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SYNTHESIS AND CHARACTERIZATION OF ALKYD RESINS FROM RUBBER SEED/SOYBEAN OIL BLENDS

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Abstract

The polymerization and properties of environmentally friendly resin binders applicable in the surface-coating industry were studied. Rubber seed oil (RSO) and RSO blended with 10% and 20% soybean oil (SBO)-based alkyd resins were synthesized by alcoholysis-polyesterification of the oil with glycerol and phthalic anhydride. Physicochemical properties (such as colour, specific gravity, acid value, saponification value, iodine value, and drying schedule) of the alkyd resins were determined to establish the possible industrial potential of the resins. The saponification value and iodine value ranged from 250.19 mgKOH/g to 279.55 mgKOH/g and 29.58 gI$_2$/100g to 33.77 gI$_2$/100g respectively. The drying schedule and chemical resistance of the blended alkyd resins to water, salts, acids, and alkalis were also studied. The blended resins were found to be resistant to water, salt, and acid media, except for alkali media. The colour properties were enhanced as the percentage of the blend increased. FT-IR spectroscopic study of the oil and alkyd resin samples further corroborates our findings. Therefore, the potential of rubber seed oil blended with soybean oil to produce light-coloured alkyd resins could be exploited as raw materials for the Nigerian surface coating industry.

1.0 INTRODUCTION

Alkyd resins are a group of polyesters synthesized by the polycondensation reaction of triglyceride oils (or fatty acids), dibasic acids (or acid anhydrides), and polyols with hydroxyl functionality greater than two [1]. They are widely used as important binders in the coatings and paint industries. Recent estimations revealed that alkyd resin contributes approximately 70% of the conventional binders used in surface coatings today [2]. They can also be used as raw materials for the production of industrial and household finishes [3]. The versatility of alkyd resins as vehicles for coatings is largely due to their unique properties such as film hardness, durability, gloss and gloss retention, resistance to abrasion, and compatibility with other resin systems [4]. The properties of the resins depend on the drying ability of the triglyceride oil used during their manufacture.

Drying oils owe their ability to polymerize after application on materials surfaces to form tough, adherent, impervious, and abrasion resistant films to the level of unsaturation of the triglyceride oils [5]. Similarly, The fatty acid chains in the polymer backbone are known to improve the flexibility,

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adhesion, and chemical resistivity of alkyd resin [6]. Vegetable oils like soybean oil, linseed, soybean, canola, tall oil, coconut oil, and sunflower oil are conventional oils used as benchmarks for alkyd resin synthesis[7, 8]. Similarly, non-conventional vegetable oils such as rubber seed oil, jatropha seed oil, karanja oil, nahar seed oil, African locust bean seed oil, etc. are well reported as alternative non-traditional vegetable oils with properties similar to the traditional oils used as potential sources of triglyceride oil for alkyd resin synthesis [7, 9-11]. Other, non-conventional drying oils are available locally but have remained untapped and are the subject of investigation.

Alkyd resins synthesized using drying oils had superior appearance, chemical resistance, and outstanding physical properties. Several vegetable oils reported in literature used in alkyd resin production are semi-drying in nature. The properties of the resins are mainly dependent on the type of drying oil. However, several diverse and stringent end-use applications of alkyd resins, such as farter drying and improved colour have necessitated special treatment of oil to meet these requirements. Consequently, physical and chemical modifications of vegetable oils in order to enhance their quality have been carried out over the years. For instance, non-drying oils can be transformed into drying oils through modification by dehydration [12]. Similarly, monomer modification through malenisation, fumarisation, acrylation of the oils[10], and resin from oil blends [13] have all been reported.

