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RESEARCH ARTICLE



Synthesis and Chemical Characterization of Alkyd Resins Using Maleic and Phthalic Anhydrides and Seed Oil of *Luffa aegyptiaca*

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Abstract: The study aimed to provide sustainable alternatives to reduce industries' over-reliance on edible vegetable oil for alkyd resin preparation as applicable in paint production. Alkyd resins were synthesized and characterized from sponge (Luffa aegyptiaca) seed oil. Condensation polymerization of monoglyceride with phthalic and maleic anhydride was carried out, and physico-chemical parameters such as drying time, total solids, viscosity, and chemical resistance were investigated following standard procedures. UV-visible, FT-IR, 1H, and 13C NMR spectroscopies were used to characterize the prepared alkyd resins. Sponge seed oil alkyd resins prepared with maleic anhydride (SPOMA) had a higher percentage yield (77.56%) than sponge seed oil prepared using phthalic anhydride (SPOPA) with 64.44%. The two alkyd resins showed a better drying time of 40 – 50 min than their commercial counterparts (70 min). This was attributed to the high degree of unsaturation of the seed oil due to the considerable proportion of linoleic acid in the seed oil. The alkyd resins were largely stable in 0.1 M HCl, 5% NaCl, and 0.1 M KOH, which caused the alkyd resins to whiten and shrink. The resins were generally soluble in xylene, kerosene, and petroleum ether. The nature of the alkyd resin can be described as nonpolar. This observation was consistent with the literature report. This study concluded that quality or industrial-grade alkyd resins could be prepared from sponge seed oil and thereby serve as a cheap and viable replacement for edible oils used in industries.

Keywords: Alkyd resin, sponge seed oil, spectroscopic characterization, phthalic anhydride, maleic anhydride.

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1. INTRODUCTION

Despite the abundance of renewable tropical plant seeds in Nigeria, a survey of the literature and personal interactions with vegetable oildependent companies revealed that the industries import oil as raw materials from Western countries. In Nigeria, there is a need for more of well-organized, accurate data and information about the chemical composition and potential use of non-conventional seed oils. As a result, the raw-material supply chain for oilbased businesses could increase. In addition, the information gathered on seed oil will be added to the plant knowledge base.

The lack of interest in oil-based sectors to investigate indigenous raw materials may be

partly due to a lack of data on seed oil and a lack of synergy between industry and academia. Data obtained from this study would promote academic-industry ties while also assisting in the transferring research discoveries for everyone's benefit. More jobs could be created from the utilization of locally accessible plant seed oils as alternative and sustainable bio-stock for production of various end-user items, particularly in communities where these seeds are grown. The production of alkyd resins from plant seed oils in a sustainable manner would also assist in reducing global pollution. Examining the viability of the seed oils employed in this study, it was chosen for its environmental abundance and oil content.

Nigerian enterprises that rely on vegetable oil import oils as raw materials at excessive prices. Locally accessible oils can only cover a quarter of demand, exposing companies to unjustified competition for oil with food applications (1). In addition, many manufacturing businesses employ edible oil consumed in Nigeria, resulting in domestic-industry competition. With several tropical underutilized and undervalued seedproducing plants, such as *Luffa aegyptiaca*, the country can produce enough fixed oil for both local and industrial needs, and sufficient reserves for export at extremely affordable cost.

Luffa aegyptiaca, a fast-growing annual vine that matures in about four months are found in Asia, India, Brazil, and Nigeria (2). The plant often called luffa is a cucurbit that includes gourds, pumpkins, and cucumbers, all of which are members of the Cucurbitaceae family. Luffa is known by various names, including smooth loofah, loofah sponge, sponge gourd, vegetable sponge, dishrag gourd, and Chinese okra. Luffa cylindrica and Luffa aegyptiaca are the two species of luffa(3). From the literature, sponge seeds are high in unsaturated fatty acids with linoleic acid being the most abundant fatty acid in the seed (4,5). The lack of information on the chemistry and application of accessible seeds with respect to their physicochemical features and prospective industrial applications is one of the reasons why some seeds/seed oils remain underutilized. As a result, research is essential to unravel the seed oil potentials and harness the data for profitable utilization (6,7). This research is thus aimed at exploring and characterizing the underutilized seeds of *Luffa aegyptiaca* seed as a renewable source of fixed oil and source of commercial raw materials for oil-based industries in tandem with the sustainable goal (SDG 11).

