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Alkyd Resin Synthesis from Mango Seed Oil *(Mangifer Indica, Anacardiceae)*

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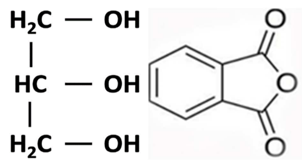
Abstract: Investigations were carried out on mango seed oil to analyze their suitability and extent of cross-linkage in alkyd resin synthesis. The oil was isolated from the seed kernel by soxhlet extraction method using n-hexane, characterized and used for bio-resin formulation. The oil yield, relative density, free fatty acid value, iodine value, and saponification value, etc, were carried out according to standard. Alcholysis and esterification processes were carried out in three neck flask (2000 ml), to convert the oil to alkyd resin. FTIR spectroscopy was used to identify the absorption peaks and formation of ester bonding sites at 11698 per centimeter, with the C═O stretching at 1240, 1221, 1188 per centimetre for C—O stretching frequency. A complete disappearance of the peak at 2920 per centimetre in the alkyd resin structure, and the appearance of the peak at 1240, 1221, 1188 per centimetre indicated occurrence of oil modification. The viscosity of 448cP compared well with the commercial grade having 457cP at temperature range between 25–33°C. Other parameters compared well to the commercial alkyd resin.

Keywords: Mango Seed Oil, Alkyd Resin, Alcoholysis, Esterification, Characterization

1. Introduction

Alkyd resins are oil-based polyesters derived from poly-condensation reaction of dibasic acid ($C_6H_4(CO)_2O$), polyols or glycerol ($C_3H_8O_3$) and fatty acid ($CH_3(CH_2)_xCOOH$; {where x is the number of carbon atoms in the hydrocarbon chain}. They are bio-resins used extensively in surface coatings and binding applications, due to their reliability, superior drying speed, high gloss, sustainability, easy processability, low cost, eco-friendliness, good weathering, and biodegradability properties. The condensation reaction between carboxylic acid (—C—O═O) and (—OH—) results into the formation of this bio-polymer, with the elimination of water molecule [10].

Studies had shown that, glycerol which is a polyol compound has three hydroxyl groups, as in (Figure 1) and can exhibit hydroscopic properties, which made them miscible in water.



(a) (b)

Figure 1. a. Structure of glycerol molecule b. Phthalic anhydride dibasic acid.

Innovations in technology and improved life style had increased the over-dependency in the use of synthetic resins from petrochemical source materials. [23] observed that alkyd resin from modified soya-bean oil had considerable drying time due to the oxidation- inhibiting effect of the variable tocopherol contents of the natural oil. The great role of alkyd resin in bindings, surface coatings and paint industries has resulted into high demand of petrochemical raw materials used in the production of synthetic alkyd resins. This has affected our economy negatively, petroleum products had been the major source of our foreign revenue. Many alkyd resin chemists had explored other sources of seed oils, for alkyd resin production [6, 10, 24, 1]. [9] observed that acid value of finished alkyd resin from rubber seed oil depended on the extent of the esterification reaction. This had resulted into more studies on polymerization status of some less economical seed oils, such as linseed oil, jatropha seeds, sunflower seeds, and neem seed, castor seed oil, palm kernel seed oil, etc, [18].

Alkyd resin could be classified, based on the 'oil lengths', which is the percentage of fatty acids content of the resin. Long oil length alkyd resin has 60 – above of fatty acids, while, s short oil length alkyd resin has below 40%, [22]. The quality of alkyd resin depends on their oil length. Long oil length alkyd resin had been reported to be suitable for brush-applied coatings or primers for steel, metal surfaces and can dissolve in ordinary solvents, such as kerosene, alcohol, etc. Also, short and medium oil length resins are good for automobile or machine part coatings. Demand for alkyd resin for surface coating and adhesive industries continue to increase every day, and there is need to discover other sources in order to meet up with the industrial demands.

