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## Structural Characterization of Sandbox Seed Oil-Modified Alkyd Resin

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### Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

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### ABSTRACT

**Aim:** To synthesize alkyd resin from sandbox seed oil and characterize the alkyd based on the physicochemical and structural properties.

**Place and Duration of Study:** Department of Polymer and Textile Engineering, and Department of Soil Science Technology, both at Federal University of Technology, Owerri, Imo state, Nigeria, from July 2014 to October 2014.

**Methodology:** Sandbox seed were properly dried, ground and sieved through a 2 mm mesh size sieve to obtain a fine powder. The seed oil was extracted by bulk cold extraction method using n-hexane, and the oil was characterized to determine the density, acid value, peroxide value, saponification value, iodine value and free fatty acid value. The oil was later converted to alkyd resin through a two stage process of alcoholysis and esterification. FTIR spectroscopy was used to analyze the functional groups present in the alkyd resin and compared with a commercial alkyd resin.

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**Results:** The results on physicochemical analysis showed a high acid value of 149.066 when compared with other seed oils, a weight percentage fatty acid content of 74.931% confirmed it as long oil since it is greater than 55%. The resulting alkyd resin was characterized to determine its viscosity and the FTIR spectra. The results of FTIR give an absorption band at wavelength above  $3000\text{ cm}^{-1}$  which confirm the material as organic compound. The FTIR analysis shows a similarity with the FTIR result of a commercially available alkyd resin, it indicates similar functional group and chemical structure. Therefore considering the results above, alkyd resin from the sandbox tree is suitable as a binder in coating industry.

*Keywords: Alkyd resi; sandbox seed; FTIR; alcoholysis; esterification.*

## 1. INTRODUCTION

Alkyd resins have become an indispensable raw material in the coating and paint industries. It is the major important binder, accounting for the largest volume base of coatings and paints especially for decorative applications [1]. They are used for decorative painting applications more than other binders due to their lower cost, this lower cost is as a result of inexpensive raw material, easy manufacturing and high solubility of resin in less expensive solvents. Other additional qualities include good adhesion and ease of application under variable environmental conditions, gloss and gloss retention, durability and film flexibility [2]. Additionally, they are to a greater extent biologically degradable polymer because of the oil and glycerol parts and they are ecofriendly compared to petroleum based polymers that constitute environmental pollution and degradation [3]. They are also found to be compatible with other film formers such as acrylic resins to form alkyd hybrid systems that combine the technical benefits of both resins than when used in isolation. Alkyd resins are not only used in decorative paints but also mainly are applied in air drying paints, inks, machine tool finishes and clear, matt and semi matt varnishes of wood furniture [4].

Alkyd resin is a product of polyesterification reaction of unsaturated oils like soybean, linseed oils and the likes with diacids and polyhydroxy alcohol. There have been several other attempts to define alkyd resins over the years. One of such definitions is that of Bobalek et al. [5] which define alkyd resins as the reaction product of polyhydric alcohol and poly functional acids modified by fatty acid or their triglyceride.

Alkyd resins are classified based on the oil length (i.e. based on the ratio of monobasic fatty acids to total weight of polymer formed during synthesis), this is expressed as a percentage [6].

$$\text{Oil Length} = \frac{\text{weight of oil}}{\text{weight of alkyd} - \text{water evolved}} \times 100 \quad (1.1)$$

$$\text{Oil Length} = \frac{1.04 \times \text{weight of fatty acid}}{\text{weight of alkyd} - \text{water evolved}} \times 100 \quad (1.2)$$

The 1.04 factor in equation (1.2) converts the weight of fatty acids to the corresponding weight of triglyceride oil.