2. MATERIALS AND METHODS

Oil was extracted from dried and pulverized seeds of *Luffa aegyptiaca* according to standard procedure (8). The pulverized dried seed material (400 g) was extracted for 7 hours using n-hexane (2000 mL) at 55 °C using a soxhlet extractor. At 40°C, the oil was recovered from the solvent using a rotary evaporator (9).

2.1. Preparation of Alkyd Resin from the Seed Oil

The alkyd resin preparation was accomplished using the standard procedure (10). The oil was first alcoholized using stoichiometric quantities of oil and glycerol (Table 1) using sodium methoxide as a catalyst. Methanol was used to produced solubility test the of the monoglycerides. The reaction was cooled after the monoglycerides were formed, and then phthalic/maleic anhydride was added, followed by xylene to aid in the removing esterified water by producing an azeotrope. The reaction temperature was then raised to 235 °C for 5 hours. To check for a drop in acid value, aliquots were obtained from the reaction mixture at 1hour intervals. By submerging the reaction vessel in cold water, the reaction was halted. The alkyd resin's formulation is presented in Table 1.

Raw materials	Composition
	(%)
Seed oil	45
Glycerol	20
Phthalic anhydride/Maleic anhydride	25
Xylene	10

Table 1: FUTHUIALION OF THE AIKYU TESIN	Table	1:	Formulation	of the	alkyd	resin.
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2.2. Physicochemical Analysis of the Alkyd Resin

To determine the acid value, viscosity, and total solids, the American Oil and Chemists Society's standard approach was used. In addition, standard procedures ASTMD1640-69 and ASTMD 1308-5-57 were used to determine the drying time and chemical resistance of alkyd samples in various solvents, as described in (11,12).

2.3. Chemical Stability of the Alkyd Resin

The chemical resistance of the prepared alkyd resins with their commercial counterparts was tested by dissolving little quantity of the alkyd in distilled water, 0.1 M KOH, 0.1 M HCl, 5% NaCl, petrol, methanol, and kerosene, respectively (13).

2.4. UV-Visible Spectroscopic Analysis of the Prepared Alkyd Resin

The synthesized alkyd resins were analyzed using UV–Vis spectroscopy operating in a range of 300 to 800 nm. A concentration of 1 μ M was prepared in methanol or dichloromethane whilst using the solvents as blank for the analysis (14).

2.5. Fourier Transform Infrared (FT-IR) Spectroscopic Analysis of the Prepared Alkyd Resin

The infrared spectra were obtained on a Shimadzu 8400s using a KBr pellet, as described (15). In order to identify functional groups present in the seed oil, phthalic anhydride, maleic anhydride, and alkyd resins, the, infrared spectroscopic analysis was, performed on individual sample.

2.6. Nuclear Magnetic Resonance (NMR) Spectroscopic Analysis

¹H (Proton Nuclear Magnetic Resonance) and ¹³C NMR (Carbon 13 Nuclear Magnetic Resonance) of SPOMA (Alkyd resin made from Sponge seed and Maleic anhydride) and SPOPA (Alkyd resin made from Sponge seed and Phthalic anhydride) dissolved deuterated DMSO (Dimethyl in sulfoxide) were recorded on a Bruker Avance III⁺ HD 500 MHz spectrometer equipped with a 5 mm wide helium-cooled probe and an automatic 24sample converter. Avance III HD 500, three channels, BOSS III (36 magnetic field homogenity corrections), BSMS 2, amplifiers BLAX2H 300/100 and BLAX 300.

Table 2: Physicochemical Analysis of the Alkyd Resin.

