

available online *(iii)* www.pccc.icrc.ac.ir

Prog. Color Colorants Coat. 12 (2019), 121-132



The Effect of Gmelina Seed Oil on the Synthesis of Alkyd Resins

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ARTICLE INFO

Article history: Received: 17 Dec 2018 Final Revised: 18 Feb 2019 Accepted: 19 Feb 2019 Available online: 19 Mar 2019 Keywords: Gmelina seed oil Alkyd resin Chemical resistant Desaturation GC-MS Through dry time.

ABSTRACT

 \mathbf{T} he usage of edible oils for alkyd resin synthesis has lately been of great concern because they can alter the food chain. Research is on the forefront on the oils that are considerably available and do not compete with food sources. Thus, the viability of preparing alkyd resin from inedible, non-drying gmelina seed oil (GSO) was investigated. The physico-chemical properties (viscosity, iodine value, specific gravity, refractive index and acid value) of the GSO were determined and then, it was neutralized, epoxidized, hydroxylated and dehydrated. The fatty acid profiles of the raw oil were determined with GC-MS. The structure of raw GSO, chemically modified GSO and their alkyds were determined using FT-IR spectrometry. Medium GSO length modified alkyd resin was produced via a two stage alcoholysisesterification method using glycerol, maleic and phthalic anhydride. GC-MS result confirmed that GSO is predominantly saturated, while the FT-IR result confirmed the desaturation and esterification of GSO. Using nano-ZnO pigment, a through dry time of about 8 hrs was obtained with GSO modified alkyd resin C. The alkyds C and D show excellent mechanical properties; H passed and 2H passed for scratch hardness test, good for adhesion test and passed for impact test. Further comparison revealed that Alkyds A and C showed robust resistant to varying chemical medium in comparison with standard alkyd resin. Prog. Color Colorants Coat. 12 (2019), 121-132© Institute for Color Science and Technology.

1. Introduction

Surface coating is a universal term for any material that may be applied as a continuous film to a surface and dry to form adhering films [1]. The varying price, environmental issues, and uncertainty in petroleum resources are some of the reasons why the research communities are searching for alternative raw materials in diverse industrial fields [2]. Alkyd resin is the major binder of most surface coating materials. Alkyd resins are used more than other binders because of their low processing cost, inexpensive raw material, and easy manufacturing. Besides, they can be dissolved in less expensive solvents. Recently, green chemistry processes has gained wide use, especially, in the alkyd resin industry [3, 4]. While the major hindrance in the alkyd resin is their high solvent content, improvements in the synthesis of alkyd resins are still needed and developments of new alkyd resin materials are still welcomed. Alkyds have been produced from various triglycerides (plant oils and free fatty acids); many have been successfully commercialized and used in industries. There is a long history of the development and many interesting variations on the process and final modifications to obtain unusual useful properties [5-11]. Their compatibility with many polymeric materials and the outstanding formulating latitude makes alkyd resins widely used in surface coatings where they function as a binder. Alkyd resins are also used in adhesive, ink, caulks and plasticizers.

Gmelina seed oil (GSO) is an inedible saturated vegetable oil that is extracted from Gmelina arborea fruitlet. Gmelina arborea fruit is obtained mostly in Nigeria, Sierra Leone, India, Malaysia, China, Vietnam, Laos and Thailand, etc. There is a lot of research investigations on the economic importance of GSO [12-18]. To be more specific, the potentials of the GSO resource have been investigated in the area of medicine and pharmacy [12-14], bio-diesel production [14-17], and production of lubricants [18]. Recent solvent extraction process designed for GSO extraction from the seed further indicated that the oil is a renewable, viable and sustainable material in term of processing technique and their availability [19]. Gmelina seeds have been shown to contain 54.6 % oil, but 45-52 % oil can be recovered by optimized solvent extraction [19]. This oil content is substantial and can compete satisfactorily with other seed oil plants such as Hematostaphis berter (54.5 %wt), Sapindus mukorossi (51 %wt), Jatropha curcas (30-50 %wt), Hevea brasiliensis (51 %wt), Mellia azadirachta (33-45 %wt), and 55-65 %wt for Simarouba glauca [20]. Considering the oil content, gmelina arborea seed is most appropriate for industrial applications, since it has been reported that any oil bearing seed that can produce up to 30% oil are regarded as one of the numerous industrial crops [21]. The recent emphasis, therefore, is to find oil bearing plants that produce nonedible oils as the feedstock for industrial alkyd resin production. Hence, this opens opportunities for further research on the prospective of the highly available GSO in commercial production of alkyd resins.

