Application Of Luffa Aegyptiaca Seed Oil In The Synthesis Of An Oil Modified Alkyd Resin

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Abstract: In this study, alcoholysis-polyesterification method was used to investigate the utilization of Luffa aegyptiaca seed oil in the synthesis of an oil-modified alkyd resin. The extracted oil seed gave an oil yield of 43.2% (w/w) and the physico-chemical characterization of the oil indicated it is a semi-drying oil with iodine value of $132.28 \text{ g } I_2 100 \text{ g}_1$. Three grades of alkyd resins were formulated at 40% (I), 50% (II) and 60% (III) oil lengths (concentrations) respectively. The progress of the reaction was monitored by determining the acid value from the aliquots taken from the reaction mixture at various time intervals. The extent of reaction (%P_{av}) determined at the initial stage of the reaction ranged from 64.52 to 78.60%, indicating an encouraging degree of conversion. The corresponding average degree of polymerization (D_p) ranged from 2.7 to 4.7 which indicated the formation of high molecular weight alkyd resin. Physico-chemical characterization of the synthesized Luffa aegyptiaca seed oil-modified alkyd resin (LASOMAR) gave iodine value of 72.1 ± 0.74 , 78.1 ± 1.05 and $83.2 \pm 1.0 \text{ g } I_2$ 100 g⁻¹ for samples I, II and III respectively. The corresponding saponification values were found to be 205.2, 295.8 and 332.5 mg KOH g⁻¹. Samples I and II of the Luffa aegyptiaca seed oil-modified alkyd resin (i.e. 40% and 50% oil length) were resistant to Brine and Water with little resistance to Alkali.

Keywords: Alkyd, Oil-modified resin, polymer Binder, Alcoholysis method, Polymerization, Condensation.

I. INTRODUCTION

Natural oils are derived from vegetable's oils such as linseed oil, Soya bean oil, Tung oil, dehydrated castor oil, fish oil, casein; and animal fats using several methods [10]. Most natural oils qualify as bio-based products as defined by the United States Secretary of Agriculture in 'farm security and Rural Investment Acts' (2002). There are limited numbers of naturally occurring vegetable oils (triglycerides). Chemically these are glycerides (esters) of unsaturated organic higher fatty acids like oleic, linoleic, linolenic and ricinoleic acids with alcohols.

The properties of an oil depend on the proportion of these different long chain fatty acid radicals in the molecule [10].

Among these oils, castor oil is the only commercially available natural oil that is produced directly from a plant source. Other natural oil sources require chemical modification of the oils available from plants before use [4]. The ultimate use of natural oils is in the production of polymeric materials which are used industrially for different applications especially in the paint and coating industries [24]. For example, natural oil binders are used as bio-resins for vehicle or binders in the formulation of surface coatings in paint manufacturing industries [10].

A binder (vehicle) is a film-forming oil, to which other liquids are added. These liquids are diluents or thinners which are generally volatile liquids like mineral turpentine or organic solvents like xylol or acetone [17]. However, the diluents are classified as Volatile Organic compounds, VOCs that are known to be environmentally unfriendly [21]; [22]. Therefore, there is need to formulate paints that are environmentally friendly. Formulations such as modified alkyd paints, dimethylol urea formaldehyde paints and amino resins paints among others have been found to be environmentally manageable [22].

Environmental pollution has been a matter of great concern worldwide, hence the need to check it [8]. Furthermore, there is need for the reduction of the demand of non-renewable fossils currently used in the production of polyol or natural oil binders and different chemicals in many industries of the world, thereby promoting the use of biomass (bioresins) due to the fact that the widely used non-renewable source might be exhausted sooner or later [5].

Luffa aegyptiaca is fast becoming an indispensable crop because of its very wide industrial applications and many medicinal properties [13]. Hence the need to explore the full potentials of this renewable resource as an environmentally friendly substitute for the surface coatings industry.

This study aimed at synthesizing an oil-modified alkyd resin from *Luffa aegyptiaca* seed oil (LASO).

II. MATERIALS

Luffa aegyptiaca seed was obtained from Girei local Government area of Adamawa State. Oil was extracted from the seed using soxhlet extraction apparatus. Analytical grade reagents of Dam's Iodine, potassium iodide (5%, 10%), Sodium thiosulphate, starch solution (1%), Sodium hydroxide, Phthalic anhydride, brine solution, xylene, Glycerol, N-Hexane and methanol. The glass wares used were 500 ml three neck round bottom flask fitted with dean and stark, water bath, magnetic stirrer, thermometer, reflux condenser and heating mantle.