Parameters	SPOPA	SPOMA	Control
			Resin
Yield, %	64.44	77.56	-
Acid Value	8.42	7.67	11.52
Viscosity (cst)	818.65	811.06	815.52
Total Solid (%)	89.42	94.37	51.56
Drying time (min)	40	50	70

SPOMA – Alkyd resin made from Sponge seed and Maleic anhydride SPOPA – Alkyd resin made from Sponge seed and Phthalic anhydride Control Resin – Commercial Alkyd resin

3. RESULTS AND DISCUSSION

3.1. Physicochemical Analysis of the Prepared Alkyd Resin

The two alkyd resins, SPOPA and SPOMA with distinct properties, were prepared by esterifying the resulting intermediate product of sponge seed oil with glycerol. According to the reviewed literatures, no precise standard exists because each alkyd is unique (16). However, two elements were discovered to influence the features of the alkylated resin produced. Table 2 shows the results obtained compared with commercial alkyd resin purchased from the market.

Alkyd resins prepared with maleic anhydride had a higher percentage yield than those made with phthalic anhydride. Sponge seed oil and maleic anhydride (SPOMA) afforded a maximum yield

3.2. Chemical Resistance of the Alkyd

of 77.56%, while SPOPA made from phthalic anhydride and the seed oil afforded a yield of 64.44%. The variation observed could be as a result of higher degree of unsaturation of phthalic anhydride. Low acid content, high viscosity, total solids, and short drying time are other properties that determine the quality of alkyd resin (11, 17).

SPOMA (40 min) has a shorter drying time than SPOPA (50 min), while both have comparable drying period of 70 mins for commercial alkyd resin. The prepared alkyd resins outperform the commercial alkyd resin in total solids, ranging from 89.42% for SPOMA to 94.37% for SPOPA, compared to 51.56% of the commercial alkyd resin. All prepared alkyd resins had a viscosity in the range of 811.06 cst to 818.65 cst, which is comparable to the commercial alkyd resin's viscosity of 815.52 cst.

Resins

The chemical stability of the alkyd resin was

determined by immersing different alkyd resins (prepared and commercial) alkyd in various solvents with different polarities. In distilled water, 0.1 M HCl, and 5% NaCl, all of the alkyds were largely stable. However, the KOH caused the alkyd resin to whiten and shrink, and it was generally unstable in xylene, kerosene, and petroleum ether (Table 3). The nature of the alkyd resin can be described as nonpolar. This conclusion is consistent with the findings of others (18,19).

3.3. Result of UV-Visible Spectroscopy Characterization of the Alkyd resin

UV-visible spectroscopy was also used to predict extent of the alkyd formed. The alkyd resins displayed a bathochromic (red) shift due to π – π^* and n – π^* transitions, as shown in the UVvisible spectrum in Figures 1 – 2 and Table 4. In the synthesis of SPOPA alkyd resin, a red shift to 426 nm was detected, compared to 314 and 278 nm for raw sponge seed oil and phthalic anhydride, respectively. The appearance of a new absorption peak at 417 nm, consistent with literature report (20), lends credence to the claim about the alkyd resin formation (Table 4, Figure 1). This behavior was attributed to the elongation of conjugation coupled with the presence of several auxochromes in the new product (21). Similar observations were obtained in the synthesis of SPOMA alkyd resin, in which the alkyd resin exhibited a redshift in the absorption λ_{max} to 417 nm compared to 247 nm for maleic anhydride (Table 4, Figure 2).

3.4. FT-IR Characterization of the Alkyd Resin

Physical and chemical analyses of the resin, and FT-IR characterization, were used to determine the formation of the alkyd resin. As illustrated in Figures 3 and 4, the FT-IR spectrum revealed that the starting reagents (seed oil and polybasic acids) utilized all had distinct absorption bands. The FT-IR data of sponge seed oil, maleic/phthalic anhydride, and alkylated resins are given in Tables 5 and 6.

Table	3:	Chemical	Resistance	of the	Alkyd	Resin.
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Parameter	SPOPA	SPOMA	Control
			Resin
Distilled water Resistance	Stable	Stable	Stable
0.1M HCl Resistance	Stable	Stable	Stable
0.1M KOH	Decolorize/Shrinkage	Decolorize/Shrinkage	Shrinkage
5% NaCl	Stable	Stable	Stable
Xylene	Unstable	Unstable	Unstable
Petroleum ether	Unstable	Unstable	Unstable
Kerosene	Unstable	Unstable	Unstable

Table 4: UV-Visible Spectroscopic Characterization Data of the Alkyd Resin.