In our previous studies [14], we reported the potential of rubber seed oil (RSO) in the production of alkyd resins suitable as binders in solvent-borne and water-reducible coatings. This paper reports the modification of RSO to enhance its use in producing bright-coloured coatings by partially blending with soybean oil. Previous studies have shown that rubber seed oil alkyd resin is darker in colour [10]. The coloured nature of the resin may not be suitable for the formulation of non-pigmented coatings where brighter colour is a necessity. Although, several reports have shown that RSO based alkyd resins are valuable raw materials in formulating dark coloured non-pigmented coatings [14]. Since soybean oil is brighter in colour (light yellow) can be used in formulating bright coloured alkyd resin. Soybean oil is an edible oil that cannot solely be used in the production of alkyd resin since higher food prices pose an immediate threat to the food security of poor net food buyers [15]. In a recent study, alkyd resin prepared from RSO blended with linseed oil (LSO) was reported [13]. The result from that study showed that alkyd resin samples[13] synthesized from RSO blended with LSO exhibited good resistance to brine, water, and acid with improved drying properties.

Similarly, studies on the properties of alkyd resins synthesized from blends of RSO and SBO [16]. Both studies showed that alkyd resins with better properties can be produced by blending other oil types with RSO. In both studies, lead oxide was used as a catalyst, and the studies focused on blending RSO with a high proportion of SBO or LSO [13, 16]. This contradicts the objective of production cost reduction, where demand for more expensive traditional oil could be reduced. Also, the alcoholysis stage in alkyd resin production is normally catalyzed by Brønsted bases such as lithium, sodium hydroxides, calcium, and lead oxides [17, 18]. Lead oxide was reported to be detrimental to the production of bright coloured resins [17]. Also, a higher percentage of SBO used as a blend will put unnecessary strain on the demand for food commodities. The implications, opportunities, and limits of crops applicable as resources for food, industrial, and technical productions and possible consequences of the global food price spike are reviewed [15, 19]. Although, there have been knowledge gaps over the years on the global capacity for sustainable plant-based polymer production while maintaining food security. Therefore, the effect of rubber seed oil blended with a small proportion of soybean oil on the physicochemical properties, drying performance, and chemical resistance of its derived resins were investigated.

2.0 MATERIALS AND METHOD

2.1 Materials

Rubber seeds were collected from the plantation of Rubber Research Institute of Nigeria, Iyanomo, Benin City and soybean seeds were obtained from Oba market, Benin-City, Nigeria. The oils were soxhlet extracted from the milled seed using *n*-hexane as the extracting solvent. Laboratory grade glycerol and analytical grade phthalic anhydride were all obtained from Aldrich Chemicals. All of the chemicals were used without further purification.

Table 1: Recipe for the Formulation of Alkyd Resins

Component (g)	I	II	III	IV
RSO	178.85	166.50	148.00	-
SBO	-	19.20	38.40	178.85
Glycerol	76.64	76.64	76.64	76.64
Phythalic anhydride	119.94	119.94	119.94	119.94

2.2 Methods

2.2.1 Physicochemical properties of rubber seed oil and soybean oil

The physicochemical properties properties, like specific gravity (Ta 1b-64), acid value (Cd 3a- 63), saponification value (Cd 3–25) and iodine value (Cd 1–25) of rubber seed oil (RSO) and soybean oil (SBO) were determined by American Oil Chemists' Society (AOCS) methods [20].

Alkyd resin preparation

Four samples of alkyd (I–IV) comprising of (I- 100% RSO, II: 90% RSO + 10SBO, III: 80% RSO + 20% SBO, IV: 100% SBO) were prepared with RSO, SBO and RSO blend with SBO, glycerol and phthalic anhydride according to the formulation in Table 1. All the alkyds were formulated to an alkyd constant of about 1.0. Two different samples of RSO blends were prepared by thoroughly mixing RSO with (A) 10% and (B) 20% SBO (w/w).

The reactions were carried out in a 1-L three-necked-round-bottom flask. The flask was fitted with a mechanical stirrer and a Dean–Stark apparatus carrying a water-cooled condenser. Nitrogen gas was bubbling through the reaction medium to create an inert atmosphere. Xylene (10% w/w) was employed as the cooking solvent. In a typical reaction, RSO, a catalyst (calcium oxide) and glycerol were heated with continuous stirring. The reaction temperature was raised and maintained at 230 ^{0}C for about 2.5 h under nitrogen. The reaction was stopped when the sample of the alcoholysis product formed a clear solution in anhydrous methanol (1:3 w/v oil:methanol). The reaction was allowed to cool to about 120°C, phthalic anhydride was added, and the temperature was quickly raised to 250 ^{0}C for the polycondensation reaction. The efflux was drained into the Dean–Stark apparatus, in which xylene was separated from the water of condensation and returned to the reaction flask through an overflow point. The progress of the reaction was monitored by the determination of the acid values of aliquots of the reaction mixture until an acid value below 10mg KOH/g was achieved [14].