III. METHODOLOGY

Pretreatment and extraction of *Luffa aegyptiaca* seed oil was carried out as outlined by [7]. The physico-chemical properties of the oil were determined using American Oil Chemists Society Methods (AOCS, 1996).

Three grades of alkyd resins were prepared from Luffa aegyptiaca seed oil, phthalic anhydride and glycerol as illustrated in Table 2. The two staged Alcoholysispolyesterification reaction normally begins with the formation of monoglyceride, usually by heating a mixture of oil, glycerol and a small amount (1g) of CaO (as catalyst) in a 500 ml three neck round bottom flask fitted with dean and stark [2]. This mixture was heated at 200 °C by heating mantle for 2 hours after which a small quantity of the aliquot was taken to test its solubility in methanol which confirms the formation of monoglyceride. The second stage of the reaction is characterized by lowering the temperature to 180 °C during which a measured quantity of phthalic anhydride was added and this was followed by addition of xylene which will help remove the water of esterification. The temperature was raised to 230 - 250 °C and the reaction lasted for another 1 hour [7]. Aliquots are withdrawn from the reaction mixture at 30 minute intervals to determine drop in acid value and volume of water evolved as the reaction progressed. The reaction was stopped when the acid value attained the value of about 10 mg KOH g-

¹ and the alkyd resin was allowed to cool. The acid value of in-process samples taken at intervals were determined by titrating with a 0.1M KOH solution to the phenolphthalein end point after dissolution in a mixture of toluene and ethanol (1:1). The acid value was related to the extent of the reaction P_{av} and the average degree of polymerization D_p by the relationship:

$$P_{av} = \frac{C_0 - C_t}{C_0}$$
 and $D_p = (1 - P_{av})^{-1}$

And the rate equation for the reaction is expressed as: $(1 - P_{av})^{-1} = C_0 (kt + 1)$

Where:

 C_0 = initial acid value, C_t = acid value after time, t, k = rate constant and t = time of reaction.

Statistical packages for social sciences (SPSS) were used to arrive at the mean, standard deviation and correlations.

Ingredient (100g)	Alkyd Samples		
	I	II	III
Luffa aegyptiaca seed oil			
(LASO) (%)	40	50	60
Phthalic anhydride (%)	36.31	30.2	24.09
Glycerol (%)	23.69	19.8	15.91
Mole ratio	1:2	1:1.4	1:0.9
Alkyd Constant	1.02	1.02	1.02

Table 1: Recipes for the formulation of oil-modified alkyd resin

IV. RESULTS AND DISCUSSIONS

A. RESULTS

a. CHARACTERIZATION OF LUFFA AEGYPTIACA SEED OIL (LASO)

The physico-chemical properties of LASO are given in Table 3.1

Properties	LASO
Colour	Dark-yellowish
	Green
Oil yield (%) (w/w)	43.2
Specific gravity (g cm ⁻³ , 30 °C)	0.87
Refractive index (30 °C)	1.464
Viscosity (mm ² /s, 30 °C)	33.28
Saponification value (mg KOH g ⁻¹)	178.5
Iodine Value (g I ₂ 100 g ⁻¹)	132.28
Acid value (mg KOH g ⁻¹)	20.62
Free fatty acid (%)	9.07
Peroxide Value (m Eq kg ⁻¹)	280
Moisture content	4.62
Table 2 2. Dresents the ETIP Absorpti	on hand of Luffa

Table 3.2: Presents the FTIR Absorption band of Luffa aegyptiaca seed oil (LASO)

b. CHARACTERIZATION OF LUFFA AEGYPTIACA SEED OIL-MODIFIED ALKYD RESIN (LASOMAR)

Table 3.3 Presents the physico-chemical properties of *Luffa aegyptiaca* seed oil modified alkyd resins samples I, II and III.