Samples	λ_{max}
	(nm)
Sponge seed oil	314
Maleic Anhydride	247
Phthalic anhydride	278
SPOPA	426
SPOMA	417



Figure 1: UV–Visible spectrum of SPOPA Alkyd Resin.



Figure 2: UV-Visible spectrum of SPOMA Alkyd Resin.

Frequency of Absorption bands (cm ⁻¹)				
Functional	Sponge seed	Sponge seed Phthalic SPOPA		
groups	oil	anhydride		
C-0	1262	1109 – 1257	1288	
C=O	1712	1763	1716	
C=C		1599	1599	
C-H	2854 – 2926	3091	2926	
O-H			3398	
011			5550	

 Table 5: FT-IR spectra analysis of sponge seed oil, phthalic anhydride and SPOPA Alkyd Resin.

SPOPA – Alkyd resin made from Sponge seed and phthalic anhydride.

The stacked spectra of the seed oil, anhydrides, and corresponding alkyd resins produced are shown in Figures 3 and 4. The absence of a hydroxyl (OH) functional group in the seed oil, which is prominent in the alkyd resin at 3398 cm^{-1,} substantiated the success of the synthesized alkyd resin. Similarly, the absence of a hydroxyl (OH) functional group in the maleic and phthalic anhydrides in Tables 5 (SPOPA) and 6 (SPOMA), which were prominent in the alkyd resin at 3398 cm⁻¹ and 3431 cm⁻¹, respectively, in Figures 3–4, confirmed the formation of the alkyd resin. For the C-O carboxylic functional group, corresponding peaks were detected in the range of 1109–1288 cm⁻¹ for seed oil, maleic anhydride, and SPOPA, whereas the same peaks were observed in the range of 1273 cm⁻¹ for SPOMA. For both SPOPA and SPOMA, the C=O carbonyl group was found in 1712–1763 cm⁻¹ range. This is in line with (22) about FT-IR characterization of palm oil alkyd resin results.

Table 6: FT-IR spectra Analysis of Sponge seed oil, Maleic anhydride and SPOMA Alkyd Resin.

	Frequency of Absorption bands (cm ⁻¹)			
Functional	Sponge seed	Maleic	SPOMA	
groups	oil	anhydride		
C-0	1262	1109 – 1257	1172 – 1273	
C=0	1712	1763	1720	
C-H	2854 – 2926	3091	2929	
O-H			3431	

SPOMA – Alkyd resin made from Sponge seed and Maleic anhydride



FT-IR spectra for Sponge seed oil Alkyd resin with Phthalic anhydride (SPOPA)

Figure 3: FT-IR Overlaid Spectral of Sponge seed oil, Phthalic anhydride and SPOPA.



FT-IR spectra for Sponge seed oil Alkyd resin with Maleic anhydride (SPOMA)

Figure 4: FT-IR Overlaid Spectra of Sponge seed oil, Maleic anhydride and SPOMA.

3.5. ¹H and ¹³C NMR Characterization of the Alkyd resins

Figure 5 depicts the structure of SPOPA alkyd resin, while Figure 6 depicts the SPOPA alkyd resin ¹H NMR spectrum with the assignment of the signal indicate in Table 7. The terminal alkyl group protons were confirmed at $\delta_{\rm H}$ 0.85. Protons of all methylene (–CH₂) present in the long aliphatic chain are responsible for the strong signals between $\delta_{\rm H}$ 1.24 and 1.38. The chemical shift observed between $\delta_{\rm H}$ 3.38 and $\delta_{\rm H}$ 3.75 is due to the protons connected to the carbon in which the hydroxyl group is attached, whereas the signal observed at $\delta_{\rm H}$ 3.62 is due to

a methylene proton bonded to the carbon in which the hydroxyl group is attached.