From the classification; Short oil length alkyd contains less than 40% oil and are applied in industrial coating sector as anti-corrosive primers and finishes. Other applications are; stoving enamels, agricultural machinery finishes, commercial and car refinishes. The medium oil length alkyd contains 40-55% of oil, they are air and oven drying; aliphatic and aromatic solvent compatible and are often used in low cost anticorrosion primers and some industrial stoving enamels such as agricultural machinery. The long length alkyd contains greater than 55% of a drying oil; they are air drying and aliphatic solvent compatible and are used in building interiors. Other applications include marine and maintenance applications as primers and finishes.

Another important classification of alkyd resin is as oxidizing and non-oxidizing type [7]. An oxidizing alkyd contains drying (unsaturated) or semi-drying oils or fatty acids and is able to form film by air oxidation. This type of alkyd usually has an oil length in excess of 45%. While a non-oxidizing alkyd contains non-drying (saturated) oils or fatty acids and therefore the alkyd is not capable of forming coherent film by air oxidation. Hence, the non-oxidizing alkyd is used as polymeric plasticizer or as hydroxyl-functional resin, which are cross linked by melamine-formaldehyde or urea-formaldehyde resin, or by isocyanate cross linker. The oil length for non-oxidizing alkyds usually is below 45%.

Many research works have been carried out on the production of alkyd based resin from local seed oil. Keskin [8] reported on the processing of long oil alkyd using an oil mixture of 40% linseed oil and 60% sunflower in combination with melamine based hyper-branched polymer having 24 functional groups on its structure. Momodo and Co [9] extracted Walnut seed oil (WSO) from walnut seed and employed in the production of 50% oil length Alkyd Resin (oil modified polyester) using the monoglyceride method. This involved reacting the oil first with glycerol (alcoholysis) and subsequent treatment of the monoglyceride obtained with phthalic anhydride. Kyenge et al. [10] reported on the preparation of alkyd resins using condensation polymerization from both epoxidized and unepoxidized soybean oil monoglycerides by alcoholysis with phthalic acid, which were cured with 10% paint drier. Rubber Seed Oil (RSO) was used in the production of alkyd resins of varying oil length (35%) for short alkyd and 50% for medium alkyd (oil modified polyester) using the monoglyceride method as reported by Umeobika et al. [11]. In the research work done by Asiagwu et al. [12], oil was extracted from *Tetracarpidum conophorum* nut (Nigerian Walnut) using solvent extraction method with hexane as solvent. The oil obtained was used to synthesize an alkyd resin, which in turn was utilized in the production of white gloss paints and varnishes. Igbo et al. [13] and Cruz [14] have also synthesized alkyd resin from beniseed oil and virgin coconut oil respectively.

Sandbox tree (*Hura crepitans*) is an evergreen tree that belongs to the spurge family (Euphorbiaceae), it grows in the tropical regions of the world and in Western Africa. It is abundant in Nigeria especially in the eastern and western parts and is commonly planted along roads and in villages for shade. The seed-pod is explosively dehiscent when ripe by which it received the West Indian name of "monkey's dinner bell" and also "the French bombardier" [15]. If the unripe seed pod is pierced and boiled in oil, its explosiveness is lost. It then constitutes the "sandbox" for dusting sand in writing (a precursor of blotting paper) from which the other names derive. It has been reported that the seed contain oil and according researchers the oil was found to be useful in industries for food, feed, paints, and cosmetics [16].

Most literature found on the seeds of the sandbox tree (*Hura crepitans* Linn) was limited to its agricultural applications and determination of its chemical composition. However, a study

carried out by Awosanya Abayomi [17] showed the suitability of sandbox seed oil alkyd on paint formulation. He characterized the oil obtained from the sandbox tree and obtained various properties and values leading to his classification of the oil. However, his work is limited because it was based on theories rather than facts as he never actually used the oil derived from the seed of the sandbox tree in the formulation of paint.