GSO has been categorized as saturated oil [15, 19]. The implication is that GSO or its derivatives cannot dry by auto-oxidation. This renders the potentials of GSO unattractive as raw material for surface coating where good drying characteristics are considered extremely important. Certain standards must be met for an oil to be used as feed stock for industrial coating application; the resulting products must satisfy the technical and industrial specifications such as durability, fastness to exposure, resistances, etc.; it must satisfy all the

ecologically important criteria [22, 23]. Drying oil usage in alkyd synthesis is not only expensive, but the alkyds are prone to yellowing effect due to the significant unsaturation in the dry oil. Issam and co-workers discovered that in the absence of drying agent, the curing process is difficult, but with drying agents, the synthesized resins exhibit excellent adhesion onto aluminum surface as a substrate [24]. In previous studies, non-drying palm kernel oil, palm oil and coconut oil have been exploited in alkyd resin production by attaching an appropriately chosen functional group to the oil molecules that changes its behavior and structure, converting it from a non-curable to a curable product [25-27]. In another account [28, 29], alkyd resin was produced from non-drying palm oil by increasing the unsaturation level and incorporation of carbon nanotubes. A major noticeable concern with the produced resins was the inability of the film to dry thoroughly overnight.

Although GSO has been reported by many authors in medicine, pharmaceutical and biodiesel applications, but its use in surface coating is rare in literature. It may be concluded, apparently, that GSO have not been investigated for alkyd resin synthesis especially from the view point of comparative analysis with other standard resins. This study therefore can be a good approach to establish that GSO is an industrially relevant coating material of sustainable and renewable resource. A procedure was adopted to increase the level of unsaturation in the GSO chain by modification via epoxidation, hydroxylation and dehydration.

2. Materials and Methods

2.1. Materials

The gmelina aborea seed were procured locally from forests in Awka, Anambra state. It was soaked in water for about 8 days in order to easily separate the fruit pulp from the seed (de-pulping). The seeds were subsequently sun-dried and grounded with corona blender. The oil was extracted under optimum conditions established in [19]. Analytical grade glacial acetic acid, acetic acid, H₂SO₄, phthalic anhydride, sodium bicarbonate, maleic anhydride, lithium oxide and glycerol were obtained from commercial sources (Merck Chemicals, BDH Chemical Limited, Poole, England and May and Baker Limited, Dangeham, England) and used without further purification.

2.2. Methods

2.2.1. Desaturation of the Crude GSO

The crude GSO was desaturated according to the steps described elsewhere [3] and shown in Figure 1. This scheme encompasses epoxidation (introduction of oxirane ring in the carbon chain), hydroxylation (subsequent introduction of hydroxyl group) and dehydration. GSO (100 cm³) was poured in a roundbottomed flask, and 10 cm³ of 30% H₂O₂, 5 cm³ glacial acetic acid and 1 cm³ conc. H₂SO₄ were added in sequence to the oil and stirred constantly. Peracetic acid was generated from acetic acid and hydrogen peroxide in the presence of a strong mineral acid (H₂SO₄). The reaction was maintained at room temperature and left overnight. The resulting oily layer was separated from the aqueous layer and washed with 10 cm³ saturated solution of sodium bicarbonate. The oily layer obtained was subsequently washed five times with distilled water after which it was dried at 80 °C for 30 min using vacuum pump (91 cm³ oil was obtained). The above epoxidized oil (90 cm³) was heated to 98-100 °C. Dilute H_2SO_4 (10 cm³) was added and the mixture was stirred intermittently for 90 min. Then, the oily layer was separated from the aqueous layer using a separating funnel after which it was dried at 80 °C for 30 min using vacuum pump. Hydroxylated (75 cm³) oil was poured into a three-necked reaction flask. Three drops of concentrated H₂SO₄ were added as catalyst. The temperature of the reaction mixture was then raised to 260±5 °C, and this temperature was maintained for an hour. The evolved water (3 cm³) was collected as distillate. An inert atmosphere was ensured by passing nitrogen gas through the reaction mixture [3].