Preparation of alkyd coatings

The RSO, SBO and blended alkyd samples were thinned to 50% with xylene in which cobalt and calcium drier (0.5% weigh of alkyd) were added and applied on thin glass panels at room temperature. The drying schedule of the samples in terms of time of set-to-touch and dry through was determined. The films were considered dry through when there is no loosening, detachment, wrinkling, or distortion of the film [10].

Chemical Resistance of the Alkyd Resins

The chemical resistances of the dried films were determined using the dried film on the test panel prepared above. The test panels were assessed for their resistance to different service media by immersion method for a period of 24 hours. The service media used were distilled water, 5% (w/v) sodium chloride solution, 0.1M KOH and 0.1M H_2SO_4 solutions [10] (ASTM D1308-57).

FTIR spectroscopy

Infrared spectra of the RSO, SBO and its derivates were recorded with SHIMADZU, FTIR-8400S spectrophotometer. The samples were spread over NaCl cells, and their spectra were recorded. IR spectra were recorded in the range 4000–400 cm^{-1}.

3.0 RESULTS AND DISCUSSION

3.1 Physiochemical Properties of the Oils

The physicochemical properties of the oils (RSO and SBO) are listed in Table 2. The percentage oil yield of RSO is higher than SBO. The colour of RSO was yellow compared with that of the light-yellow obtained SBO. The value of the specific gravity of RSO is 0.908, which is comparable to the value of 0.901 observed for SBO. The acid value indicates the level of free fatty acids formed from hydrolytic decomposition of glycerides to free fatty acids [21]. High levels of free fatty acids are usually caused by age, storage conditions, and an increase in the extraction temperature of oils. The high content of free fatty acids in oils has been attributed to a reduction in oil quality [21, 22]. The acid value of 48.28, obtained for RSO, was higher than 0.44 obtained for SBO in this study. These values translate into 23.68 and 0.20 free fatty acid of RSO and SBO respectively. The acid values obtained in this study show that both RSO and SBO are good starting materials for the production of oil paints and varnishes.

Table 2: Physico-Chemical Properties of the Oil

Properties	RSO	SBO
Yield (%)	41.30	15.82
Colour	Yellow	Light Yellow
Specific Gravity [30^{0}C]	0.908	0.901
Acid Value [mg KOH/g]	48.28	0.44
Saponification Value [mg KOH/g]	185.30	202.20
Iodine Value [gI_2/100g]	122.45	127.56

Table 3: The Main FT-IR Peaks and their corresponding functional group of RSO and SBO

Frequency (cm^{-1})		Assignment	Remark
RSO	SBO		
3010	3009	C-H	Stretching frequency of non-conjugated unsaturation
2854	2854	C-H	Stretching frequency of alkane
1747	1745	C=O	Stretching frequency of carbonyl group
1653	1654	C=C	Stretching frequency of alkene

1464	1464	-C-H	bending frequency of unsaturated alkene
1240	1230	-C-O	Stretching bending
1165	1161	C-O	Stretching frequency of esters
1099	1099	-C-O	Stretching frequency of ester
723	723	$-(C-C)_n-$	bending frequency of saturated carbon atom

Similarly, the iodine values for the oils were 122.45 and 127.56 gI_2/100g for RSO and SBO, respectively. The iodine value recorded in this study was also indicative of the high unsaturated fatty acid content of both oils. The iodine value is a measure of the unsaturation of triglyceride oil [23] and is useful for predicting the drying properties of oils. The iodine value of the RSO obtained in this study was slightly lower than that recorded for SBO. The iodine values of both RSO and SBO reported in this study fall within the range of semi-drying oil, which is a potential raw material in the paint, resin, polyol, and lubricating oil industries [12]. This observation is further corroborated by the FTIR study of both RSO and SBO, which supports the presence of unsaturated acyl groups in the oils presented in Figure 1. The results in Table 3 show that the functional groups present in RSO are similar to those in other vegetable oils [24].