Idowu et al. [18] investigated the effect of moisture content on the engineering properties of sand box seed. The effect of the moisture content on the physical properties such as linear dimensions, mass, volume, porosity, and bulk density were studied. The result showed that the properties increased with increase in moisture content in the range of the moisture content investigated. The findings from the research showed good agreement with some of the general trend and ranges obtained for other similar crops. He suggested that his experimental data will be useful in the design and development of the appropriate machines for handling and processing of the seed.

Oyelek GO et al. [16] studied on the chemical examination of Sandbox (*Hura Crepitans*), showing the characterization, proximate, elemental and fatty acid profile. The purpose of the study was to investigate further on the proximate, elemental as well as fatty acids profile of sandbox (*Hura crepitans*) seed in order to give adequate information on its suitability for consumption. The proximate composition showed that the seed contained appreciable quantity of useful substances (crude protein, fat, ash and carbohydrate) with crude fibre which is desirable in digestibility, decrease in blood cholesterol and reduces the risk of large bowel cancer. The minerals and the fatty acid profile were also significant. The seed could therefore be used as one of the underutilized food source to solve the problem of mal-nutrition associated with food (balanced diet) among developing and under developed countries of the world. However, conclusions drawn from this research does not still recommend the seed or its oil derivative for food consumption. Further literature suggests that the seed of the sandbox is not good for human consumption and also states its use as fish poison in some other countries of the world [15].

This study is aimed at synthesizing alkyd resin from sandbox seed oil and comparing the

structural characteristics with a commercial alkyd resin.

## 2. MATERIALS AND METHODS

The materials used include: Sand box seed, water bath, kinematic viscometer, industrial blender, burette, electronic weighing balance, 500 ml reactor flask, soxhlet extractor, thermostatic heating mantle, condenser, 2 mm sieve, kipp's apparatus, FTIR – 8400 s (Fourier Transfer Infrared Spectrophotometer). The reagents include: n-hexane, glycerol, phthalic anhydride, HCl and xylene.

### 2.1 Seed Oil Extraction

The seeds were removed from their pods and then sun dried properly for about three (3) weeks. After the sun drying process, the seeds were also put an oven and allowed to stay for two hour at temperatures of about 40°C – 50°C. The seed samples were then ground using an industrial grinding machine and then passed through a 2 mm sieve to obtain a finer sample size after which it was stored in a desiccator for further study.

The oil from the seed of the sandbox tree was extracted using soxhlet extraction method [19]. The extraction process was done using n-Hexane of Analar grade (Nave and Dave, England) of boiling range 64-70°C for 24 hours

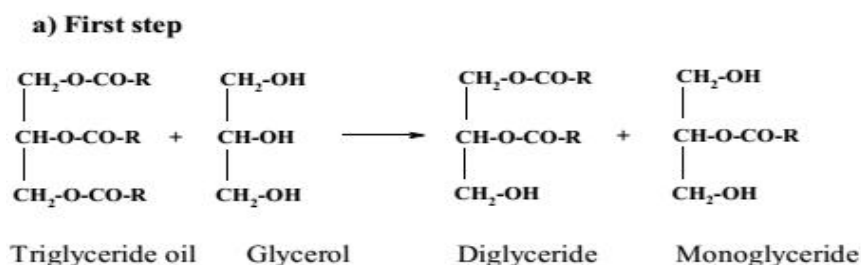
[20]. The extracting solvent was evaporated leaving the concentrated oil sample for analysis. The extracted oil was characterized for colour (physical examination), density, free fatty acid value (ASTM D1398—58), iodine value (Wij's method), peroxide value, saponification value (ASTM D5558) and acid value (ASTM D974).