2.2.2. Characterization and Synthesis of GSO Based Alkyd Resin

A Thermo Finnigan Trace GC/Trace DSQ/A1300, (E.I Quadropole) fortified with a SGE-BPX5 MS fused silica capillary column (film thickness 0.25 lm) for GC–MS detection, and an electron ionization system with ionization energy of 700 eV was used to analyze the oil. Carrier gas was helium at a flow rate of 10 mL min⁻¹ injector and the MS transfer line temperatures were set at 220 and 290 °C, respectively, as described elswhere [5]. The chemical composition of the oil was analyzed by SHIMADZU FTIR-84008. Viscosity was determined by Brookfield viscometer, RVT Model (#Spindle 3, RPM 21). The physico-chemical properties of the raw and modified oil were determined by standard methods (ASTM, 1973).

Four types of alkyds were synthsized with the modified gmelina seed oil employing lithium oxide as catalyst according to the formulations shown in Table 1. Alkyd-A was synthesized from the refined saturated gmelina seed oil, Alkyd-B was synthesized from the refined desaturated gmelina seed oil and phthalic anhydride (PA) while Alkyd-C and Alkyd-D were synthesized from the refined desaturated gmelina seed oil by varying the amount of phthalic anhydride and maleic anhydride (MA) (Table 1). Alkyd resins were prepared via two step alcoholysis-polyesterification method. A three-necked 1000 mL round-bottom flask was equipped with a mechanical stirrer, thermometer, nitrogen gas inlet and condenser with dean stark apparatus to remove the water of the reaction, azeotropically. Xylene was used as the azeotropic solvent. The reactor was flushed with nitrogen.



Figure 1: Proposed modification of a section of GSO triglyceride chain.

Resin type	Oil length (%)	Glycerol (g)	LiOH (g)	PA (g)	MA (g)
Alkyd-A	50	50	1	50	
Alkyd-B	50	50	1	50	
Alkyd-C	50	50	1	25	25
Alkyd-D	50	50	1	15	35

Table 1: Recipe of the synthesis oil GSO modified alkyd resin.

The triglycerides of GSO were converted to monoglycerides by reaction with glycerol at 180 -200 °C during the alcoholysis stage. Then the mixture was heated first at about 165 °C and raised to about 220-235 °C for 60 min until monoglyceride was formed. The first stage alcoholysis reaction was confirmed by solubility in methanol (resin: methanol ratio of 1:3 v/v). The heating was stopped and the mixture was allowed to cool to 120 °C under stirring. Then, the glycerol, stoichiometric amount of PA (Table 1) and xylene were introduced, and the polymerization was carried out at 220-260 °C for more than 6 h as shown in Figure 2a. The required MA was then introduced to complete the reaction (Figure 2b). The concentration of alkyd solutions was evaluated and finally diluted to 70-75% by addition of xylene. The progress of reaction was monitored by measuring the reaction evolved water and intermittently checking the acid number by titration.

2.3. Performance Appraisal

The synthesized resins were thinned with xylene and applied as thin panels on an aluminum panels. Nano-Zinc oxide (ZnO) pigments were added to the synthesized resins to aid the drying. The synthesized resins were applied onto glass plates of $50 \times 50 \times 0.1$ mm³ by means of a bar-coater at 24-30 °C and relative humidity of 50%. The drying time and physicochemical properties were also evaluated. ASTM D 1640 was used for checking the surface drying time, set-to-touch time and through dry time. Set-to-touch is defined as the non-transferability of the coating upon slightly touching the film with finger. Surface dry is defined as the failure of the film to adhere to the finger when pressed firmly and does not rub up when rubbed lightly. Through dry time is the non-distortion of the film when the thumb is pressed on it in a definite manner and rotated through a 90 °C angle. The samples were checked regularly to trace the dryness or tackiness by means of finger tips and filter paper.

The chemical and mechanical properties of the synthesized alkyds were evaluated by standard methods [23]. IR spectra of the alkyds were also recorded on the FT-IR spectrometer.

