



Synthesis and Evaluation of Alkyd Resins from Underutilized (*Delonix Regia* and *Theventia Peruviana*) Seed Oils

Aliru O. Mustapha*, Salihu F. Adisa, Simeon G. Oladele, Yemisi T. Afolabi

Kwara State University, Malete –Nigeria

Article information

Article history:

Received: March, 11, 2022

Accepted: April, 12, 2022

Available online: June, 14, 2022

Keywords:

Alkyd resin,

Polymerization,

Delonix regia,

Theventia peruviana

*Corresponding Author:

Aliru O. Mustapha

aliru.mustapha@kwasu.edu.ng

DOI:

<https://doi.org/10.53523/ijoirVol9I1ID148>

Abstract

The inedibility of *delonix regia* and *theventia peruviana* seeds represents a significant waste of resources. The physicochemical properties of underutilized seed oils after refinement were studied using Association of Officials of Analytical Chemists (AOAC, 1990) techniques in this study. The refined *theventia peruviana* oil (RTPO) has a yield of 40.55 %, but its long alkyd resin (LTPOAR) has iodine value of 80.12 (gI₂/100g), viscosity 24.17 (30° C, m²/s), saponification value of 177.04 (mg/KOH/100g), and acid value of 9.53 (mg/KOH/g), while refined *delonix regia* oil (RFBO) has a yield of 52.71 %, with its long alky resin (LDROAR) having iodine value of 133.87 (gI₂/100g), viscosity 21.15 (30° C, m²/s), saponification value of 266.42 (mg/KOH/100g), and acid value of 5.71 (mg/KOH/g). For both TPOAR and DROAR, six grades of alkyds were created at 25 % (short), 40 % (medium), and 60 % (long) oil lengths. The acid values of the aliquots for the reaction mixture at various time intervals were used to track the reaction's progress. At the initial stage of the reaction, the extent of reaction (% Pav) ranged from 78.5 to 80.8 %, indicating a significant degree of conversion. The average degree of polymerization (Dp) of the alkyds ranged from 1.03 to 5.20, indicating the synthesis of high molecular weight alkyds. The alkyd films were acid, brine, and water resistant, but not alkali resistant. All alkyd resins were characterized with surface drying and solubility times and were found to be around 2 hours in respect of the 60% oil length.

1. Introduction

The expenses of utilizing petrol based monomers in the assembling of surface covering are expanding continuously, and there is a likely going to be shortage of the consumption of the petroleum [1]. A shortage of oil based goods combined with expendable petrol have caused vulnerability in the supply of oil based commodities for both home use and mechanical utilizations [2,3]. There has been enormous expansion in the interest for alkyd tar creation for use in the surface covering industry because of its quick development, Thus, huge amounts of oil are required for the creation of alkyds. Henceforth, there is need to explore indigenous non-edible oilseeds that can compare to those being imported for paint production. Plant seed oils from flamboyant and thevetia seeds are of recharged interest in the hunt and advancement of sustainable alkyd resins that would fill in as significant option in contrast to imported based commodities are underway [4]. Notwithstanding the obvious use of fats and oils are enormously supported for use in surface coatings, cleansers, makeup, drugs, oils, surfactants and polymers. Their wide acknowledgment in these fields of uses is owing to their being sustainable, renewable and biodegradable, thus climate friendly [5].

This improvement has given various studies on the quality and utilizations of tremendous number of indigenous African seed oils. The serious climate challenges give the drive and inspiration to create coatings that uses more affordable materials [6].

The strength or weakness of alkyd resin is largely determined by the feedstock used in its production. Long oil alkyd resins have a high proportion of drying oil content and are utilized as consumer coatings. They dry slowly and are used in high-gloss glass coatings and wood finishes. Medium oil alkyds have a lower amount of big molecular weight polyester backbone and less drying oil. In comparison to the basic polyester polymer or backbone chain, the percentage of drying oil in short oil alkyds is quite low. These coatings will not harden or dry unless they are heated. They're used to finish metal objects as baked enamel [7, 8].

Interestingly, seed oils like flamboyant and theventia seeds are among the undocumented, more affordable and sustainable assets that can be utilized in different regions as they are climate favorable. Investigation of indigenous non-edible oil seeds that may have physicochemical qualities are required to produce alkyd resins that would of the same standards as imported alkyd resins. Plant seeds of delonix regia and theventia peruviana have rekindled interest in the search for sustainable alkyd resins production as a viable alternative to imported because of their availability as they largely grow in poor soils. Ezugwu and Ezugwu [9] reported that many applications of delonix regia and theventia peruviana seeds have been used for cleansers, cosmetics, medications, oils, surfactants, but their benefits in the production of surface coatings, alkyd resins and paints are scanty in the literature [10].

Various investigations into the quality and use of a large number of indigenous African seed oils have improved as a result of progress in their widespread acceptance in the industry due to the fact that they are environmentally friendly, sustainable, renewable, biodegradable more and cost-effective materials [11]. Seed oils from delonix regia and theventia peruviana seeds, for example, are no exception as they have qualities such as climate-friendly, non-edible, more economical raw materials that can be used in the manufacturing of alkyd resin [12].

In this work, oils extracted from delonix regia and theventia peruviana seeds would be optimized and evaluated for their suitability in the production of alkyd resins, and thereby contributing to the global interest in producing environmentally friendly alkyd materials [13].

2. Experimental Procedure

Extraction and Refining of Crude Seed Oils

The pulverized seed samples (250 g) were extracted for 72 hours at room temperature with constant agitation in n-hexane (500 mL). After 72 hours, the crude extract was decanted, filtered, and concentrated using a rotary evaporator at 40°C under reduced pressure. The extracted crude oils of delonix regia (CDRO) and theventia peruviana (CTPO) were dried in desiccators, weighed, and the percentages of yields were determined. The refinement of crude oils was an important step in the production of alkyd resin because it removed impurities like phosphorus compounds and allows for efficient alkyd synthesis. Degumming, alkaline, and bleaching treatments were carried out during the refining process to produced refined oils of delonix regia (RDRO) and

theventia peruviana (RTPO) [14]. The physicochemical properties of refined oils and alkyd resins were carried out with Association of Officials of Analytical Chemists techniques [15].

Alkyd Resin Synthesis

Alcoholysis Stage

Table (1) contains the recipes for varied proportion of materials for the synthesis of short, medium and large alkyd resins. For short alkyd resin (R-DROAR), the reactor was charged with 25 g of each RDRO and 0.1g of CaCO₃ (catalyst) after the apparatus was set up. The mixture was heated to about 120 oC. The glycerol (4.91 g) was added at 120 oC and heated to 230–250 oC with vigorous agitation for 30 minutes triglyceride transesterification into a mixture of mono- and diglyceride oils. When a sample of the resulting mixture is soluble in 1 to 3 volumes of anhydrous methanol and forms a clear solution, alcoholysis is complete. To allow for a smooth transition to the esterification stage, the reaction temperature was reduced to 140 °C.

Esterification Stage

After 30 minutes, the temperature was lowered to 150 °C, and 14.93 g of phthalic anhydride was introduced. After that, reaction liquor was charged with xylene (5% of the total oil weight charged). The esterification water forms an azeotrope with xylene and was removed at intervals before the temperature was raised to 245 °C with continuous stirring to increase the resin's molecular weight. By heating the bulk to temperatures higher than 250 °C with vigorous stirring, water was removed with the unreacted acid. The 3-hours reaction process was monitored by checking the acid number and viscosity on a regular basis. When the resulting solution became viscous and the acid value dropped below 10, it was stopped. Then, while still hot, it was cooled to some extent and poured into a storage container for further study. After allowing the solid to cool to room temperature, it was washed three times in cold ethanol and then three times in diethyl ether. The solid was purified further by stirring it with tetrahydrofuran (THF) for several hours. Finally, the solid was vacuum-dried. After dissolving in a mixture of toluene and ethanol (1:1), the acid value of in-process samples taken at intervals was determined by titrating with a 0.1 M KOH solution to the phenolphthalein end point [9].

Table (1). Recipes for the formation of standard alkyd resins.