### 2.2 Synthesis of Alkyd Resin

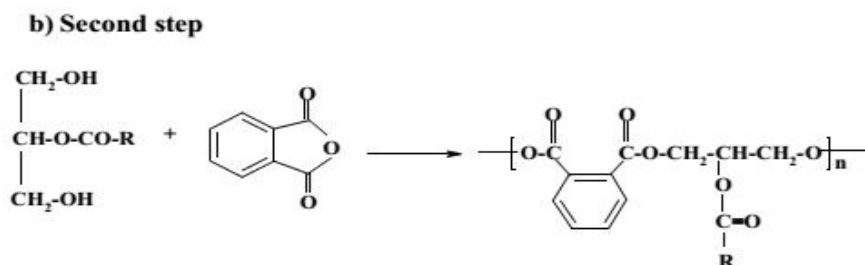
The alkyd resin synthesis involves a two stage process, which include:

Alcoholysis stage (Scheme 1) where the seed oil, Glycerol, antioxidant and the initiator were charged into the reactor. The mixture was then heated to 270°C under a CO<sub>2</sub> environment and maintained at that temperature until a sample obtained from the reactor gave clear ethanol.

The second stage (Scheme 2) is the esterification process; after alcoholysis the reactor contents were cooled to 160°C, phthalic anhydride, hydrochloric acid (HCL), antioxidant and xylene were added to reactor. The mixture was then heated to 250°C at that temperature; a xylene/water azeotrope was collected overhead. The rest of the xylene was added to maintain temperature. After 2 hours the acid number and viscosity was determined and the reaction stopped. The resultant product obtained was an alkyd.



Scheme 1



Scheme 2

The resultant alkyd was characterized using Fourier Transfer Infrared (FTIR) to determine the functional groups present and compared to a commercial alkyd resin.

### 3. RESULTS AND DISCUSSION

#### 3.1 Physicochemical Properties of the Sandbox Seed Oil

The 0.91 g/cm<sup>3</sup> density observed in Table 1 below shows similar values as those found in the literature such as 0.92 g/cm<sup>3</sup> [17] and 0.91 g/cm<sup>3</sup> [21]. The acid value of 149.066 mgKOH/g is much higher when compared with the acid value of the seed flour of 7.29 mgKOH/g [21], this high acid value may be as result of soil conditions. The high acid value proves that the oil is not edible; this is because, for edible oil, the acid value is not more than 3.00 mgKOH/g, but high acid value oil is used in soap making. The oil can be classified as long oil since the weight percentage fatty acid content is greater than 55%. Also the high free fatty acid value could be attributed to the high acid content of the oil. The saponification value of the oil of 136.466

mgKOH/g shows that the oil would be a very good material in soap making. The iodine value of 20.928 mmglodine/g obtained is similar to that reported by Oderinde et al. [22] where he obtained 20.81 mglodine/g, and this suggests that the oil is a non-drying oil. The peroxide value of 34.769 mg/g obtained is not within the rancid range and therefore suggests that the oil can withstand deterioration or decomposition for a long period of time under normal conditions.

#### 3.2 Strutral Analysis of the Alkyd Resins

Fig. 1 and Fig. 2 show the FTIR spectra of the alkyd resins and identified similar functional groups in the alkyd samples. The presence of absorption band at wavelength above 300 cm<sup>-1</sup> is a typical characteristic of organic compounds. In the spectra of the oil modified alkyd resin sample, the absorptions at 3434.84 cm<sup>-1</sup> and 1710.60 cm<sup>-1</sup> confirms the presence of hydroxyl group and carboxylic acid respectively. In the spectra of the commercial alkyd resin, the hydroxyl and carboxylic acid groups stretches were seen at 3453.23 cm<sup>-1</sup> and 1725.81 cm<sup>-1</sup>