		Alkyd Samples	
Property	Ι	II	III
Colour	Dark-Brown	Dark-Brown	Dark-brown
Refractive index (30 °C)	1.500	1.494	1.491
Viscosity (mm ² /s, 30 °C)	Very viscous	Very viscous	Very viscous
Acid value (mg KOH g ⁻¹)	6.34	10.37	7.85
Iodine Value (g I ₂ 100 g ⁻¹)	72.1 ± 0.74	78.1 ± 1.05	83.2 ± 1.0
Saponification Value (mgKOH g ⁻¹)	205.2	295.8	332.5
Drying time (h)			
Surface Dry	<3	<3	<3
Dry Through	Overnight	Overnight	Overnight
Density (g/cm ³)	1.68	1.04	1.51
Specific gravity	1.91	1.18	1.72

Figures 1, 2, and 3 gives the variation in acid value with time, variation in extent of reaction with time and variation in average degree of polymerization with time respectively for LASOMAR sample I, II and III respectively.

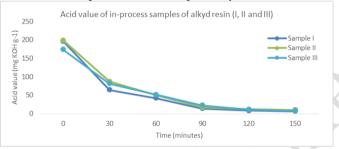


Figure 1: variation in acid value with time Table 3.4 gives the Variation in acid value of in-process samples of alkyd resin (I, II and III), extent of reaction (%P) and corresponding average degree of polymerization (D) with reaction time.

ALKYD SAMPLES									
		Ι			II			III	
TIME (MIN)	AV	$\%P_{av}$	$\mathbf{D}_{\mathbf{p}}$	AV	$\%P_{a\nu}$	D_p	AV	$\% P_{av}$	D_p
0	196.35	-	-	199.15	-	-	173.91	-	-
30	64.52	67.14	3.04	86.95	56.34	2.29	81.35	53.22	2.14
60	42.07	78.57	4.66	50.91	74.44	3.91	51.20	70.56	3.40
90	14.00	92.87	14.03	16.83	91.55	11.83	22.44	87.10	7.75
120	8.42	95.71	23.31	12.16	93.89	16.37	10.66	93.87	16.31
150	6.34	96.71	30.40	10.37	94.79	19.19	7.85	95.49	22.17

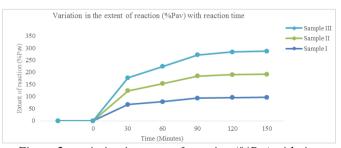


Figure 2: variation in extent of reaction (% P_{av}) with time

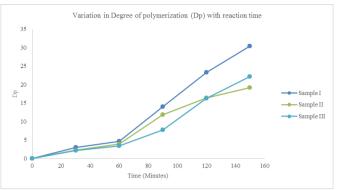


Figure 3: variation in Degree of polymerization (D_p) with time Table 3.5 presents the FTIR absorption bands of Luffa aegyptiaca seed oil-modified alkyd resin (LASOMAR) samples I, II and III.

Compound	Frequencies,	Assignment					
wavenumber (cm ⁻¹)							
C=O	1747.39; 1854.52;	Carbonyl					
	1747.91	group of ester					
$C=C-C^{a}$ (aryl)	1853.77;1991.35;	Aromatic ring					
	1853.72	Stretch					
$-CH_2$	2857.08-2664.73;	$-CH_2$					
	2687.81-2368.53;	Symmetric					
	2928.82-2684.84 (Broad)	vibration					
$-CH_3$	2367.13; 2368.53;	Asymmetric					
	2368.71 (sharp)	stretching of -					
		CH_3					
C-H	1465.28; 1463	Bending of					
		Methylene (C-					
		H)					
-OH	3480.28; 3480.47;	Hydroxyl					
	3480.78 (Broad)	groups of					
		carboxylic					
		acid,					
		-OH Stretch					
$-CH_2$	719.80; 744.17	-CH ₂ Rocking					
		mode					

Table 3.6 gives presents the Chemical resistance of Luffa aegyptiaca seed oil-modified alkyd resin

071			
	Media	Time (Hour)	Effect on film
	Distilled water	18	А
	Brine	18	А
	3M NaOH	8	В
		16	с
		24	d

Key:

a = No effect

b = Whitening

c = Blistering

d = Film removal

A. DISCUSSIONS

a. DISCUSSION ON THE PHYSICO-CHEMICAL PROPERTIES OF LASO

Oil was extracted from the seeds of Luffa aegyptiaca plant. The extracted oil seed gave an oil yield of 43.2 % (w/w), this is comparable to the 45.7% that was earlier