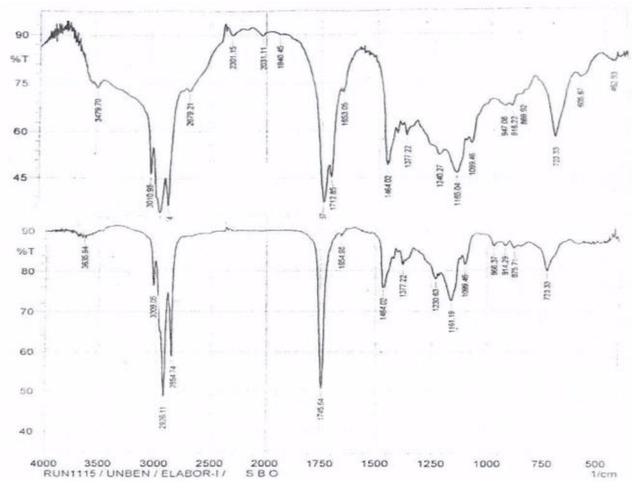

Figure 1: IR spectra of RSO and SBO

The characteristic absorption frequency observed between 3010 and 3009 cm^{-1} of the oils showed C-H stretching vibration of the olefinic functional group, and the stretching frequency of alkene C=C was observed at approximately 1654 cm^{-1} [24]. The level of unsaturation as well as the iodine value of vegetable oil have been determined using the ratios of intensities of the observed (νC=C) bands at 1650 cm^{-1} and δCH2 at 1464 cm^{-1}, as well as ν=C-H at 3010 cm^{-1} and 2853 cm^{-1}, which are well reported in the literature [25, 26]. Soybean oil is a commercial vegetable oil used as an industrial standard for alkyd resin production, where a moderate drying rate and good colour retention are desired [27]. The physicochemical characteristics of RSO and SBO obtained in this study are similar and comparable to those of the seed oils currently used in the commercial production of alkyd resins.

3.2 Properties of Alkyd Resins

The acid values, reaction time and water evolved for the finished alkyds were monitored until the values fell below 10 mgKOH/g to ensure complete reaction with corresponding higher degree of polymerization, as presented in Table 4. Alkyd resins with acid numbers less than 10 mg KOH/g are suitable for paints, as reported in the literature [10, 14]. The acid values of the prepared resins ranged from 3.4 to 4.8 mgKOH/g. The reaction time decreased with increasing SBO content. However, the reverse is true for the acid value, except for the neat RSO alkyd. The esterification reaction time ranged from 4 h 50 min to 5 h 15 min. It appears that the time required for the complete reaction of the SBO-based alkyds was lower than that of the RSO-based alkyd. The average volume of water evolved during the polyesterification reaction was approximately 14.4 ml (Table 4). The variation in the volume of water evolved during the polyesterification reaction was attributed to the variation in the agitation rate of the reaction mixture and the contribution of glycerol and xylene [28]. The volumes of water released during esterification are generally lower than the calculated 22.0 ml of water evolved during the polycondensation reaction of these resins. This observation is attributed to the difficulty of the polycondensation reaction hardly gets to completion before termination of the reaction [28].

Table 4: Parameters of the alkyds

Samples	Sample Code	Reaction Time (Hour)	Acid Value (mgKOH/g)	Water Evolve (ml)
RSO	PI	5hr 15mins	4.10	15.0
90%RSO+10%SBO	PII	5hr 10mins	3.40	13.5
80%RSO+20%SBO	PIII	5hr 10mins	4.85	12.0
SBO	PIV	4hr 50mins	4.74	17.0

Table 5: The main FT-IR Peaks and their corresponding functional group alkyd resins