Mango seed is a genus of *Mangifera* which belongs to the family of *Mangifera indica L.,* and widely produce edible fruit, with attractive colour and sweet test when they are ripe. Mango kernel are always discarded after enjoying the softy fruits, to litter the surroundings. The quest for under-utilized vegetable oil in the production of alkyd resin has necessitated the approach, to extract its oil and convert it to a useful industrial raw material, such as alkyd resin. Phytochemical composition of Mango seed had been studied by some scientists, and some of their health benefits were discovered [8, 19]. They disclosed that mango kernel is a potential source of wide range of bioactive compounds and antioxidants, that has both cardio or hepatic protective effects, anti-carcinogenic, and anti-ageing effects of phenolic compounds.

(a) (b)

Figure 2. *Mango kernel and seed respectively.*

The mango seed also known as the pit of mango fruit, as shown in Figure 2a and 2b is the centre of our studies. Reports had shown that Mango seed extract has high antioxidant capacity, and have lipids rich in unsaturated fatty acids [21].

Our target therefore is to explore other under-utilized seed oils that could be modified, to serve as good source material for our local coating industries other than the over-dependency on petrochemicals sources. This will help to reduce the huge resources wasted in importation of alkyd resin for our surface or paint industries. The synthetic process of alkyd resin formulation with phthalic anhydride or phthalic acid and glycerin is illustrated in Figure 3 below.

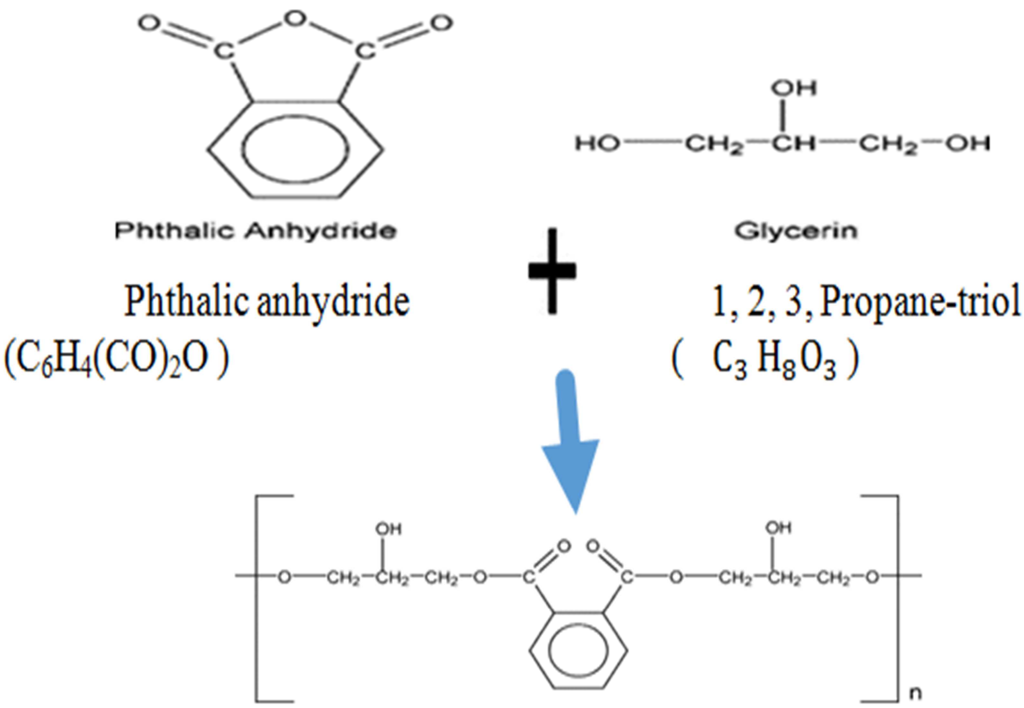


Figure 3. *The process of Alkyd resin synthesis.*

2. Materials and Method

The major raw materials are glycerine (an alcohol), phthalic anhydride (an acid), and an oil (a triglyceride) in the presence of a catalyst.