reported for Ximenia americana seeds by [7]. The acid value of 20.62 mg KOH g⁻¹ suggests low in-vivo lipolytic activities in the seed, and indicates a good non-degraded state of the oil and falls within limits for industrially useful oils [25]. Williams (1966) reported that low acid values for oils are useful in the manufacture of paints and varnishes. The free fatty acid concentration of 9.07% is low, this is consistent with the low acid value observed. This is in close agreement with the 8.07% also reported by [7] for Ximenia americana seed oil. The saponification value of the LASO is 178.5 mg KOH g ¹. [3] reported the saponification value of Luffa aegyptiaca seed oil as 167 mg KOH g⁻¹. This suggests that the seed oil contain high molecular weight of fatty acid and may be suitable for preparation of soap. The iodine value of 132.28 g $I_2 100 g^{-1}$ is comparable to the one obtained from literature reports ([20]; [6]), and thus indicates that the oil is a semidrying oil suitable for the Synthesis of alkyd resin, formulation of liquid soap and hair shampoo. The seed oil has a peroxide value of 280 mEq kg⁻¹. The value indicates that the oil is susceptible to oxidative rancidity [11]. The oil has a viscosity value of 33 mm²/s, 30 °C, this indicates that the oil is viscous at ambient temperature. The refractive index of the seed oil is 1.464, it is in close range with the refractive indices of other drying oils which are comparatively in the range 1.46 to 1.54 and this suggest that it can be used as a test for purity and as an aid to identification (BS 6900). The specific gravity of the oil is 0.87 g cm⁻³. It is the lowest in comparison with 0.93, 0.912 and 0.931 reported for Castor oil, XASO and linseed oil respectively [20]; [7] and [18]. The moisture content of Luffa aegyptiaca Seed oil was found to be 4.62. This suggests that the seed has low susceptibility to microbial attacks [23].

b. DISCUSSION ON THE FTIR SPECTROSCOPIC ANALYSIS OF LASO

Fourier transform infrared spectroscopy (FTIR) is one of the powerful tools used in identifying and investigating the presence of functional groups in a molecule, as each specific chemical bond often has a unique energy absorption band and can obtain structural and bond information on a complex to study the strength and the fraction of hydrogen bonding and miscibility of the compounds [14].

From Table 3.2. The broad peak at 3475.34 cm⁻¹ suggests the presence of hydroxyl groups (-OH) of carboxylic acid (– COOH) in the oil as indicated by (John, 2000). The broad peak in the range 2937.12 – 2857 cm⁻¹ suggests -CH₂ symmetric vibration. The sharp peak at 2367.80 cm⁻¹ suggests asymmetric stretching of –CH₃, confirming alkyl groups suspected in the oil as indicated by [12]. The peak at 1748.07 cm⁻¹ suggests the stretching of -C=O (carbonyl group) of the aldehyde. Bending of –CH₂ group was observed at 1460.92 cm⁻¹ while that of CH₃ was observed around 1374.92 cm⁻¹ respectively. The peaks in the range 1238.42 – 1114.72 cm⁻¹ suggests the stretching of C-O. In-plane and out-of-plane stretching vibrations was observed at 721.61 cm⁻¹ which indicates –CH₂ rocking mode [14].

c. DISCUSSION ON THE PHYSICO-CHEMICAL PROPERTIES OF LUFFA AEGYPTIACA SEED OIL-MODIFIED ALKYD RESIN

From Table 3.3, the physico-chemical properties of Luffa aegyptiaca seed oil- modified Alkyd resins. Three grades of alkyds were formulated at 40% (I), 50% (II) and 60% (III) oil length (concentration) with various amounts of phthalic anhydride and glycerol employing the two-stage alcoholysispolyesterification method as was described by [7]. The reaction was monitored by determining the acid value for the aliquot of the reaction mixture at various time intervals as the reaction progressed. It was observed that as the oil concentration increases, the total volume of water evolved for each alkyd sample increases. This is corroborated by the changes in acid value with reaction time as it was shown in table 3.4 and figure 1 respectively. There was initial sharp decrease in acid value followed by a more gradual decrease. Similar observations had been reported by [20] and [11]. The decrease in acid value is explained as arising from the selective reactivity of primary and secondary hydroxyl groups of glycerol with carboxyl groups of phthalic anhydride [19]. [19] reported also that the primary hydroxyl groups of the monoglyceride reacts more rapidly than the secondary hydroxyl groups with phthalic anhydride. It is established that at a temperature of about 160 °C, primary hydroxyl groups of glycerol react rapidly with carboxyl groups of phthalic anhydride, while secondary hydroxyl groups react at temperature above 230 °C [9]. Thus, the initial marked decrease in acid value observed for all the three alkyd samples may be due to increase in the rate of esterification accompanying the rapid increase in temperature to 230 °C. In confirmation of the above, the acid value for all the samples decreased with increase in amount of oil used (oil length), consistent with high amounts of di- and triglycerides present in the reaction mixture and the concentration of the phthalic anhydride [20]. Characterization of the alkyd gave iodine value of 72.1 \pm 0.74, 78.1 \pm 1.05 and 83.2 \pm 1.0 (g I₂ 100 g⁻¹) for samples I, II and III respectively. The corresponding saponification values are 205.2, 295.8 and 332.5 mg KOH g These are all in close agreement with the values reported by [7]; [2].

