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Synthesis, Characterization and Performance Evaluation of Breadfruit Seed Oil Modified Alkyd Resin

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ABSTRACT

Long and medium oil length alkyd resins were synthesized using breadfruit seed oil by the alcoholysis process. Oil extracted from the seeds of breadfruit were refined and characterized in terms of acid, iodine and saponification values. Glycerol and phthalic anhydride were used as the polyol, and polybasic acid respectively in the synthesis of the resins. Soyabean oil modified alkyd resin served as the reference alkyd resin. The drying properties of the alkyd resin investigated showed that the synthesized alkyds dried satisfactorily at alkyd film thickness of 0.10 mm, irrespective of alkyd resin oil length. While the synthesized alkyds showed good water, and acid resistance. All the synthesized alkyds failed the alkali resistance tests. The adhesion properties of the synthesized alkyd samples were good. Similarly, there was no mildew formation in any of the synthesized alkyd samples, an indication of bio-stability of the synthesized alkyds. The alkyd samples exhibited good storage property, an indication of better performance in coatings, and hence compared favourably with the reference oil, soyabean. This study has highlighted the utility of breadfruit seed oil in the coatings industry.

Key words: Alkyd resin, alcoholysis, breadfruit seed oil, phthalic anhydride

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INTRODUCTION

The preparations and applications of alkyd resins have attracted the attention of alkyd scientists who have been working not only to elucidate the structure of alkyd resins, but also improve their properties as non-volatile binders or vehicles in the manufacture of paints and vanishes [1].

Over the years, there has been an increase in the cost of basic raw materials for alkyd resin synthesis. This has in turn caused an astronomical increase in the cost of finished coating products such as paints, varnish, etc. For alkyd resin to still maintain a pride of place in the surface coatings industry in the face of stiff competition from the use of low cost synthetic resins, alkyd chemists have intensified search for alternative source of raw materials for alkyd resin production. The drying phenomenon associated with alkyd resins based on soyabean oil was reported by Williams and Cock [2]. Variations in drying time of seemingly identical soyabean oil alkyd resins, especially when pigmented, were traced to the oxidation-inhibiting effect of the variable tocopherol contents of natural oils. The tocopherol found, could be successfully destroyed by oxidizing agents in the presence of Cu, Co or Fe^{3+} as catalysts at temperatures greater than 50ºC. Ghaffar [3] prepared alkyd resin from Egyptian apricot oil blended with coconut and linseed oil. A stoving vanishes based on the apricot oil-melamine formaldehyde resin was applied on glass plates and baked for 30 minutes at 130ºC. The results showed that the Egyptian apricot oil could be used as a substitute in stoving non-drying short alkyd resins.

Synthesis of alkyd resins using a partially, and fully epoxidized vernonie oil, linseed oil, and soyabean oil, was reported by Muturi-Nwangi *et al* [4]. The fully epoxidized linseed oil exhibited the least yellowing property, and this yellowing of a vegetable oil could be controlled by reducing the unsaturation level. Gogte and Sarwadakar [5] prepared alkyd resin from fractionated rice bran oil in combination with castor oil. The result showed that rice bran oil permitted the use of maleic acid without the danger of gelation of the prepared resin. Igwe and Ogbobe [6] studied the synthesis of alkyd resin using melon seed, and rubber seed oils. Results obtained revealed that rubber seed oil could be substituted for linseed, and soyabean oils in the synthesis of both long and medium oil-length alkyd resins. Similarly, melon seed oil was found to be a substitute for linseed oil, and soyabean oil in the synthesis of long oil-length alkyd resins.

Studies on the enhancement of alkyd resins using methyl esters of rubber seed oil was reported by Ikhuoria *et al* [7]. Methyl ester of rubber seed oil was more promising in the production of alkyd resin than rubber seed oil. Aghaie *et al* [8] prepared alkyd resin using soyabean oil fatty acid, and compared the properties of the resin with the resin prepared using other vegetable oils. There was a decrease in acid value of the resin, while the viscosity of alkyd resin increased in the condensation polymerization reaction.