Ingredient (g)	Long	R-DROAR		R-TPOAR		
		Medium	Short	Long	Medium	Short
Oil	60	40	25	60	40	25
Glycerol	11.8	7.9	4.9	9.2	6.2	3.9
Phthalic anhydride	35.8	23.8	14.9	28.0	18.7	7.8
CaCO ₃ (Catalyst)	0.24	0.16	0.1	0.24	0.16	0.1

Fourier Transform Infrared Spectroscopy (FTIR) Structural Analysis

Shimadzu equipment was used for the FTIR analysis. As a background, KBr (potassium bromide, spectroscopy grade) was ground into a powdery form, pelletized (with a hydraulic press), and scanned. The alkyd sample was then mixed with KBr and pelletized using a hydraulic press. The pellets were then inserted into the instrument and scanned in transmittance mode at a frequency range of 4000–400 cm⁻¹.

The Alkyd Resin's Performance Characteristics

Drying and Film Properties Tests

Solidification Time: 5 g of each sample was collected and heated to the melting point using a laboratory heating mantle, then cooled and solidified at room temperature. The solidification time was determined by the time each solidified without flowing. Ethanol was used to clean mild steel sheets (3 cm × 3 cm × 1 mm) to ensure there were no impurities. Each test sample was placed on the sheet's surface. The drying time was the time it takes for the samples on the sheets to dry at room temperature.

Set to Touch: This was the amount of time it took for an alkyd resin to dry to the point where there was no physical deformation on the surface when touched.

Chemical Resistance Test

On the alkyd resins, the effects of acid, brine, water, and alkali were studied. To ensure the absence of impurities, steel sheet was cleaned with ethanol. They were given test samples, and replicates were immersed separately in dilute alkali (0.1M KOH), acid (0.1M H₂SO₄), brine (5% w/w NaCl), and distilled water. After 30 minutes, the metal sheets were removed, and chemical resistance was tested.

Solubility Test

The solubility of the alkyd resins produced was tested and determined in four different solvents (xylene, ethanol, methanol, and acetone). Each sample was weighed into conical flasks at a weight of 5 g. Each of the solvents was added separately in 5 mL increments, and the mixture was vigorously shaken. The solubility of each mixture was measured after it had been allowed to stand for 1 hour at room temperature [9].

3. Results and Discussion

Physico-chemical Properties of the Theventia Peruviana Alkyd Resin (TPOAR) and Delonix Regia Alkyd Resin (DROAR)

In Table (2), a comparison of the dark-brown in colored TPOAR and DROAR that were produced to a commercially manufactured alkyd (colorless) are shown. The alkyd's deepening color could be attributed to high reaction temperatures, oxidation, and the catalyst [9].

Table (2). Physico-chemical properties of the theventia peruviana(TPOAR) and delonix regia seed oil (DROAR) compared to commercially prepared alkyd resin (CPAR).

Parameters/units	LTPOAR	LDROAR	CPAR
Colour	Brown	Brown	Colourless.
Specific Gravity (30° C)	0.862±0.01	0.814±0.01	47.40
Viscosity (30° C, m ² /s)	24.17±0.28	21.15±0.21	32.34
Saponification Value (mg/KOH/100g)	177.04± 0.68	266.42±0.59	263.97
Iodine Value (gI ₂ /100g)	80.115 ± 1.62	133.87± 0.19	78.22
Acid Value (mg/KOH/g)	9.53 ± 0.37	5.71 ± 0.13	6.31

The iodine values of the alkyd resins prepared (DROAR and TPOAR) were discovered to have 133.87 and 80.115 mgKOH/g as compared to 161.395 and 104.41mg KOH/g. of crude seed oil. This could be as a result of dimerization and polymerization reactions at the reactive double bond of unsaturation during the alkyd production process. The ranges iodine values (40-60gI/100g) shows that it may be of the same class of semi-drying oil but except for the TPOAR (80.115mgKOH/g) and commercially made alkyd (78.22mgKOH/g), which were suggested to be in the class of semi-drying oil. The results of the iodine values were supported by literatures [17-19] for rubber seed oil.

Saponification values DROAR and TPOAR (266.84 and 177.23 mgKOH/g respectively) were found to have decreased compared to the seed oil DRO and TPO (300.21 and 315.15 mg KOH/g and that of commercially alkyds prepared (263.97 mg/KOH/g) [18]. This implies that these seed oils may be of great essential in industrial purposes for soap and inks production [19].

At 30oC, the specific gravity and viscosity of the test alkyd resins were found to be substantially higher when compared to the seed oils' comparable values. This could be due to polymerization processes at the unsaturated reactive double bonds. During alkyd synthesis molecular weights also increased. At 30°C, however, the specific heat viscosity was between 21.00 - 24.19 and gravity between 0.863 -0.812.

The specific gravity of the test alkyd resins was not considerably greater than the specific gravity of the control alkyd resins (47.40) and the viscosity of commercially manufactured alkyd resin (32.34 m²/s). These results were comparable to those found in Otabor's work [20], implying that the test alkyd resins will outperform commercially available varieties in industrial applications if well-controlled.

Acidity was detected in alkyd resins made from oil samples (DRO and TPO) 5.61 and 9.53 mg KOH/g are comparable to the acid value of the 6.31 mg KOH/g from commercially manufactured alkyd resin. The acidity levels derived from the alkyd resins (5.61-8.6mgKOH/g) were consistent with those found (5.61-8.6mgKOH/g) in the literature [21, 22]. According to literature [20-23], the acid values of the test alkyd resins were lower than those of deodorizer distillate, soyabean oil, and walnut seed oil (9.6–14.24 mg/KOH/g). The acidity level in the alkyd content is a key factor in determining its chemical resistance. As solvents, water, brine (NaCl), acid (H₂SO₄), and alkali (KOH) have all been used as solvents. The lower an alkyd resin's acid value, the greater it's chemical resistance to various solvents media and the other way around. These alkyd resins have low acid values and shown to be promising in the formulation of coatings such as nitrocellulose lacquers [17].

Variation in Acid Value (AV), Extent of Reaction (Re), Viscosity, and Degree of Polymerization (Dp) of In-Process Alkyd Resins with Time

Tables (3-6) represent the AV, viscosity, Re, and Dp of in-process samples of delonix regia (DROAR) and theventia peruviana oil (TPOAR) alkyd resins variation with time as shown in Figures 1. At each time interval, the level of acid value in an aliquot of the reaction mixture was measured, as well as the volume of water released, to ensure that the poly condensation reaction was working properly. The increased values of the samples were owing to the reactions progressing (Figure 2). As the reaction progressed and the reaction time passed, the acid values decreased, as indicated in (Figure 1) which showed that there was an initial fast decrease in acid value followed by a steadier decrease [17]. The interactions of hydroxyl groups in glycerol with carboxyl groups in phthalic anhydride may be associated to the acid value decrease [19, 21, 23]. It has been found that secondary hydroxyl groups of monoglycerides react with carboxyl groups less than primary hydroxyl groups at different temperatures (primary hydroxyl groups at 160°C and secondary hydroxyl groups of monoglycerides at 230 °C [23]. Meanwhile, the previously reported considerable fall due to an increase in acid value for all alkyd samples esterification rate correlates with rapid temperature increases to 230 °C.

Table (3). Variation in acid value (AV) versus time for alkyd resins.