The olefinic proton signal in the seed oil was verified in the alkyd resin at a slightly greater chemical shift of $\delta_{\rm H}$ 5.32, attributed to the seed oil's unsaturation. In the same way, the aromatic proton signals in phthalic anhydride were found to exhibit a lower chemical shift signal in the $\delta_{\rm H}$ 7.69–7.88 range. The peaks in the alkyd resins which were absent in the seed oil or anhydride used helps to confirm the SPOPA alkyd resin manufacture. Islam *et al.* (2014) (23), who used ¹H NMR to describe alkyd resin produced from palm oil, reported similar findings.



Where R = fattyl acyl



Figure 5: Structure (Proposed) for SPOPA alkyd resin.



¹ H Signal (ppm)	Chemical Shift	Assignment
0.85	CH ₃	Terminal methyl (Saturated)
1.24 - 1.38	$(CH_2)n$ saturated aliphatic	All acyl chains
2.0	CH ₂ -OCOR	Bonded to the acyl chain
2.29	CH ₂	α- methylene group
3.38	ОН	Monotriglycerol
3.62	CH-(OH)	Monotriglycerol
3.75	ОН	Monotriglycerol
4.33	CH ₂ -(OH)	Monotriglycerol
5.32	CH=CH (Olefinic group)	All unsaturated fatty acids
7.69	H-C*C-H	Aromatic ring
7.88	H-C*CH*C-H	Aromatic ring

Table 7: ¹H NMR Characterization of SPOPA Alkyd Resin.

The ^{13}C NMR spectra of SPOPA Alkyd resin is shown in Figure 7. In Table 8, the signal assignments are listed. At the chemical shift of δ_{c} 14.2, the signal for the terminal methyl group was confirmed. Due to overlaps of every methylene $(-CH_2)$ present in the long aliphatic chain, strong signals between the area on δ_c 27.00–29.4 are caused. The chemical shift signal between $\delta_{\rm C}$ 63 and $\delta_{\rm C}$ 77.1 is due to the C–O carbon of esters or carboxylic groups, whereas the signal between δ_{c} 131.67 and 132.7 is due to the C=C on the benzene ring of the phthalic anhydride component of the alkyd resin. The presence of C=C in linoleic and vaccenic acids discovered in the fatty acid component of the alkyd resin can also be attributable to this finding. The presence of carbonyl (C=O) signals at δ_{C} 167.8, 168.6, and 168.9 indicates the formation of esters, thus confirming successful formation of the SPOPA alkyd resin. The absence of a signal at or above δ_{C} 180 indicated the absence of carboxylic acids, implying that all of the C=O in the fatty acids of the oil had been successfully converted to ester. Kanai *et al.* (2007) (24), who used ¹³C NMR to describe alkyd resin produced from soybean oil, found similar results.

Figure 8 corresponds to the proposed structure of SPOMA alkyd resin, while Figure 9 corresponds to the SPOMA alkyd resin ¹H NMR spectrum with the signal assignment reported in Table 9. In the chemical shift of $\delta_{\rm H}$ 0.84, was assigned to the terminal alkyl group. methylene (–CH₂) protons in the long aliphatic chain are

responsible for the prominent signals at $\delta_{\rm H}$ 1.24–1.48. The $\delta_{\rm H}$ 3.34 and 3.64 signal was due to a proton on the monotriglycerol hydroxyl group, while the signal at $\delta_{\rm H}$ 3.54, 4.19, and 4.42 are due to methine and methylene protons linked to the carbon in which the hydroxyl group is connected. Similarly, the vinyl protons in maleic anhydride were observed at $\delta_{\rm H}$ 6.63–6.67. The peaks in the alkyd resins that were absent in the seed oil and anhydride help confirm the SPOMA alkyd resin formation and the data were consistent with literature (25) wherein ¹H NMR were adopted for the characterization of alkyd resin produced from sunflower seed oil.