3. Results and Discussion

3.1. Characteristics of the Raw and Desaturated GSO

The physico-chemical properties of the raw and desaturated GSO are shown in Table 2. It was observed that the neutralization reduced the free fatty acid (FFA) content of the raw GSO from 1.85 to 1 and a refractive index of 1.465 from 1.441. In the neutralization process, the FFA content of oil was converted to oil soluble soaps. Contaminants like proteins and /or protein fragments, phosphotides and gummy or mucilaginous substances were also removed by the neutralization process. The acid value increases from 3.92 to 6.28 mg KOH/g. Exceptional changes were noticed in the properties of the oil as it went through the various modification procedures (epoxidation, hydroxylation and dehydration) to obtain chemically modified oil. Initially, there was an increase in the density through epoxidation and hydroxylation, but an apparent decrease in density was observed after dehydration. The increase in density during epoxidation and hydroxylation shows an increase in mass per unit volume of the GSO sample which could be attributed to the reduction of low molecular weight FFA content in the GSO and inclusion of oxygen atom in the fatty acid structure. The increase in acid value and the reduction in density of the GSO may be attributed to the loss of water molecules.





(ii) Second stage-Reaction with Maleic anhydride (MA)

Figure 2: a) Alcoholysis, b) Esterification and c) Second stage-Reaction with Maleic anhydride (MA).

Also, the increase of acid value during chemical modification step may be due to the competing side reactions such as hydrolysis of triglycerides to FFA due to the presence of mineral acid (H_2SO_4) in the epoxidation process. The GSO color turned from golden yellow to whitish yellow after epoxidation and hydroxylation but became brown after dehydration due to the relatively high temperature at which the reaction was done. Moreover, the viscosity of the oil increased

slightly during the modification. The iodine value showed a very large increase (39.3 g to 125.5 g $I_2/100g$) showing the increase in the level of unsaturation after chemical modification process. This observation was anticipated since epoxidation, hydroxylation and dehydration reactions actually took place at the point of unsaturation (Figure 1) of the aliphatic chain by ring-opening, addition and elimination reactions, respectively. It is evident from

the results that the modified GSO is suitable for the synthesis of alkyd resin, and its unsaturation level will allow the cross-linking reactions for alkyds to form a dry hard solid film [26]. The set to touch time and drying time show the drying properties of the dehydrated GSO sample. The saponification values of the raw and the dehydrated GSO were 38.23 and 143.61 mg KOH/g, respectively. The saponification value shows the average molecular weight of fatty

acids of triglyceride present in GSO. The iodine value and the viscosity of the dehydrated GSO were within the limits of the ASTM standard. The drying time and set to touch time gave suitable result. Therefore these dehydrated oil samples were acceptable for the synthesis of alkyd resin.

The fatty acid profile of the raw GSO and the FT-IR spectra of the raw and modified GSO are shown in Figure 3 and Figure 4, respectively.

Properties	Crude oil	Neutralized oil	Epoxidized oil	Hydroxylated oil	Dehydrated oil
Free fatty acid value (%)	1.85	1			1
Color	Golden yellow	Golden yellow	Whitish yellow	Whitish yellow	Brown
Refractive index (25°C)	1.441	1.465			
Specific gravity (25°C)	0.893	0.899	0.9393	0.9456	0.9012
Viscosity (pa s)	3.336	3.392			3.486
Saponification value(mgKOH/g)	38.23				143.61
Acid value	3.92				6.28
Iodine value (gI ₂ /100g)	34.9				125.5
Set to touch(hr)					4
Drying time(hr)					6
Oil content (wt%)	55.16				4.38
Physical state (25°C)	Liquid	Liquid	Liquid	Liquid	
Ash content (%)	5				

Table 2:	The	characteristics	of the	crude and	modified	amelina	seed	oil
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Table 3: Fatty acid profile of gmelina seed oil.