Time (min)	LDROAR	MDROAR	SDROAR	LTPOAR	MTPOAR	STPOAR
0	44.88	26.73	46.93	44.88	47.22	29.17
15	43.73	25.81	40.12	38.15	45.44	25.81
30	29.73	22.44	37.50	33.60	33.66	15.71
45	18.513	21.32	29.85	25.25	24.44	11.20
60	16.27	10.96	18.63	19.64	17.95	8.40
75	10.77	6.73	11.22	12.90	11.80	6.73
90	9.63	5.61	8.98	9.53	8.90	5.61

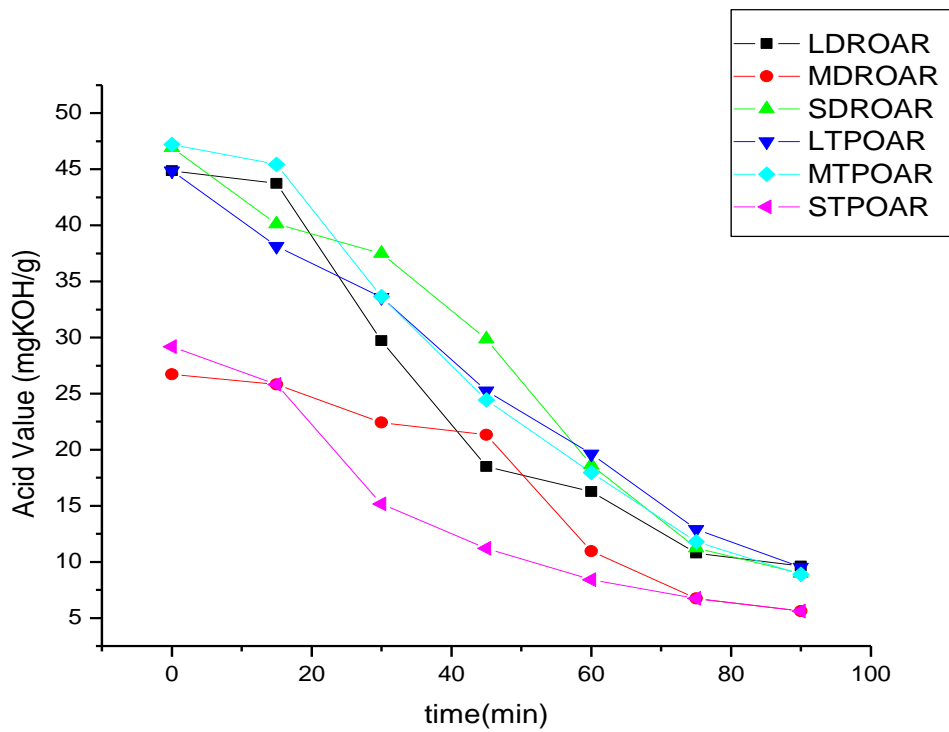


Figure (1). Acid value against reaction time.

Table (4). Viscosity versus time for alkyd resins.

Time (min)	LDROAR	MDROAR	SDROAR	LTPOAR	MTPOAR	STPOAR
0	12.80	10.22	10.08	9.20	8.10	8.00
15	14.70	12.66	11.80	10.75	10.11	10.80
30	18.11	14.00	13.60	12.77	13.22	11.18
45	20.42	16.50	15.60	14.63	14.37	12.80
60	22.45	19.20	18.65	18.63	18.54	14.00
75	24.37	22.50	21.75	20.89	21.10	15.59
90	25.20	24.86	22.84	24.19	23.20	17.30

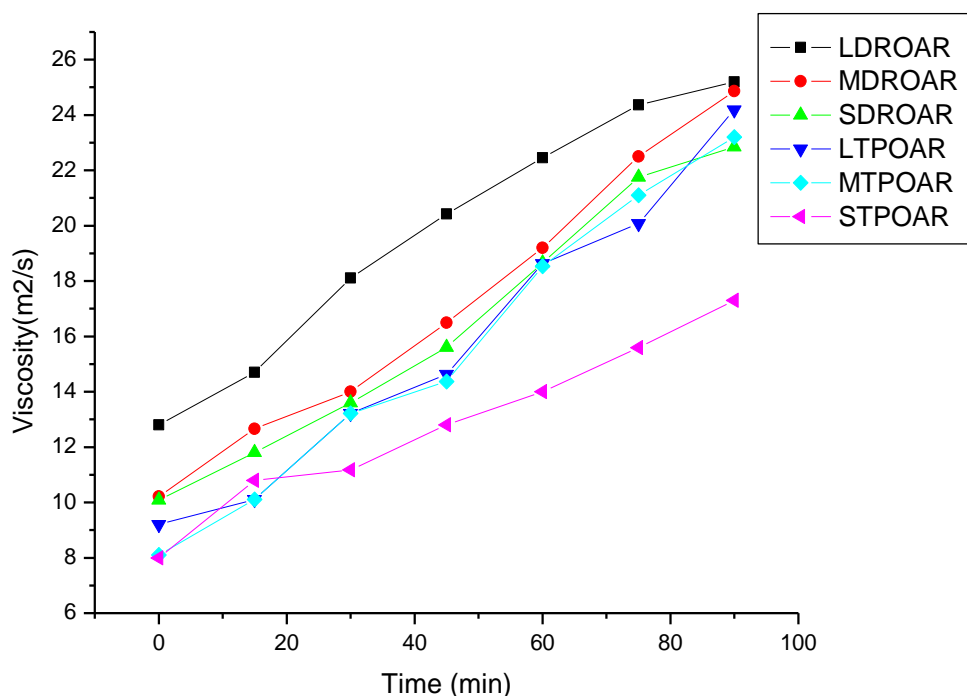


Figure (2). Viscosity versus time for alkyd resins.

Table (5a). Variation (AV) of in- process DROARs with the corresponding extent of reaction (%PaV)

Time (min)	LDROAR		MDRO AR		SDROAR	
	Acid value (mg KOH/g)	%PaV	Acid value (mg KOH/g)	%PaV	Acid value (mg KOH/g)	%PaV
0	44.88	-	46.93	-	47.22	-
15	43.73	2.56	40.12	3.44	45.44	14.01
30	29.73	33.76	37.50	16.05	33.66	20.09
45	18.513	58.76	29.85	20.24	24.44	36.40
60	16.27	63.75	18.63	59.00	17.95	60.30
75	10.77	76.00	11.22	74.82	11.80	76.09
90	9.63	78.54	8.98	79.01	8.90	80.87

Table (5b). Variation (AV) of in- process TPOARs with the corresponding extent of reaction (%PaV).

Time (min)	LTPOAR		MTPOAR		STPOAR	
	Acid value (mg KOH/g)	%PaV	Acid value (mg KOH/g)	%PaV	Acid value (mg KOH/g)	%PaV
0	44.88	-	47.22	-	29.17	-
15	38.15	21.68	45.44	20.59	20.81	11.52
30	33.60	25.13	33.66	41.17	15.71	46.14
45	25.25	43.74	24.44	57.29	11.20	61.60
60	19.64	56.24	17.95	68.63	8.40	71.20
75	12.90	71.26	11.80	79.38	6.73	76.93
90	9.53	78.77	8.90	84.48	5.61	80.77

The variation in the (AV and %PaV) with reaction time is shown in Figures (3 & 4). The first region between these plots shows the period when the primary hydroxyls of glycerol react with carboxyl groups of Phthalic anhydride leads to the formation of linear chains. Immediately, the primary hydroxyl groups are esterified, there

is an abrupt drop in acid value, the reaction slackens until a temperature is reached when the secondary hydroxyl groups begin to react, Thus, the increasing %PaV of the alkyd chains [3].

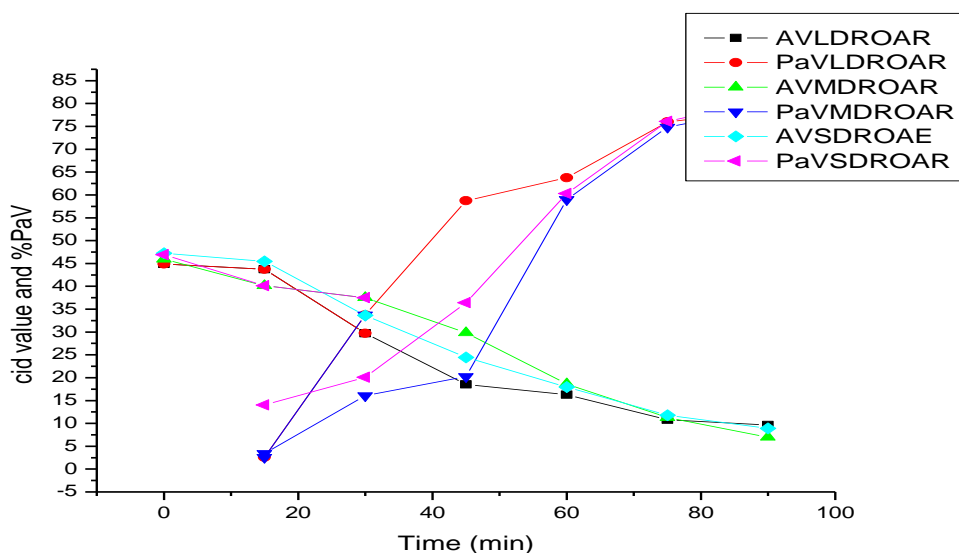


Figure (3). Variation (AV) of in- process samples and the corresponding extent of reaction (%PaV) (LDROAR, MDROAR, SDROAR) with time.