Synthesis of soybean seed oil modified alkyd resin, and epoxidized soybean seed oil modified alkyd resins was reported by Kyenge *et al* [9]. The epoxidized soybean seed oil had more potential for use in surface coating formulation based on the drying properties of the films. The alkyd resins obtained from both the soybean modified, and epoxidized soybean modified alkyd resins exhibited excellent chemical and alkali resistance. Shaker *et al* [10] prepared jojoba seed oil modified alkyd resins by the alcoholysis-polyesterification processes. The prepared resins were resistant to water, alkali, acids and solvents. The resins had good impact, adhesion, and flexibility properties. Synthesis and physico-chemical properties of air-drying alkyd resin based on palm oil were reported by Uzoh *et al* [11]. The resin synthesized had high drying time in the presence of a drying agent, and exhibited excellent adhesion to substrates. Oladipo *et al* [12] studied alkyd resins based on derivative of Ximenia Americana (Wild Olive) seed oil. 40, 50, 60 and 70% oil length alkyd resins were prepared and their performance characteristics were evaluated. Results indicated that the paints based on 40, and 50 % oil length had surface dry and tack free dry properties in less than 2, and 5 hours respectively, exhibiting good drying properties. Three medium oil length alkyd resins of cotton seed oil prepared using different polybasic acids via, the alcoholysis process have been reported by Isaac and Nsi [13]. Property investigations on the prepared resins showed that the paint samples were highly resistance to 5 % NaCl, and poorly resistant to 0.1 M KOH. Succinic acid alkyd paint sample was poorly resistant to distilled water. Blaise *et al* [14] prepared blends of palm oil and long oil-modified alkyd resins. These blends contained qualities of palm oil ranging from 10 to 50%. Results obtained showed that alkyd resins blended with palm oil exhibited high drying time. However, the blend exhibited low viscosity and density, which indicated good rheological properties. In a continuous bid to formulate a cost effective paint, efforts have been made to investigate the utilization of local breadfruit seed oil in the synthesis of alkyd resins.

MATERIALS AND METHOD

Materials

The breadfruit Seed (*Treculia Africana*), from which the breadfruit oil was extracted was sourced from Ideato South Local Government Area, within the South-Eastern region of Nigeria. The oil was solvent extracted from the seeds of breadfruit tree. Soyabean Oil (*Glycine Soja*) which served as a reference alkyd resin was obtained from Taraku Mills Limited, Benue State, Nigeria. Fuller's Earth, a product of Fulmont Ltd, England consisting mainly of alumina silica ($Al_2O_3.2SiO_2.2H_2O$) impregnated with iron, magnesium and calcium was used. Lead and cobalt naphthanates with metal content of 35.30 % Pb, and 6.81 % Co were used as driers.

Extraction of the Vegetable Oils

The vegetable oil (breadfruit seed oil) was extracted from their oil bearing seeds by soxhlet extraction using n-hexane. In this method, the oil-bearing seeds were shelled, and sun dried. The dried seeds were ground to fine meal using a grinder. Weighed quantities of ground meal of breadfruit seeds were placed in a 500 cm^3 round-bottomed flask. 150 cm^3 of n-hexane was poured into the flask which was later placed on a heating mantle. The soxhlet apparatus was set-up for the extraction of oil from the ground seeds. The extracted oil breadfruit seeds were weighed, and the oil yield was noted.

Alkali Refining of Oils

The extracted oils from breadfruit were alkali refined to reduce the percentage of their free fatty acids contents. The method of Cocks and Rede [15] was adopted for the alkali refining of the oils of breadfruit seeds. The amount of NaOH used in the refining process was in 10% excess of that actually required to neutralize the free fatty acids in the oil. This was calculated using the Cocks and Rede [15] formula.

$$\frac{\text{1M NaOH required + 10\% excess = weight of oil taken x \%FFA x 1.1x1000cm}^3}{\text{100 x Mw x (molarity of}} \quad (1)$$

Where Mw = weight average molecular weight of oil.
%FFA = Percentage of free fatty acid present in the oil.