Carbon molecules	Name	%wt
C ₁₀	Capric acid	1.27
C ₁₁	Undecyclic acid	10.49
C ₁₂	Lauric acid	6.00
C _{20:2}	Arachidic acid	17.16
C ₁₄	Myristste	4.59
C _{16:1}	Palmitoleate	31.94
C ₁₇	Magaric acid	4.84
C ₁₈	Methyl stearate	14.22
C ₂₅	Pentacosylic acid	3.70
C ₃₀	Melissic acid	5.75





The fatty acid profile (Figure 3) examined by the GC-MS revealed the abundance of palmitoleate (31.94 %wt) and arachidic acid (17.16 %wt). The most abundant unsaturated and saturated fatty acids were palmitoleate (31.94 %wt) and methyl stearate (14.22 %wt), respectively. The oil contains 50.86 % saturated fatty acid and 49.10% unsaturated fatty acid. The high saturation in GSO indicates that if it is applied in the synthesis of alkyd resin, it will give alkyd of slower drying rate but color retentive. Therefore, structurallymodified GSO can give alkyd resin with better performance index. In the spectrum of the raw GSO in Figure 4, 3298 cm⁻¹ peak corresponds to the hydroxyl group (O-H) of the unsaturated fatty acid in the oil. The carboxyl group (C=O) is indicated at 1954 cm⁻¹. The stretch of -CH chain in aliphatic compound is seen at 2934 cm⁻¹. Alkene group (CH=CH) is attributed to the band at 3206 cm⁻¹. The FT-IR spectra of the raw GSO show two peaks at 938 cm⁻¹ and 1378 cm⁻¹ which correspond to cyclic ester of saturated oil. IR spectra and functional compounds of modified GSO showed the broad band shoulder around 3902 cm⁻¹, 3870 cm⁻¹ and 37684 cm⁻¹ (initially at 3298 in the unmodified oil) corresponding to the hydroxyl (OH) group in the unsaturated fatty acid in the mixture [11], which reveals an increase in unsaturation after GSO modification. The peaks of the epoxidized GSO were similar to those of raw samples while the peaks of the hydroxylated GSO were similar to those of the dehydrated samples.

3.2. Synthesis and Performance Appraisal of the GSO Modified Alkyd Resin

Polycondensation was conducted by heating a mixture of monoglyceride mixture and phthalic anhydride/and maleic anhydride according to the stoichiometric amount and the product obtained from the alcoholysis step until the acid value dropped to about 10. This particular test was conducted following ASTM D 1980-87. This indicates that the acid value decreases gradually with time. Application of maleic anhydride as a polybasic acid introduces an additional complexity to the formulation of alkyd resins. Water, a by-product of esterification under vacuum is carried off in the xylene. Overall, the heat and mass transfer of chemical reaction is increased substantially.

The performance of the GSO modified alkyd resin was evaluated in terms of their phsico-chemicomechanical properties (acid number, adhesion, impact, drying schedule, color test, chemical and abrasion resistance, and scratch hardness test). The results of color test and acid number are presented in Table 4. The brown color of the alkyd resins is mostly due to the relatively high temperature of polycondensation process. It is found that alkyd resins containing varying amount of double bond had obviously different color and molecular weight. As a monofunctional component, saturated GSO tends to terminate chain growth. For instance. although formulation compositions for ALKYD A and contains PA while C and D contain vary concentration of PA and MA, the increased saturation in A (raw GSO) tend to terminate chain growth which results in lower molecular weight. This is undesirable as low molecular weight can lead to poor dry rate of alkyd resin. The results presented in Table 5 show that all the resin films exhibited excellent resistance to water, acid and brine but poor resistance to alkali. The ester linkages in the alkyd resins are prone to alkaline hydrolysis which causes the poor performance of all the alkyd resins in alkali. The chemical film properties of sample ALKYD A and C are as good as that of the standard alkyd resins. The chemical film properties of samples ALKYD B and D are relatively inferior to the standard alkyd resin. The results revealed that alkyd C possesses a very good chemical superior resistance.