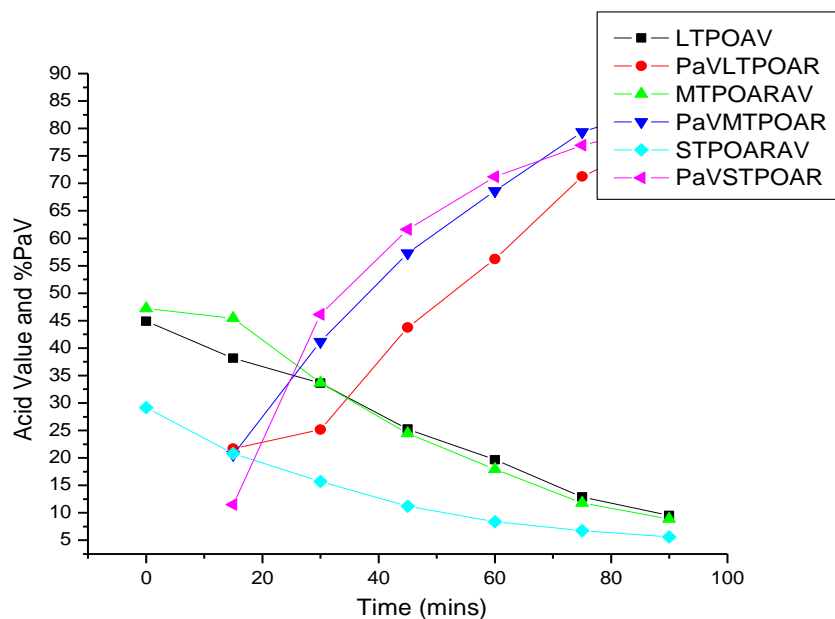


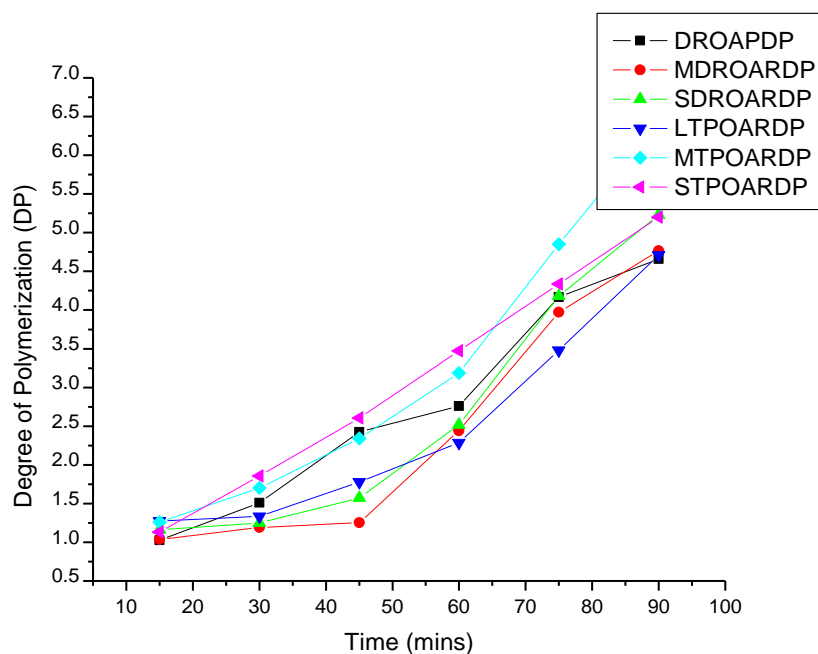
Figure (4). Variation (AV) of in- process samples and the corresponding extent of reaction (%PaV) (LTPOAR, MTPOAR, STPOAR) with time.

The average degree of polymerization (D_p) of the alkyds ranged from (1.026 – 4.66) for LDROAR, (1.036 – 4.764) for MDROAR, (1.163 – 5.227) for SDROAR, (1.277 – 4.71) for LTPOAR, (1.259 – 6.431) for MTPOAR, and (1.130 - 5.20) for STPOAR, indicating the synthesis of high molecular weight alkyds.

Table (6). Variation of degree of polymerization (Dp) with the reaction time.

Time (min)	LDROARMDROAR		SDROAR	LTPOAR	MTPOAR	STPOAR
	Dp	Dp	Dp	Dp	Dp	Dp
0	-	-	-	-	-	-
15	1.026	1.036	1.163	1.277	1.259	1.130
30	1.499	1.191	1.251	1.336	1.670	1.857
45	2.425	1.254	1.572	1.777	2.341	2.604
60	2.759	2.439	2.519	2.285	3.188	3.472
75	4.167	3.971	4.182	3.554	4.847	4.335
90	4.660	4.764	5.227	4.710	6.431	5.20

The variation of the degree of polymerization (Dp) of all the six alkyd resins with the reaction time is shown in Figure (5).

**Figure (5).** Variation of degree of polymerization (Dp) versus reaction time.

In contrast to acid value, an initial fast increase in the extent of the first reaction were seen, followed by a steadier increase (Figures 6–9), which is consistent with Igbo *et al.*, [19] and Oladipo *et al.*, [21]. The reactivity of the functional group in polyesterification reactions, on the other hand, is independent of the size of the molecule to which it is connected [3].

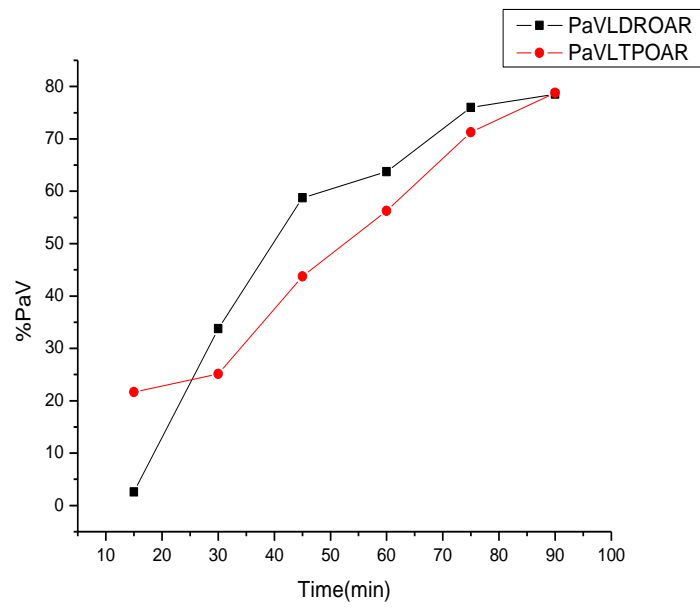


Figure (6). Extent of reaction versus time for LDROAR AND LTPOAR.

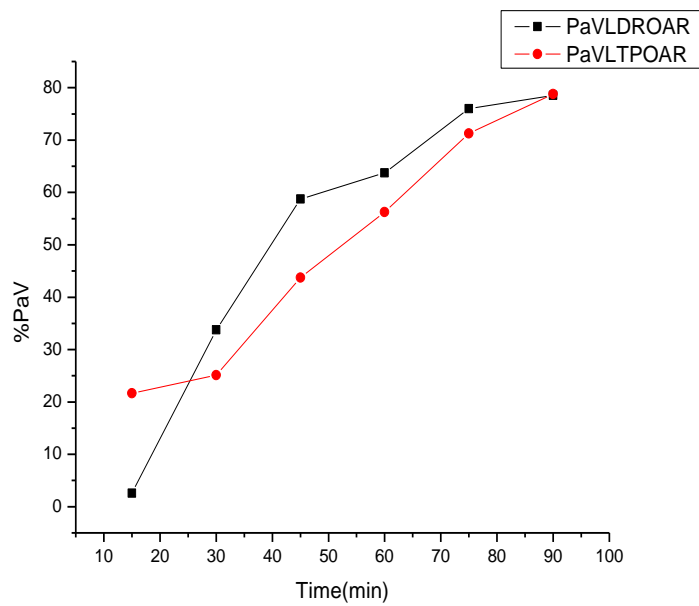


Figure (7). Extent of reaction versus time for LDROAR and LTPOAR.