Purification of the Alkali Refined Oils

The alkali refined oil was purified to free the oil from soap residues and un-reacted NaOH. In this method, the oil was dissolved in a reasonable amount of petroleum benzene (b.pt 60-80^oC) and filtered using Buckner funnel to remove the soap and other impurities. The oil solution was then subjected to vacuum distillation at a temperature of 50^oC in a heating mantle to remove the solvent from the oil. The recovered oil was washed with boiling water in a separating funnel under careful shaking to avoid emulsification of the oil. The washing was stopped when the aqueous extract was clear and neutral to litmus. The washed oil was subjected to vacuum distillation at 70^oC to remove any trace of water in the oil.

Bleaching of Oils

In this method, 400g of the alkali refined oil of breadfruit oil was mixed with 60g of Fuller's earth (15wt % based on oil) in a three-necked round bottomed flask. The flask with its content was immersed in an oil bath maintained at a constant temperature of 80^oC, and heated under vacuum. The mixture was stirred continuously with an electrical stirrer, and the bleaching lasted for 40 minutes. At the end of the bleaching, the contents of the flasks were filtered hot under vacuum using a Buckner funnel. Filtration was repeated with a whatman Grade 1 filter paper to obtain optically clear oil.

Characterization of Oils

American Standard Testing Methods (ASTM) was used to characterize the oil for acid value (ASTM D 1639 -61, 1966), and iodine Value Determination, (ASTM D 1959 – 69). The American Oil Chemist Society method (AOCS Cd 3-25, 1966) was used to determine the saponification value of the oil.

Standardization of Sodium Thiosulphate Solution

25cm^3 of standard potassium dichromate solution (containing 4.904 g $K_2Cr_2O_7$/litre of diluted water) was pipette into a conical flask and diluted with 50cm^3 of distilled water. 10% KI was added, followed by 10cm^3 concentrated hydrochloric acid (HCl sp.gr. 1.19), and the solution was shaked to mix. The liberated iodine was titrated with $Na_2S_2O_3$ solution while shaking constantly until the yellow colour had almost disappeared. 1 – 2 cm^3 of starch indicator solution was added, and titration continued until the blue colour had disappeared. The molarity of the $Na_2S_2O_3$ was calculated as follows:

$$(\text{Vol. of } Na_2S_2O_3) \times (\text{Molarity of } Na_2S_2O_3) = (\text{Vol. of } K_2Cr_2O_7) \times (\text{Molarity of } K_2Cr_2O_7) \quad (2)$$

Synthesis of Alkyd Resins

The formulations used for the synthesis of long and medium oil length of breadfruit seed oil modified alkyd resins are shown in Table 1.

Table -1 Formulation for the Synthesis of Alkyd Resin

Ingredients(g)	Long Oil Length Alkyd Resin	Medium Oil Length Alkyd Resin
Vegetable Oil	153.6	103.4
Litharge (PbO)	3.84	2.54
Glycerol	30.7	30.7
Phthalic Anhydride	74	74

PROCEDURE

A750 cm^3 three necked, round bottomed resin kettle heated with a heating mantle was equipped with a variable speed stirrer, a dean and stark water collector, reflux condenser, nitrogen inlet tube, a thermometer and a sampling device. The kettle which had been flushed with nitrogen was charged with 153.6g of the refined oil (breadfruit seed or soyabean) for the synthesis of the long oil modified alkyd resins or 103.4 g of the refined oils for the synthesis of the medium oil alkyd resins. The oil was heated to the reaction temperature of 245 ± 2^oC, 3.84g of the catalyst, litharge (PbO), was added immediately for the synthesis of the long oil alkyds or 2.59 g (PbO) for the synthesis of medium oil alkyds, followed by 30.7g of glycerol which was added slowly from a dropping funnel over a period of 2 minutes with vigorous agitation. Samples were withdrawn at intervals for methanol tolerance test. The alcoholysis stage was completed when 1 volume of the alcoholysis mixture was soluble in 2 volumes of anhydrous methanol. At the end of the alcoholysis, 74g of phthalic anhydride was added all at once, and the nitrogen flow was increased. The mixture was heated and stirred at the reaction temperature. The progress of the reaction was followed by a frequent check on the acid number of the reaction mixture. The reaction was stopped on attainment of acid number of less than 10, and the contents were allowed to cool by dropping the reaction kettle in a bath of cold paraffin oil.