Table 3.4 presents the variation in acid value of in-process samples of alkyd resin (I, II and III), the corresponding extent of reaction ($^{\circ}P_{av}$) and average degree of polymerization (D_p) with reaction time. There is no common standard to compare alkyd resins. Each alkyd resin has its own properties. According to [19], the alkyd resin that has acid number of less than 15 is suitable for application of paint because it indicates a considerable amount of conversion.

From figure 2, the first region (i.e. at 30 minutes' reaction time) of the plot indicates the stage where the primary hydroxyls of glycerol reacted with carboxyl groups of phthalic anhydride leading to formation of linear chains [19]. After the primary hydroxyl groups have been esterified, there was an abrupt drop in acid value and the reaction rate decreased until a temperature was reached when the secondary hydroxyl groups begin to react [25]. Consequently, the increasing D_p of the alkyd chains (From figure 3) results in increasing viscosity

of the reaction medium. Similar observations have been reported earlier [7]; [1].

d. DISCUSSION ON THE FTIR SPECTROSCOPIC ANALYSIS OF LASOMAR

From Table 3.5, the peaks at 1747.39 cm⁻¹, 1854.52 cm⁻¹ and 1747.91 cm⁻¹, suggests the presence of carbonyl (C=O) group which could be for ester for samples I, II and III respectively as indicated by [14]. The spectrum also shows peaks at 1853.77 cm⁻¹, 1991.35 cm⁻¹ and 1853.72 cm⁻¹ for samples I, II and III respectively which could be ascribed to the aromatic ring ester bands as according to [14]. The broad peaks in the ranges 2857.08 - 2664.73 cm⁻¹. 2687.81 - 2687.81 2368.53 cm^{-1} , and $2928.82 - 2684.84 \text{ cm}^{-1}$ for samples I, II and III respectively suggests -CH₂ symmetric vibration. While the sharp peaks at 2367.13 cm⁻¹, 2368.53 cm⁻¹ and 2368.71 cm⁻¹ suggests the asymmetric stretching of -CH₃ (alkyl) groups present in samples I, II and III respectively. Bending of methylene (C-H) groups suspected in the resins could be ascribed to the peaks that were observed at 1465.28 cm⁻¹ and 1463 cm⁻¹ in samples I and III respectively. The broad peaks at 3480.28 cm⁻¹, 3480.47 cm⁻¹ and 3480.78 all suggests characteristic hydroxyl (-OH) groups of carboxylic acid (-COOH) present in samples I, II and III respectively. In-plane and out-of-plane stretching vibrations were observed at 719.80 cm⁻¹ and 744.17 cm⁻¹ which suggests -CH₂ rocking mode for samples I and III. These spectrums are all in close agreement with what has been reported by [15].

e. DISCUSSION ON THE CHEMICAL RESISTANCE OF LASOMAR

The Chemical resistance of *Luffa aegyptiaca* seed oilmodified alkyd resin was presented in Table 3.6. The result shows that the resin was unaffected by brine and distilled water. However, the resin shows poor resistance to Strong alkali. According to [16], the presence of alkali hydrolysable ester groups in the resin is responsible for the poor resistance of alkyd film to alkali mediums.

V. CONCLUSION

An oil-modified alkyd resin was synthesized by alcoholysis in this research using *Luffa aegyptiaca* seed oil, Glycerol, Phthalic anhydride and CaO (as catalyst). The physico-chemical properties and high chemical resistance of the alkyd resin film indicates it's a potential binder for the Surface coating industries and showed that they were promising in formulating of paint.

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