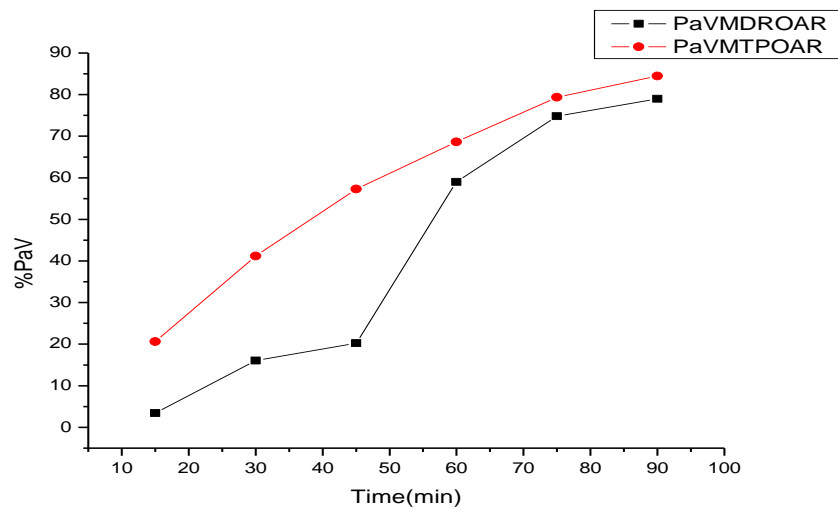


Figure (8). Extent of reaction versus time for MDROAR AND MTPOAR.

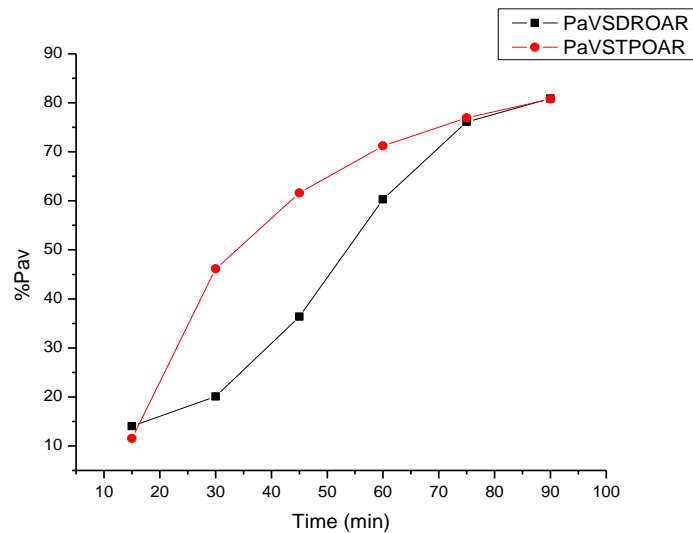


Figure (9). Extent of reaction versus time for SDROAR and STPOAR.

Table (7) shows the results of evaluating the chemical resistance of alkyd resin samples in various solvent media. All of the alkyd samples were resistant to water, acids and solvents but not to alkali. Polyesters were easily dissolved by alkali, so this was to be expected. Throughout the immersion period, the films were found to be extremely resistant to distilled water and aqueous sodium chloride solutions.

Table (7). Chemical resistance.

Medium	STPOAR	LDROAR	MDROAR	SDROAR	LTPOAR	MTPOAR
Distilled H ₂ O	FP	FP	FP	FP	FP	FP
NaOH 0.1M	FA	FA	FA	FA	FA	FA
H ₂ SO ₄ 0.1M	FP	FP	FP	FP	FP	FP
5% w/w NaCl	FP	FP	FP	FP	FP	FP

Film present (FP), Film absent (FA)

Another key attribute of alkyds is their drying schedule, which is, of course, the most crucial for their use as binders. The ability of an alkyd to dry to a hard and durable film through the autoxidation process. The amount

of double bonds presents in the oil, as assessed by the iodine value [24,25] is related to this. The drying time is also affected by the length of alkyd resin oil, as shown in Table 8. It was discovered that the 40 and 50 % oil lengths reached surface-dry conditions in less than 2 hours; however, the 60 % oil length took two hours. The LDROAR/LTPOAR resins > MDROAR/MTPOAR resins > SDROAR/STPOAR resins were in the increase drying order. This finding is in line with that of Anshuman *et al.* [26] and others [27-29], who established the relationship between unsaturation and the drying time. As the length of the oil diminishes, the alkyd's solubility in the solvent (xylene) reduces, increasing the amount of solvent embedded in the alkyd resin.

Table (8). Drying test.

Coating Property	LDROAR	MDROAR	SDROAR	LTPOAR	MTPOAR	STPOAR
Dry-Through (hrs)	2hrs	1hr	30 mins	1hr.30mins	50 mins	25mins
Set to touch (hrs)	10mins	8mins	4mins	5mins	5mins	3mins
Surface-dry (hrs)	1hr	40mins	20mins	45mins	39mins	20mins

Table 9 shows the solubility of the six oil-based alkyd resins produced. To determine solubility, 0.5 g of each resin was dissolved in 10 ml of ethanol, methanol, xylene and acetone; all alkyds were not soluble in ethanol, methanol, xylene except in acetone. The xylene and alcohol resistance of the synthesized alkyd resins was generally good, as shown in Tables 9. The acetone resistance test failed all of the produced alkyd resins, indicating that alkyds have low acetone resistance in general. The alkyds' poor acetone resistance could be related to the presence of ester groups, which are known to be sensitive to hydrolysis due to acetone's aprotic nature. The effects of alkyd oil length type on the media resistance of the alkyd resins studied were not found.

Table (9). Alkyd resins solubility in solvents.

Medium	LDROAR	MDROAR	SDROAR	LTPOAR	MTPOAR	STPOAR
Ethanol	PS	PS	PS	PS	PS	PS
Methanol	PS	PS	PS	PS	PS	PS
Xylene	SS	SS	SS	SS	SS	SS
Acetone	S	S	S	S	S	S

Soluble(S), Sparingly Soluble (SS), Partial Soluble (PS)

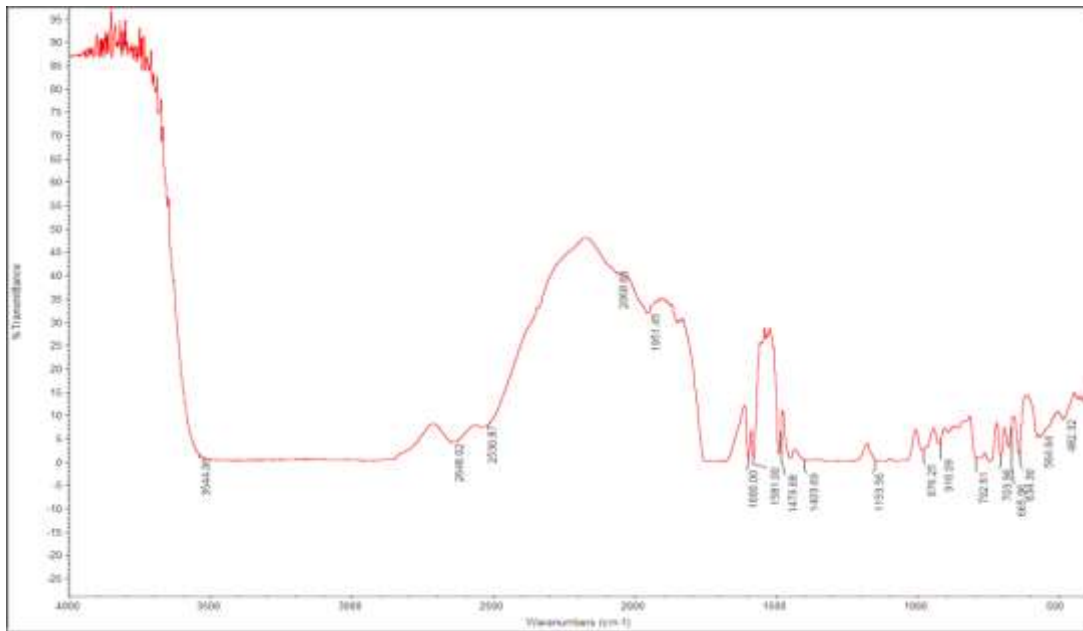
Long and medium oil alkyds with higher oil content have been found to dry slower at first because long oil alkyds have more thermosetting than short oil alkyds. As a result, gloss retention in long oil alkyd is greater than in medium and short oil alkyd (Table 10)

Table (10). Solidification Time.