Preparation of Resin – Drier Mixture

119 g of the synthesized long oil alkyd resin or 84 g of the synthesized medium oil length alkyd resin was accurately weighed out into a 500 cm^3 beaker and dissolved in 70 g or 139 g by weight of xylene respectively. This gives a resin solution of 64% or 38% solid respectively. 1.75 g lead naphthanate and 0.9 g cobalt naphthanate driers for long oil length alkyd resin or 1.2 g lead naphthanate and 0.6 g cobalt naththanate driers for medium oil length alkyd resin

were added. The resin – drier mixture was mixed thoroughly using a mechanical stirrer. The amount of driers added represent 0.50 wt. % Pb and 0.05 wt. % Co which are the amounts of these metals normally used in the surface coatings industry [16]. Film thicknesses of resins investigated (in mm) are: 0.10, 0.40, 0.70, and 1.0.

Performance Evaluation of Synthesized Alkyd Resins

The alkyd resins synthesized were evaluated for their drying properties, resistance to chemicals, adherence to surfaces, and mildew formation using standard methods.

RESULTS AND DISCUSSIONS

Characterization of the Vegetable Oils

The properties of the vegetable oils are presented in Table 2. The oil yields are not very considerable. However, Kyari [17] considered an oil yield of 26 – 42% to be reasonable for commercial purposes. Differences in oil yields arise because these oil seeds are natural products which are subject to variations in properties as a result of factors such as temperature, soil variety, seed variety and maturity, post-harvest storage, among the other factors.

Acid value is a property that is very useful in assessing the quality of oilseeds during extraction. Therefore, good storage of oilseeds and sun-drying prior to extraction of oil is very important to prevent the oilseeds from deterioration, and minimize the production of free fatty acids in oilseeds. The observed variations in acid values from Table 2 could have arisen from a number of factors, notably, seed variety and post-harvest storage, age of the oil seeds prior to oil extraction. The iodine values determined revealed that the breadfruit seed oils were in the category of semi-drying oils. Oils are considered semi-drying when they have iodine value that fall in the range of 90 – 130 mg I/g [18].

It was observed that linolenic acid was absent in the breadfruit seed oil. Linolenic acid present in oil leads to the yellowing associated with alkyds prepared from highly unsaturated (oxidizing) oils. It is expected that alkyds prepared from this oil will be colour retentive because of the absence of linolenic acid in breadfruit seed oil.

The saponification value obtained for the oil of breadfruit seeds is 161.30mgKOH/g while Soyabean oil had a saponification value of 191.50mgKOH/g. The saponification value determinations on the oils revealed that soyabean oil contained glycerides with low molecular weight than breadfruit seed oils. The later observation was consistent with their iodine values. Saponification value is used to assess the mean molecular weight (M_w) of the fatty acids present in oil. Saponification values above 200 indicate the presence of fatty acids of low or fairly, low molecular weight, and values below 190 indicate the presence of high molecular weight fatty acids [19].