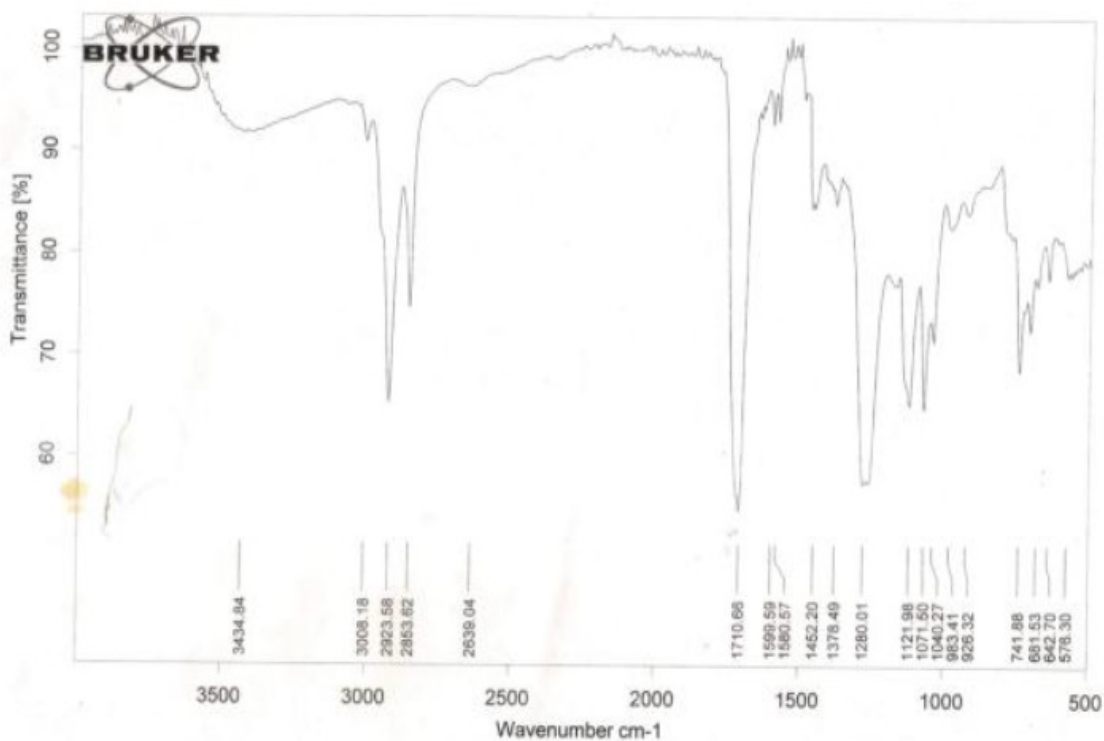
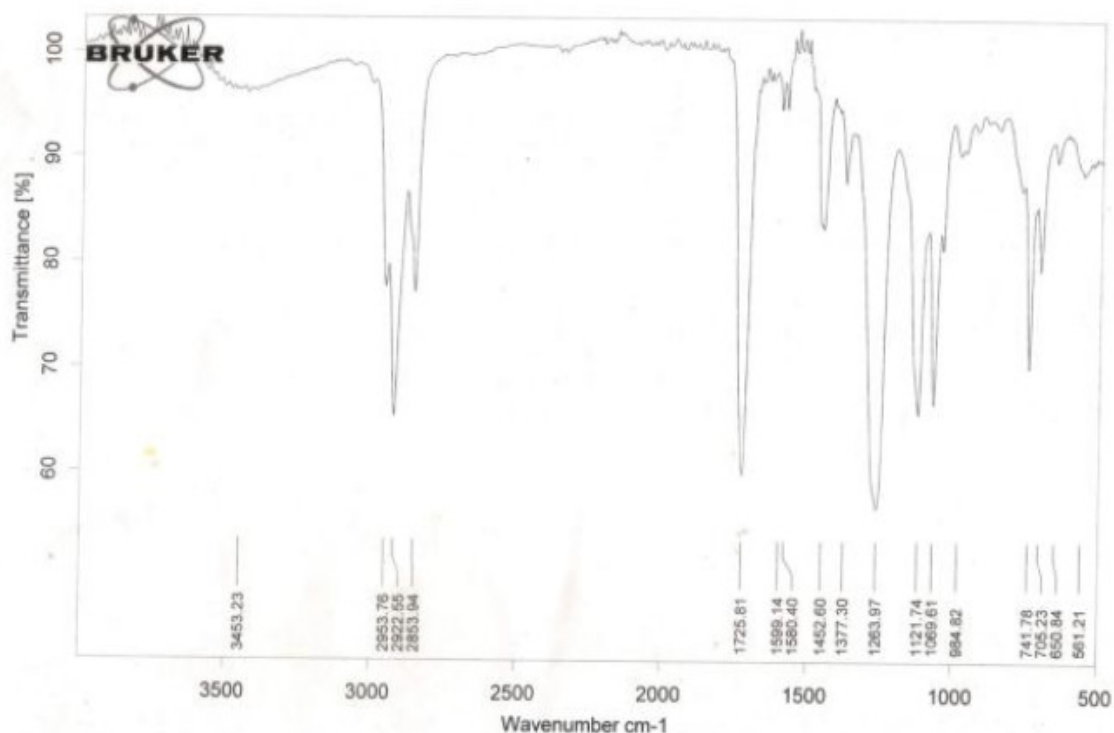