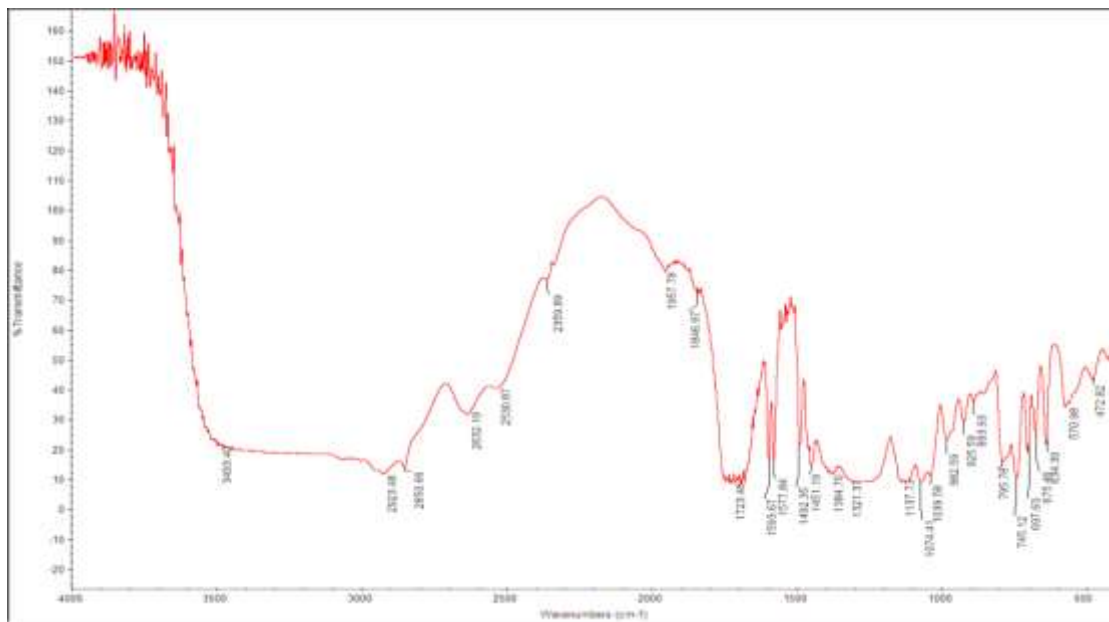
	LDROAR	MDROAR	SDROA	LTPOA	MTPOAR	STPOAR
Time (mins)	4.22	3.20	1.80	3.59	2.10	1.39.

The Fourier Transform Infrared Spectroscopy Analysis

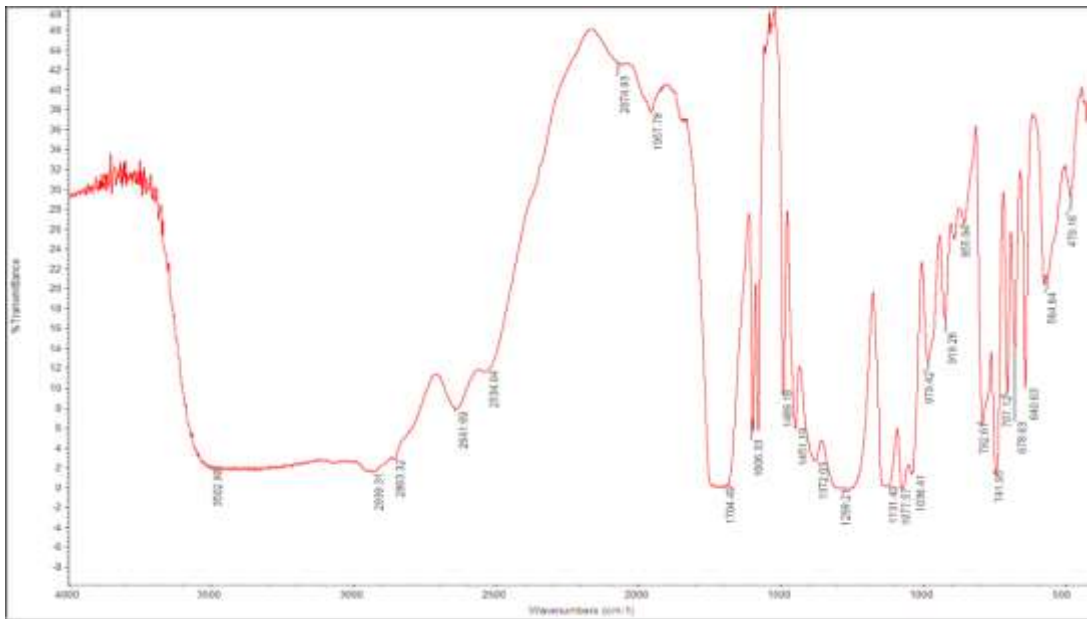
The Fourier Transform Infrared Spectroscopy (FTIR) spectral shown in Figures 10 (a-f) represents Long, Medium and Short alkyd resins obtained from DRO and TPO. The spectral data is a useful tool for identifying the typical functional groups, which are useful for determining the functional groups and organic compounds present in any sample (Table 11).



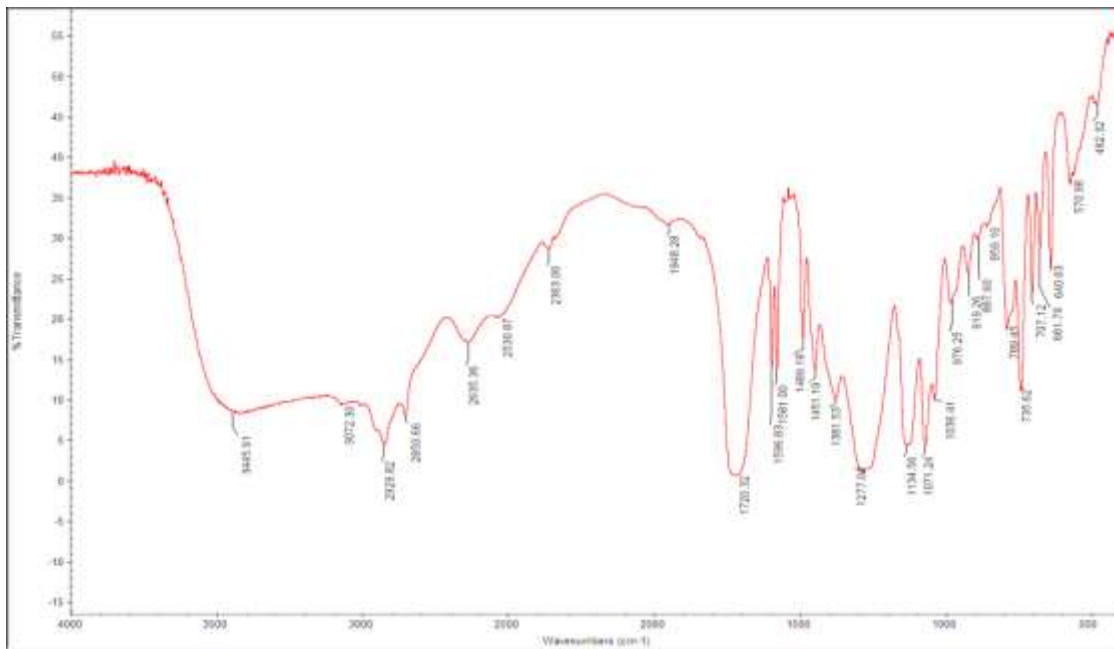
(a). FTIR spectral of long *delonix regia* –alkyd resin (LDROAR).