Table – 2 Properties of the Vegetable oils of Breadfruit and Soyabean Seeds

Vegetable seed	Yield (%)	Acid value mg KOH/g		Iodine value	Saponification value
		Crude	Refined		
Breadfruit	25.73	2.26	0.32	107.30	161.30
Soya bean	-	-	0.16	128.40	191.50

Table -3 Acid Values of Alkyd Resins

Alkyd Resin	Acid value (KOH/g resin)	
	Long oil length alkyd resin	Medium oil length alkyd resin
BFSOA	4.50	5.21
SBSOA	1.60	1.01

BFSOA = Breadfruit Seed Oil Alkyd; SBSOA = Soyabean Seed Oil Alkyd

Table -4 Drying Properties of Long Oil Length Alkyd Resins

Alkyd Resin	Film Thickness (mm)			
	0.10	0.40	0.70	1.0
BFSOA	S	NS	NS	NS
SBSOA	S	S	NS	NS

Table -5 Drying Properties of Medium Oil Length Alkyd Resins

Alkyd Resin	Film Thickness (mm)			
	0.10	0.40	0.70	1.0
BFSOA	S	NS	NS	NS
SBSOA	S	S	NS	NS

Note: S= Satisfactory, NS = Not Satisfactory

Table -6 Media Resistance of Long Oil Length Alkyd Resins

Alkyd Type	Media Resistance			
	Distilled	2%H_2SO_4	2%NH_3	2%Na_2CO_3
BFSOA	2	0	3	4
SBSOA	1	0	3	4

Table -7 Media Resistance of Medium oil Length Alkyd Resins

Alkyd Type	Media Resistance			
	Distilled	2%H_2SO_4	2%NH_3	2%Na_2CO_3
BFSOA	1	0	4	4
SBSOA	1	0	3	4

Characterization of Alkyd Resins

Acid Value

The Alkyd resins synthesized in this study were characterized in terms of acid number. The determined acid values are presented in Table 3. For the long oil length alkyd resins, the determined values for soyabean and breadfruit seed oil alkyd are 1.60 and 4.50 mgKOH/g resins respectively. The acid values of the synthesized medium oil length alkyd resins for soyabean seed oil and breadfruit seed oil alkyd are 1.01 and 5.21 mgKOH/g) resins respectively. The acid value of an alkyd resin is important in assessing the stability of the synthesized resin. The low acid values (<10 mgKOH/g resin) obtained for the resins synthesized in this study is an indication that the resins were synthesized near to completion of the reaction, and therefore very stable.

PERFORMANCE EVALUATION OF SYNTHESIZED ALKYD RESINS

Drying Properties of Synthesized Alkyd Resins

The drying properties of the synthesized long and medium oil length alkyd resin samples are presented in Tables 4 and 5 respectively. Tables 4 and 5 showed that all the prepared alkyds dried satisfactorily at alkyd film thickness of 0.10 mm. Only soyabean seed oil modified alkyd resin dried satisfactorily at film thickness of 0.40 mm for medium and long oil length alkyd resins. The drying studies showed the superiority of soyabean seed oil and breadfruit seed oil in the preparation of medium oil length alkyd resin. The result indicates that breadfruit seed oil is semi-drying oil, and the order in the degree of their unsaturaction as determined by iodine number is soyabean seed oil > breadfruit seed oil. The present study showed that the oil length of the synthesized alkyd resins did not exhibit marked effect on the drying of alkyd resin films.

The thickness of the alkyd resin film was observed to exact a marked effect on the drying of the alkyd resin samples, namely decrease in drying properties of the resins with increase in alkyd resin film thickness. For an alkyd resin film to dry effectively, the oxygen induced crosslinking (drying) reaction should occur throughout the film from the surface to the interior. Since polymerization and drying are related to oxygen contents, the concentration of which is greatest at the surface and least in the interior. Also observed from the study, the thicker the alkyd resin film, the smaller the oxygen concentration in the inner portion of the film this leads to an overall reduction in the degree of drying.

The Media Resistance Tests on Alkyd Resin Films

The media resistance of dry alkyd resins to distilled water, 2 % NH_3, 2 % H_2SO_4, and 2 % Na_2CO_3 are shown in Tables 6 and 7. From the Tables it is evident that the water and acid resistance of the synthesized alkyd resins were generally good. All the synthesized alkyd resins failed the alkali resistance test, an indication that alkyds generally have low alkali resistance. The poor alkali resistance of the alkyds may be due to the fact that they contain ester groups, which are known to be susceptible to hydrolysis by alkali [20]. The influence of alkyd oil length type was not observed to have any effect on the media resistance of the alkyd resins investigated.