Fig. 1. FTIR spectra of an alkyd resin synthesized from sandbox seed oil (Sample A)



**Fig. 2. FTIR spectra of a commercial alkyd resin from a surface coating manufacturing industry (Sample B)**

respectively. The additional moderate peaks (out-of-plane bends) at the range of  $741.88/576.30\text{ cm}^{-1}$  support the presence of OH group in the oil-modified alkyd while in the commercial alkyd resin, the occurrence were seen at  $741.78\text{ cm}^{-1}/561.21\text{ cm}^{-1}$ . The peaks at  $2923.58/2853.63\text{ cm}^{-1}$  prove that the compound contains long linear aliphatic chain for the oil modified alkyd resin while it was observed at  $2923.78\text{ cm}^{-1}/2853.64\text{ cm}^{-1}$  for the commercial alkyd resin. Another important feature is the two set of peaks at  $1599.59\text{ cm}^{-1}$  and  $1580.57\text{ cm}^{-1}$  which are consistent with aromatic rings. These aromatic stretches were observed at  $1599.14\text{ cm}^{-1}$  and  $1580.40\text{ cm}^{-1}$  in the spectra of the commercial alkyd resin. Also the moderate absorption at  $3008.18\text{ cm}^{-1}$  for the oil-modified alkyd resin and  $2953.76\text{ cm}^{-1}$  for the commercial alkyd resin could be assigned to aromatic C-H stretching; this is used to support the presence of the aromatic ring bands at  $1599/1580\text{ cm}^{-1}$  and  $1599.59/1580.57\text{ cm}^{-1}$  respectively [23]. In-plane bend of the primary alcohol group were noticed at  $1280.01\text{ cm}^{-1}$  for the oil-modified alkyd resin and  $1263.97\text{ cm}^{-1}$  for the commercial alkyd resin.

The FTIR analysis suggesting similarities in the chemical structure of the alkyd resin samples

means that the oil-modified alkyd resin could also be applied in surface coating like the commercial alkyd resin.

**Table 1. showing the results of the physicochemical analysis of the sandbox seed oil**

Parameter	Value
Colour	Brown
Density ( $\text{g/cm}^3$ )	0.91
Acid Value ( $\text{mgKOH/g}$ )	149.066
Free Fatty Acid Value	74.931
Saponification Value ( $\text{mgKOH/g}$ )	136.466
Iodine Value ( $\text{mgIodine/g}$ )	20.928
Peroxide Value ( $\text{mg/g}$ )	34.769

#### 4. CONCLUSION

From the results of this research, we have observed that the FTIR analysis of alkyd resin from sand box tree oil has the similar characteristics with the FTIR analysis of the commercially available alkyd resin use in surface coatings. These indicate similar chemical structure and presence of similar functional groups. As a result alkyd resin from sandbox

seed oil can also be used as a binder in coating industry.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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