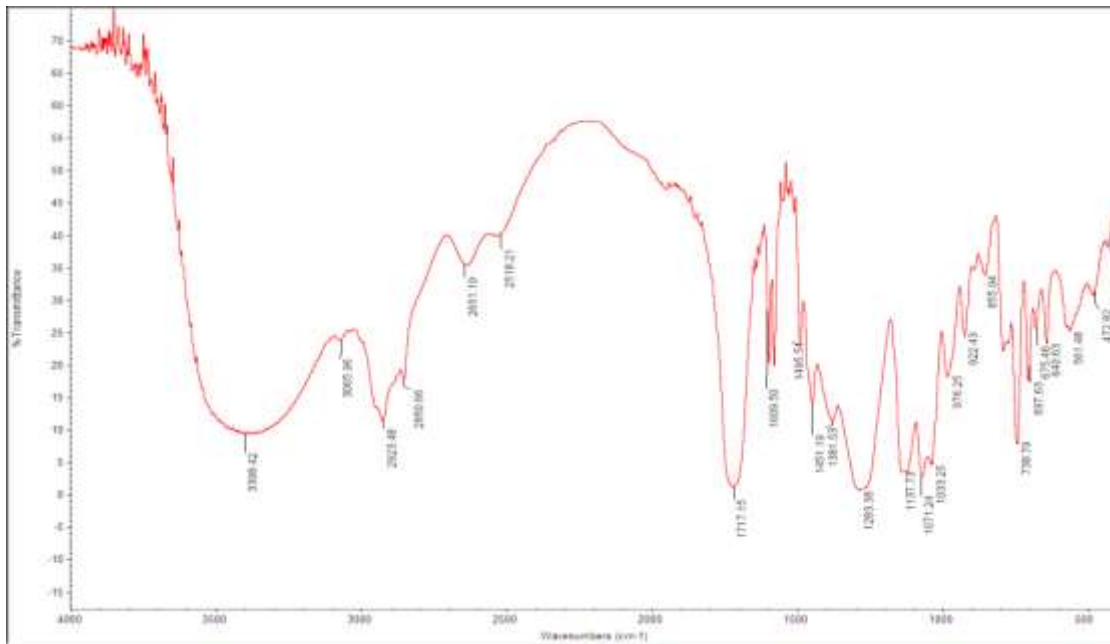
(b). FTIR spectral of long *theventia peruviana* –alkyd resin (LTPOAR).



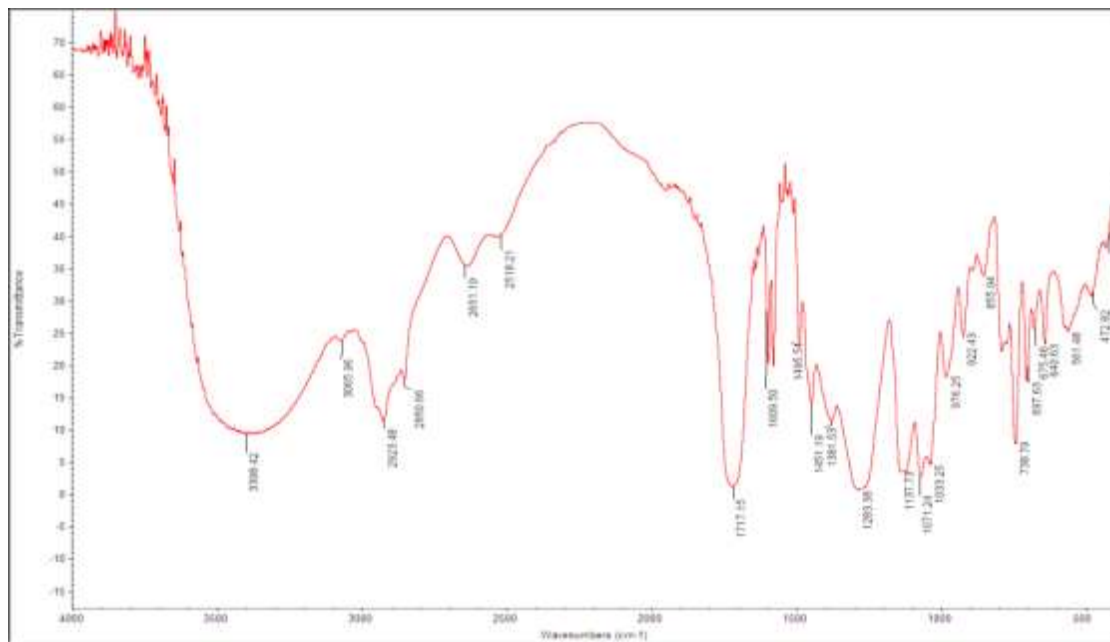
(c). FTIR spectral of medium *delonix regia* –alkyd resin (MDROAR).



(d). FTIR spectral of medium *theventia peruviana* –alkyd resin (MTPOAR).



(e). FTIR spectral of short *delonix regia* –alkyd resin (SDROAR).



(f). FTIR spectral of short *theventia peruviana* –alkyd resin (STPOAR).

Figure 10. FTIR spectral of (a). long *delonix regia* –alkyd resin (LDROAR), (b). long *theventia peruviana* –alkyd resin (LTPOAR), (c). medium *delonix regia* –alkyd resin (MDROAR), (d). medium *theventia peruviana* –alkyd resin, (MTPOAR), (e). short *delonix regia* –alkyd resin (SDROAR), (f). short *theventia peruviana* –alkyd resin (STPOAR).

Table (11). Interpretation of FTIR spectra of alkyd resins compare with a commercially alkyd resin.

Observed vibration band/peaks Frequency (cm-1) *	CPARL	DROAR	MDROAR	SDROAR	LTPOAR	MTPOAR	STPOAR	Functional Goup
3600-3200	3500.92	3544.06	3502.90	3487.07	3493.40	3445.91	3398.42	O-H stretching
2950-2840	2880.78	-	-	2939.31	2923.48	2923.48	2929.82	C-H aliphatic
1750-1720	1727.31	-	1704.49	1736.15	1723.48	1720.32	1717.15	C=O stretching
1465-1440	1452.45	-	1451.19	1444.85	1451.19	1451.1	1451.19	-CH ₃ bend
1390-1365		-	-	1372.03	1372.03	1384.70	1381.53	O-CH ₂ bend
1680-1600	1131.29	1600.00	1606.49	-	-	1609	-	C=C (alkene)
1200-1020	1131.29	1153.56	1131.40	1131.7	1137.73	1134.56	1137.73	O-CH ₃ bend
1035-1149	113.1.94	-	-	-	1039.58	1036.41	1033.25	C-O bend
980-965	976.25	979.42	979.42	-	976.25	976.25	-	RCHCHR trans
730-650	703.96	640.63	640.63	643.30	-	640.63	-	RCHCHR cis
500-730	881.50	703.96	707.12	703.96	-	707.12	-	C-Cl
490-620	449.43	564.64	564.64	551.98	570.98	570.98	561.4	C-I

Note: *CPAR-Commercial alkyd resin.

The values of 1750-1720 C=O stretching (ester). The 2939.31, 2923.48, 2923.48, 2929.82 and 2923.48 cm⁻¹ were for C-H aliphatic stretching respectively. The peak at 1153.56, 1131.40, 1131.73, 1137.73, 1134.56, and 1137.73 cm⁻¹ showed O-CH₃ bend. The hydroxyl (OH) group of unsaturated fatty acids was ascribed to the broad bands around 3544.06 cm⁻¹. SCN and CN stretches of thiocyanate and nitrite compounds were in the peak values of 2930.93 cm⁻¹ and 2353.23 cm⁻¹, respectively. The C-O stretch of cyclic ester compounds of monoglyceride was allocated to the medium bands at around 1704.49, 1736.15, 1723.48, 1720.32 and 1717.15cm⁻¹ values compared with peaks reported by Asiagwu *et al* [1]. Further significant peaks, such as of 1372.03, 1372.03, 1384.70 and 1381.53cm⁻¹, for O-CH₂ bend(Pavia *et al.*, 1996), also peak of 976.25, 979.42, 79.42, 976.25, 976.25, for RCHCHR trans and 703.96, 640.63, 640.63, 643.30, 640.63RCHCHR cis, 703.96, 707.12, 703.96, 707.12 C-Cl and 564.64, 564.64, 551.98, 570.98, 570.98 and 561.4 cm⁻¹ for C-I bend were observed respectively, demonstrating that all long oil resins made with different polybasic acid catalysts have the same chemical nanostructures.

4. Conclusions

In this work, the delonix regia and thevetia peruviana seed oils were extracted and the extracted oilseed yields of 52.71 and 40.55 % (w/w) were observed, respectively, indicating that the oilseed plant could be classified. Based on the iodine value, the physicochemical characterization of the oils revealed that they are semi-drying in nature and acceptable for alkyd resin synthesis. The oil's physicochemical properties were compared with the ASTM standards and were found to be in good accord and were utilized to make alkyd resin with varying oil

concentrations (short, medium and long alkyd resins). The alcoholysis-polyesterification processes were used to make six classes of alkyd resins and their properties were appropriately determined.