Frequency (cm^{-1})				Assignment	Remark
PI	PII	PIII	PIV		
3497	2431	3497	2431	O-H	Stretching frequency hydroxyl group
3009	3009	3009	3009	=C-H	Stretching frequency of unsaturation
2854	2854	2854	2854	-C-H	Stretching frequency of alkane
1732	1732	1732	1732	C=O	Stretching frequency of carbonyl group
1599	1600	1599	1600	C=C	Stretching frequency of alkene
1284	1284	1284	1284	C-O	Stretching frequency of ester
856	866	856	866	C-H	Stretching frequency of alkane

3.3 Structural Analysis of Alkyd Resins

The IR spectra of the RSO alkyd compared with those of the blends and neat SBO alkyd are presented in Figure 2. The major functional groups are listed in Table 5. The IR spectra of the resins were identical and similar in all aspects. Consequently, the spectra support the presence of ester, aromatic, and hydroxyl groups and olefinic double bonds in the alkyd resins. The IR spectra of the alkyd resins (Figure 2) show a strong band at 1732 cm^{-1} characteristic of the stretching frequency of the carbonyl group. A sharp absorption band (doublet) appearing around 1599 and 1578 cm^{-1} are characteristic of the unsaturation of the aromatic group on the alkyd resin backbone. The broad band between 3431 and 3510 cm^{-1} present in all the alkyd resins confirms the presence of OH groups and has been shown to be characteristic of alkyd resins [1, 17]. This hydroxyl band is completely absent in both RSO and SBO, which are used as starting materials in the synthesis of resins. A strong absorption due to the asymmetrical C-O-C stretching band at 1284 cm^{-1} and a symmetrical band at 1072cm^{-1}, attributed to the stretching of the ether group, were also observed [1, 29]. Similarly, out-of-plane aromatic C-H bends around 773 and a ring C-C band at 705 were observed [29]. The additional bands within this region are due to ring-bending vibrations [29].

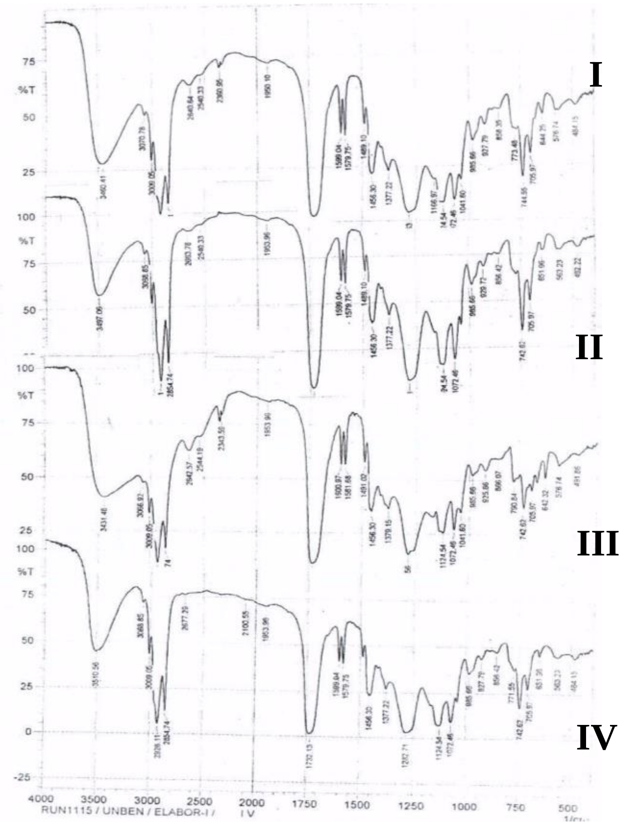

Figure 2: IR spectra of RSO alkyd (sample I), RSO alkyd blends (samples II and III) and SBO alkyd (sample IV).