The ¹³C NMR spectra of SPOMA alkyd resin is shown in Figure 10 with the signal assignment in Table 10. The methylene carbon signal (-CH₂) found in the long aliphatic chain is responsible for the prominent signals at δ_c 31.35 ppm. The chemical shifts between δ_c 60.1–72.9 ppm are due to methylene carbon bound to the hydroxyl component on the glycerol chain, while the signal at δ_c 132.9–135.5 ppm are due to the methine carbons on the benzene ring of the maleic anhydride component of the alkyd resin. The carbonyl (C=O) signals at δ_c 164.6–166.2 indicate the formation of an ester and aid in the confirmation of SPOMA alkyd resin synthesis. The absence of a signal at δ_{C} 180 ppm for carbonyl carbon indicates the absence of carboxylic acids, which implies a successfully conversion of the fatty acids carboxylic in the oil to ester. This is consistent with the literature report (26).



Figure 7: ¹³C NMR Spectrum of SPOPA Alkyd Resin.

Table 8:	¹³ C NMR	Characterization	of SPOPA Alkyd	Resin.
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¹³ C Signal (ppm)	Chemical Shift	Type of Carbon	Assignment
14.2	<u>C</u> H₃	Methyl	Terminal methyl
27.0 – 29.4	<u>C</u> H ₂	Methylene	
63.0 – 77.1	<u>C</u> H ₂ -OH	Methylene	Monoglycerol chain
131.6 – 132.7	<u>CH</u>	Methine	Benzene ring and Vinyl carbon
167.8	<u>C</u> =0	Carbonyl	Ester
168.6	<u>C</u> =0	Carbonyl	Ester
168.9	<u>C</u> =0	Carbonyl	Ester



Where R = fattyl acyl

Figure 8: Structures (Proposed) for SPOMA alkyd Resin.

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Figure 9: ¹H NMR Spectra of SPOMA Alkyd Resin.

¹ H Signal (ppm)	Chemical Shift	Assignment
0.84	CH₃	Terminal methyl (Saturated)
1.24 - 1.48	$(CH_2)n$ saturated aliphatic	Acyl chains
1.97	CH ₂ CH=CH	Unsaturated acyl chains
2.47	CH ₂ -OCOR	Bonded to the acyl chain
2.60 – 2.74	CH ₂	α- methylene group
3.34	ОН	Monotriglycerol
3.54	CH-(OH)	Monotriglycerol
3.64	ОН	Monotriglycerol
4.19 - 4.42	CH ₂ -(OH)	Monotriglycerol
5.42	CH=CH (Olefinic group)	Unsaturated fatty acids
6.63 - 6.67	HC=C-H	Vinyl chain (Maleic anhydride)

Table 9: ¹H NMR Characterization of SPOMA Alkyd Resin.



Figure 10: ¹³C NMR Spectrum of SPOMA Alkyd Resin.

Table 10: ¹³C NMR Characterization of SPOMA Alkyd Resin.

¹³ C NMR Signal (ppm)	Chemical Shift	Type of Carbon	Assignment
31.3	$\underline{C}H_2$	Methylene	
60.1 – 72.9	<u>C</u> H ₂ -OH	Methylene	Monoglycerol chain
132.9 – 135.5	<u>C</u> H= <u>C</u> H	Methine	Maleic chain
164.6 - 166.2	<u>C</u> =0	Carbonyl	Ester

4. CONCLUSION

To minimize domestic and industrial rivalry for vegetable oils in Nigeria, alkyd resins were successfully synthesized using standard analytical techniques from underutilized seeds of Luffa aegyptiaca. The prepared alkyd outperformed the commercial alkyd used as a control sample for some parameter. Both prepared alkyds have higher total solids (89.42% for SPOPA and 94.37% for SPOMA), while the commercial alkyd employed as a control had a total solid content of 51.56 percent. The modified products were confirmed using fourier transform infrared spectroscopy (FT-IR), ultraviolet-visible spectroscopy (UVvisible), ¹H nuclear magnetic resonance (¹H NMR), and ¹³C nuclear magnetic resonance (¹³C NMR) spectroscopy. The study found that alkyd resin could be made cheaply from sponge seed oil thereby freeing up more conventional vegetable oil for human use and lowering

domestic-industrial competition for vegetable oil, especially in developing countries where the seed are produced in abundance annually.

5. CONFLICT OF INTEREST

Authors declare no conflict of interest.

6. ACKNOWLEDGMENTS

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