The creation of a high molecular weight alkyd was discovered by determining the extent of the reaction and the related average degree of polymerization. Based on the degree of conversion of the reactant with reaction time, the alkyd resins had outstanding resistance to acid, brine, and water but were only moderately resistant to alkali in terms of 25, 40 and 60 % oil length. Their resistance to various service media is comparable with oil-observed paint's resistance to these media when used as a surface covering. In terms of the 60 % oil length, characterization of paint based on alkyd resin derivatives yielded values for surface drying of less than 2 hours. As a result, seed oil-modified alkyd resin from delonix regia and thevetia peruviana are good materials for oil paint formulation.

References

- [1] M. Boruah, P. Gogoi, B. Adhikari, and S. K. Dolui, "Preparation and characterization of Jatropha Curcas oil based alkyd resin suitable for surface coating," *Prog. Org. Coat.*, vol. 74, no. 3, pp. 596–602, 2012.
- [2] N. O. Shaker and A. M. A. Elsayy, "Preparation, characterization and evaluation of jojoba seed oil-modified alkyd resins," *Pelagia Research Library*, vol. 3, no. 5, pp. 1157–1162, 2012.
- [3] J. S. Basha *et al.*, "Potential of utilization of renewable energy technologies in Gulf countries," *Sustainability*, vol. 13, no. 18, p. 10261, 2021.
- [4] M. D. H. Beg, M. R. Islam, B. Ahmed, R. M. Yunus, and A. Abdullah, "Optimization of reaction parameters of esterification on the synthesis of palm oil-based alkyds using response surface methods," *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 634, p. 012038, 2019.
- [5] B. Müller and U. Poth, *Coatings Formulation: An International Textbook*. Vincentz Network, 2019.
- [6] B. Kyenge, B. Anhwange, J. Ageh, and G. Igbum, "Comparative analysis of soyabean seed oil modified alkyd resin and epoxidized soyabean seed oil modified alkyd resin," *International Journal of Modern Organic Chemistry*, vol. 12, pp. 66–71, 2012.
- [7] K. A. Ibrahim, K. A. Abu-sbeih, I. Al-Trawneh, and L. Bourghli, "Preparation and characterization of alkyd resins of Jordan Valley tomato oil," *J Polym Environ*, vol. 22, no. 4, pp. 553–558, 2014.
- [8] B. Sujata *et al.*, "Biodiesel production from mixed oils: A sustainable approach towards industrial biofuel production," *Chemical Engineering Journal Advances*, no. 10, 2022.
- [9] C. K. Patil, J. H. D. Baik, and G. V. V. Hong, "Non-edible vegetable oil-based polyols in anticorrosive and antimicrobial polyurethane coatings," *Polymers*, vol. 13, 2021.
- [10] J. O. Ezeugo, "Optimization of polycondensation process of alkyd resin synthesis from modified Picralima nitida seed oil suitable for surface coating of metal," *World Scientific News*, pp. 62–90, 2019.
- [11] I. H. Ifijen, M. Maliki, I. J. Odiachi, O. N. Aghedo, and E. B. Ohiocheoya, "Review on solvents based alkyd resins and water borne alkyd resins: Impacts of modification on their coating properties," *Chem. Afr.*, vol. 5, no. 2, pp. 211–225, 2022.
- [12] N. S. Dubovik, D. S. Matyas, S. A. Dubovik, and R. A. Lyubushkin, "Method for controlling reaction mass parameters during the synthesis of alkyd resins," *J. Phys. Conf. Ser.*, vol. 1926, no. 1, p. 012009, 2021.
- [13] S. Zhong, J. Li, L. Yi, Y. Cai, and W. Zhou, "Cross-linked waterborne alkyd hybrid resin coatings modified by fluorinated acrylate-siloxane with high waterproof and anticorrosive performance," *Polym. Adv. Technol.*, vol. 30, no. 2, pp. 292–303, 2019.
- [14] M. A. Mutar and N. M. A. Hassan, "Synthesis and characterization of new alkyd resins (short , medium and long) based on sunflower oil and linoleic acid as(short , medium and long) based on sunflower oil and linoleic acid as binder for paints," *J. Chem. Petrochemical Technol*, vol. 7, pp. 1–16, 2017.
- [15] "Official Method of Analysis: Association of Analytical Chemists," *AOAC*, pp. 121–130, 2012.
- [16] R. Salata Ryan, B. Pellegrine, and D. Mark, "Synthesis and properties of high solids triethoxysilane-modified alkyd coatings," *Progress in Organic Coatings*, vol. 133, pp. 340–349, 2019.
- [17] A. Chowdhury, S. K. Singh, S. Marker, and P. Anthony, "Structural and thermal characterization of synthesized polyester resin based on a new linseed variety (shuats-alsi 2)," *Rasayan j. chem.*, vol. 13, no. 03, pp. 1834–1841, 2020.
- [18] L. Pagnin, R. Calvini, R. Wiesinger, J. Weber, and M. Schreiner, "Photodegradation kinetics of alkyd paints: The influence of varying amounts of inorganic pigments on the stability of the synthetic binder,"

Front. Mater., vol. 7, 2020.

- [19] U. E. Igbo, C. C. Igwe, E. Akubueze, O. R. Ishola, A. O. Odusote, and A. O. Oyewole, "Utilization of beniseed oil for the production of alkyd resin," *Journal of Applied Chemistry*, vol. 7, no. 5, pp. 104–106, 2014.
- [20] G. O. Otabor, I. H. Ifijen, F. U. Mohammed, A. I. Aigbodion, and E. U. Ikhuoria, "Alkyd resin from rubber seedoil/linseed oil blend:A comparative study of the physiochemical properties *Heliyon* 5," vol. 5, pp. e01621-1621, 2019.
- [21] G. Oladipo, C. Ighodalo, I. Eromosele, and O. Folarin, "Formation and characterization of paint based on alkyd resin derivative of *Ximeniaamericana*(wild olive) seed oil," *Environment and Natural Resources Research*, vol. 3, no. 3, pp. 113–116, 2013.
- [22] H. Aghaie, A. Ilkani, and C. S. Sadat, Utilization of Soyabean Fatty Acid for Synthesis ofg Alkyd Resin and Comparison of Properties with Other Vegetables Oils. *Department of Chemistry*. Tehran, Iran, 2012.
- [23] H. I. Ikhazuagbe and N. N. Adachukwu, "Selected Under-exploited Plant Oils in Nigeria: A Correlative Study of their Properties," *Tanzania Journal of Science*, vol. 46, no. 3, 2020.
- [24] G. C. Dhruvabijaya *et al.*, "*Juglansregia* L," *Juglansregia L. Juglandaceae Chapter*, 2021.
- [25] I. Ratajczak, M. Woźniak, K. Szentner, M. Babicka, J. Jencyk, and B. Mazela, "Aminosilane binding to wood substance through an alkyd resin," *J. Wood Chem. Technol.*, vol. 40, no. 1, pp. 73–79, 2020.
- [26] M. Anshuman, K. Piyush, N. Madiha, and C. S. Umesh, "Extraction and Characterization of *Sesbaniacannabina* (Retz.) Pers. (Dhaincha) Seed Oil for Potential Engineering Applications," *Journal of Oleo Science*, vol. 70, no. 6, 2021.
- [27] S. Sumaya *et al.*, "Palm oil derived aloud resin synthesis, catalyzed by SrO/Sr(OH)₂ Nanoparticles," *Journal of Critical Review*, vol. 7, 2020.
- [28] A. N. Nkwor and P. O. Ukoha, "Evaluation of the leather fatliquoring potential of sulphonated*Afzeliaafricana* aril cap oil," *Heliyon*, vol. 6, 2020.
- [29] G. O. Otabor, I. H. Ifijen, F. U. Mohammed, A. I. Aigbodion, and E. U. Ikhuoria, "Alkyd resin from rubber seed oil/linseed oil blend: A comparative study of the physiochemical properties," *Heliyon*, vol. 5, no. 5, p. e01621, 2019.