Adhesion to Surfaces

The adhesion properties of the synthesized alkyd resin are presented in Table 8. The dried alkyd resin films exhibited good adhesion properties, an indication that paints formulated using these alkyd resins will be able to protect substrates from corrosion, and thus, function as anticorrosion paints. The general order of adhesion of the alkyd resin films to the substrate is soyabean oil alkyd > breadfruit seed oil alkyd (both for the long and medium oil length alkyds). The observed order corresponds to the order in the degree of unsaturation of the oils.

The present study shows that the adhesion properties of soyabean oil alkyd are slightly greater than that of the breadfruit seed oil alkyd (both for the long and medium oil alkyds). Breadfruit seed oil alkyds exhibited the same adhesion properties in both the long and medium oil length alkyds. The oil length of the synthesized alkyds exerted very slight effect on the adhesion properties of soyabean. Generally, the alkyds exhibited good adhesion properties. According to Nigerian Industrial Standards (NIS 268: 1989), a good oil paint should not exhibit more than 50% maximum removal of the dry paint film.

The good adhesion to the substrate of the resins obtained in this study is attributed to the inherent chemical structure and flexibility of the alkyd resins [21]. It should be noted that certain polar groups in a film such as carboxylic groups (-COOH) are very active promoters of adhesion, due to their attraction to the substrate, or by their influence in improving the wetting properties. Thus, good initial wetting of a substrate by a coating and the maintenance of wetting during the process of film formation are essential for good film adhesion [22].

Mildew Formation

There was no mildew formation on all the synthesized alkyd resin film samples when subjected to sunlight and rain for six months, an indication that the synthesized alkyds will perform well in coatings.

Table -8 Mildew Formation and Adhesion Properties of Synthesized Alkyd Resins

Alkyd Type	Long oil Length Alkyd Resin		Medium Oil Length Alkyd Resin	
	Adhesion Test (%)	Mildew formation	Adhesion Test (%)	Mildew formation
SBO	**24**	**Nil**	**26**	**Nil**
BSO	**32**	**Nil**	**32**	**Nil**

CONCLUSIONS

Long and medium alkyd resin has been successfully synthesized using breadfruit seed oil. Soyabean oil modified alkyd resin served as the reference alkyd showed satisfactory drying property at 0.40 mm film thickness. The results of this study indicate that the oil extracted from breadfruit seeds has desirable properties to be classified as semi-drying oil, suitable for modifying alkyd resin for surface coating purposes. The long oil length alkyd resins prepared showed satisfactory drying properties at 0.10 mm oil film thickness. All the oil length alkyds from breadfruit oil exhibited poor drying properties irrespective of the film thickness. The synthesized alkyd resins have good water, and acid resistance. However, all the alkyds failed alkali resistance tests. The adhesion properties of the alkyd resin films were generally good at both oil lengths prepared, with soyabean oil alkyd exhibiting better adhesion than breadfruit seed oil alkyd. The good adhesion property of the resins is an indication that paints formulated with these resins should function as anti – corrosive paints. As a result of the poor alkali resistance, formulating surfacing coating using these resins should be done in an environment where alkali resistance is not a significant factor.

The present study has highlighted the utility of breadfruit seed oil in the synthesis of long and medium oil length alkyd resins. The alkyd properties obtained with this oil was comparable to those of soyabean oil in both the long and medium oil length alkyd resins synthesized. The seeds of breadfruit are of natural resource, and indigenously available. The ultimate usage of this oil in the coatings industry will save the country the enormous resources being spent in importing vegetable oils for the coatings industry, as well as encouraging the cultivation of the trees of the oil-bearing seeds with the farmers being the ultimate beneficiary. The present study does not anticipate nor intend the total replacement of the conventional oils with these local oils. The expectation is that the local seed oils (breadfruit* and soyabean seed oils) will develop their own niche in the coatings market in the future.

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