extraction of

natural products: origins, current status, and future challenges, Trends Anal Chem 118 (2019) 248–263, https://doi.org/10.1016/j.trac.2019.05.037.

- [30] B. Marcin, M. Eugeniusz, S. Lukasz, Vegetable oils in the production of biodegradable alkyd resins, Rev Org Chem 16 (4) (2019) 399–404.
- [31] C. Fabien, D. Maxinne, N. Claire, C. Sylvain, Hybrid alkyds, the glowing route to reach cutting-edge properties, Prog Org Coating (2021) 106025–106067, https://doi.org/10.1016/j. porgcoat.2020.106025.
- [32] C.F. Uzoh, M.C. Obele, U.M. Umennabuife, O.D. Onukwuli, Synthesis of rubber seed oil waterborne alkyd resin from glucitol, J Coating Technol Res 16 (6) (2019) 1727–1735, https://doi.org/10.1007/s11998-019-00235-0.
- [33] X. Yun, Y. Xin-Yi, G. Dun-Hong, D. Yong-Bo, S. Liang, Preparation and characterization of waterborne alkyd-amino baking coatings based on waste polyethylene terephthalate, R Soc Open Sci 7 (1) (2020) 191447–191450, https://doi.org/ 10.1098/rsos.191447.
- [34] A.R. Luz, C.R. Juliana, O. Alvaro, Assessment of degumming and bleaching processes for used cooking oils upgrading into oleochemical feeds-tocks, J Environ Chem Eng 9 (1) (2021) 104610–104624.

- [35] A.S. Yusuff, Parametric optimization of solvent extraction of Jatropha curcas seed oil using design of experiment and its quality characterization, S Afr J Chem Eng 35 (2021) 60–68. https://doi:10.1016/j.sajce.2020.11.006.
- [36] C. Kezban, G. Evrim, I. Nasit, Authentication and quality assessment of meat products by Fouri-er-transform infrared (FTIR) spectroscopy, Food Eng Rev 13 (1) (2021) 66–91.
- [37] R.A. Pratiwi, A.B.D. Nandiyanto, How to read and interpret UV-VIS spectrophotometric results in determining the structure of chemical compounds, Indonesian J Educ Res Technol 2 (1) (2022) 1–20. https://doi:10.17509/ijert.v2i1.35171.
- [38] F.A.M. Al-Zahrani, L. Lin, H. Abd El-Wahab, Preparation and evaluation of high-performance modified alkyd resins based on 1,3,5-tris-(2-hydroxyethyl)cyanuric acid and study of their anticorrosive properties for surface coating applications, E-Polymers 22 (1) (2022) 781–792, https://doi.org/10. 1515/epoly-2022-0065.
- [39] A.O. Mustapha, K.A. Usman, T.A. Zakariyah, O.M. Adekoya, Y.T. Afolabi, J.O. Toluwalase, S. Amao, P.T. Joseph, Optimization of process parameters for biodiesel production from three indigenous vegetable oils, Ibn Al- Haitham J Pure Appl Sci 35 (3) (2022) 98–109. https://doi:10.30526/35.3.2810.