3.4 Physiochemical Properties of Alkyds

The physicochemical properties of these alkyds are listed in Table 6. The pure RSO alkyd resin was darker than the alkyd resins prepared from RSO blended with SBO. The colour of the SBO alkyd resin was brighter than that of the corresponding RSO alkyd resin and its blends, indicating that blending RSO with SBO enhanced the colour properties of the alkyd samples. The saponification values of the samples of RSO alkyd increased with the SBO content and were lower than the saponification value of the pure SBO alkyd resin. In addition, the saponification values of the PI and PIV resins were higher than those of the corresponding starting oils. The alkyd resin PIV exhibited the highest saponification value (279 mgKOH/g). The saponification value of the alkyd resins increased marginally with an increase in the blend content. Similarly, the iodine values of samples PI to PIII increased with the SBO content and were higher than that of the pure SBO alkyd resin. The iodine values of the alkyds were lower than those of the oils, with values of 29.58 $gI_2/100g$ PI, 31.58 $gI_2/100g$ for PII, 33.77$gI_2/100g$ for PIII and 30.21$gI_2/100g$ for PIV respectively.

The drying properties of the alkyd films were measured in terms of set-to-touch, dry-to-touch, and dry-through. The application of resins as binders is due to their ability to dry, where a thin layer of alkyd resin film dries faster upon exposure to air [30]. Therefore, thin films of the alkyd samples were air-dried in this study. The drying properties (i.e., set to touch and dry to touch) increased as the SBO content in the resin increased (Table 6). Therefore, the RSO alkyd resin exhibited the lowest drying ability. The drying time of the synthesized resins was enhanced when blended with soybean oil compared to the neat RSO alkyd resin. The fast-air-drying ability observed in this study can be attributed to the mechanisms of drying, which are mainly through evaporation of the solvent and oxidative polymerization by the crosslinking of double bonds bond [1, 30]. This observation was supported by the iodine values of the alkyd resin samples used in the coating.

Table 6: Physico-Chemical Properties of the alkyds

Properties	**PI**	**PII**	**PIII**	**PIV**
Colour	Dark Brown	Brown	Brown	Light brown
Acid value (mgKOH/g)	4.10	3.40	4.85	4.74
Saponification value (mg/KOH/g)	250.19	252.68	261.51	279.55
Iodine value($gI_2/100g$)	29.58	31.58	33.77	30.21
Drying property				
Set to touch (min)	21	17	13	10
Dry to touch (min)	65	60	56	45
Dry through	Overnight	Overnight	Overnight	Overnight

PI: Pure RSO alkyds, PII: 90% RSO + 10% SBO alkyd (blend), PIII: 80% RSO + 20% SBO alkyd (blend), PIV: Pure SBO alkyd.

3.5 Chemical Resistance of the Alkyd Resins

The performance characteristics of the alkyd resins in different service media are presented in Table 7. The results showed that none of the alkyd resins were affected by acid and salt solutions. Similar results were obtained in water, except for the neat SBO alkyd (PIV), where a slight whitish colouration that disappeared upon drying was observed. All the alkyd films presented in Table 7 showed poor resistance to alkali, which can be attributed to the hydrolyzable ester groups in the alkyd film backbone. It can be deduced that alkyd resin samples have potential applications in which resistance to alkali is not the main requirement [10]. RSO seed oil alkyds and their blends (Table 7) exhibit excellent resistance to water, acid, and salts and have potential for applications in which resistance to water, acid, and salts is necessary. RSO alkyd resins and their blends can be used for corrosion protection and to prolong the lifespan of marine vessels, offshore structures, and general household coatings and paintings.

Table 7: Chemical resistance of the alkyd film to different media

Media	PI	PII	PIII	PIV
Water	1	1	1	2
NaCl (5%)	1	1	1	1
0.1M H_2SO_4	1	1	1	2
0.1M KOH	4	4	4	4

1: No visible change; 2: Slight whitening which disappear on drying; 3: Wrinkled; 4: Film removed.

4.0 CONCLUSION

The results of this study showed that RSO blended with 10% and 20% SBO enhanced the physicochemical properties and performance characteristics of the finished alkyd resins derived therefrom. These resins are good precursors for surface-coating formulations used in paints, varnishes, and coatings. Future studies should use other non-conventional seed oils, such as jatropha seed oil and African locust bean seed oil, which have similar properties to soybean oil as a blend. This deliberate effort in the long run could expand demand and increase prices for non-conventional seed oil, which may present better opportunities for agricultural and rural development.

5.0 ACKNOWLEDGEMENT

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6.0 CONFLICT OF INTEREST

The authors declare that they have no conflict to interests.

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