Alkyd samples	Acid number of diluted alkyd resins (mg KOH/g)	Color of alkyd resins
Alkyd A	10.80	Brown
Alkyd B	12.09	Brown
Alkyd C	13.01	Light Brown
Alkyd D	14.23	Light brown

The Alkyd-A to Alkyd-D were each, hand coated on the surface of four different aluminium panels which were cleaned with an ethanol to ensure that the surface of the aluminium panels were free from contaminants that might affect the drying test carried on it. The drying time of alkyd-A to D at 32 ± 2 °C is shown in Figure 5. A remarkable progress was observed in set to touch, surface dry and through dry time from alkyd-C to Alkyd-D. Precisely, alkyd C and D gave a better drying schedule when compared to the Alkyd A and B, respectively. It may be concluded that alkyds C and D could be used as binder in surface coatings as it showed comparable coating characteristics (drying time) to other reported oil modified alkyds [30, 31]. The acid value and carboxyl functionality (F-COO) affect the drying time of the resin. As seen from Fig. 5, Alkyd-C and Alkyd-D with relatively lower acid value compared to Alkyd-A and Alkyd-B have the best drying properties. Figure 5 further discloses that none of the alkyd samples were able to attain a through dry time overnight. However, when nano-Zinc oxide (ZnO) pigments were added to the resins, Alkyd-C was able to dry hard overnight (about 8 h). The reduction in drying time of the alkyd film may be due to the increased chemical potential in the presence of nano-ZnO pigment. The influence of surface energy to total energy content of nano-material, positively affect its chemical reactivity; invariably leads to extensive acceleration of chemical reactions [32].

From Table 6, it can be inferred that the four medium oil length GSO modified alkyd resin exhibits varying mechanical properties when subjected to impact, adhesion and scratch hardness tests. Alkyds-A and D showed poor hardness and adhesion properties while C and D passed both tests. The good hardness of Alkyds-C and D might be due to the aromatic moiety present in phthalic anhydride, while adhesion is attributed to the polar ester bond [24]. There were no flaking or ribboning after making cuts and applying the adhesive tape. The maleic anhydride on the polymer backbone promotes adhesion. The hydroxyl groups (OH) in the structure of the resins also improved the adhesion to the substrates. The results suggest that these alkyds have potential in surface coatings.



Figure 5: Drying Schedule of Alkyds A to D.



Figure 6: FTIR spectra of GSO modified alkyd resin.

Properties		Standard alkyd			
Toperates	ALK	resins			
Water resistance (cold, 12 days)	4	5	5	5	5
Alkali resistance (0.1 M NaOH, 12 h)	2	1	2	1	2
Acid resistance (0.1 M H ₂ SO ₄ , 12 days)	5	3	5	3	5
Brine resistance (5%, w/w, NaCl, 12 days)	5	4	5	5	5

Table 5: The chemical film properties of gmelina based alkyd resins and standard alkyd resins.

0, completely removed; 1, cracked and partially removed; 2, partially cracked; 3, loss of gloss; 4, slight loss of gloss and 5, practically unaffected.

3.3. Characteristics of the GSO Modified Alkyds

The FT-IR spectra of the alkyds are shown in Figure 6. The IR spectra of the resins with PA and PA: MA showed similar peaks. Comparing the spectra of the alkyds and those of the modified GSO (Figure 4) additional peak/band indicating the presence of ester group 1039–1284cm⁻¹ for PA and 1094-1260 cm⁻¹ for PA:MA (O=C-O-C stretching frequency) were seen, but these were absent in the IR spectrum of the modified GSO. The absorption bands in alkyd resins at 2034 cm⁻¹, 2099 cm⁻¹, 2153 cm⁻¹ and 1977 cm⁻¹, which correspond to carbonyl group, are very sharp and

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strong. Methylene C-H stretching bands at 1377 cm⁻¹ and 1377 cm⁻¹ are conspicuous in both spectra. Aromatic C-O stretching in esters and alkene (C=C) stretch were seen at 1473 and 1622 cm⁻¹.

4. Conclusion

This study established that alkyd resin with desired coating properties can be synthesized by GSO. The resulting alkyd resins exhibited very good physical, chemical and mechanical properties. The study further showed that the inclusion of nano-pigment gave a faster drying resin. The FT-IR spectrum confirmed that the chemical structure of both the raw oil and their corresponding resins with the functional ester groups present. Therefore, it may be concluded that the desaturated GSO offers an excellent potential for surface coating applications.

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How to cite this article:

C. F. Uzoh, O. D. Onukwuli, A. C Nwabueze, The Effect of Gmelina Seed Oil on the Synthesis of Alkyd Resins. Prog. Color Colorants Coat., 12 (2019), 121-132.