2.1. Preparation of Samples

The seed was dehulled, sundried for about 2 days to remove the moisture content, ground into fine powder, dried and stored in a dessicator for further studies. The Mango seed oil was later extracted using soxhlet extraction technique at the temperature of 64 – 70°C and n-hexane used as solvent. Some of the chemicals used in this research include, 5 g of glycerol; 0.2g of Lead oxide (as catalyst); 3 ml of 5 M methanol; 28 g of phthalic anhydride; and 2 ml of xylene, etc. The apparatus and equipment used includes: 2000 ml electric heating mantle (220volts); three-necked round bottom flask (500 ml); thermometer; stirrer (40 cm); glass beakers (500 ml); condenser (300 mm); thimble; a digital weighing balance (320g); manual grinder; Fourier Transform Infra-Red Spectrometer (FTIR) was used to determine the molecular bonds in the formulated resin chain.

2.2. Isolation of Mango Seed Oil

The mango seed kernel (*German or Sweet Mangos*) were picked around Chukwuemeka Odumegwu Ojukwu University and environs. The succulent part were removed using sharp knife. The seed stone were roasted in a drum roaster, the hull removed mechanically and the kernels crushed into small pieces of 0.01 mesh in a hammer mill. The seed mill was allowed to dry for another 4 days under daylight, to lower the percentage moisture content. Soxhlet extraction method was used to isolate the oil under a controlled temperature.

2.3. Characterization of the Seed Oil

2.3.1. Percentage Yield of the Oil

This was carried out to determine quantity of the extracted seed oil. After the standard process, the percentage yield was calculated by the formula shown below:

$$Percentage\ Yield\ (\%) = \frac{weight\ of\ extracted\ oil}{weight\ of\ dry\ seed\ powder} \times = \frac{100}{1}$$

2.3.2. Oil Colour

The oil was 20 ml filled in a glass cuvette and placed in compartment of the Gardner Scale. The oil sample's colour was observed and compared with the standard according to (< 0.5 ASTM Color).

2.3.3. Determination of Acid Value (ASTM D664)

This was estimated by titrating in the presence of phenolphthalein indicator. 7.05 g of the crude oil was weighed into 250 ml conical flask and 50 ml of 1 M. alcohol added. Few drops of the indicator was added into 0.1M sodium hydroxide solution, and agitated vigorously until a permanent pink colour appeared after few minutes.

Percentage free fatty acid was expressed as oleic acid, and was calculated as:

Acid Value = Percent free fatty acid (as Oleic acid) ×1.99 (conversion factor)

2.3.4. Estimation of Saponification Value

5 g of the oil sample was weighed into a 250 ml conical flask. 50 ml alcoholic KOH was pipetted into the flask and refluxed for 30 minutes. Allowed to cool and titrated with 0.5 M HCl using phenolphthalein indicator. Blank determination was carried out with same procedure and the value calculated thus:

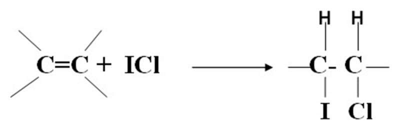
$$\text{Saponification Value} = \frac{28.05(B-S)}{Weight\ of\ Oil}$$

Where B =ml 0.5 M HCl required to titrate blank S = ml of 0.5M HCI required to titrate sample

2.3.5. Determination of Iodine Value

This test was carried out to ascertain the level of unsaturation of the oil sample. The reaction of iodine absorption by the oil is represented below (equation 1 and 2).

Equation 1. Reaction for iodine value determination.



(1)

$$ICl + 2KI \rightarrow KCl + KI + I_2) \quad (2)$$

$$I_2 + \text{starch} + 2\ Na_2S_2O_3 \rightarrow 2\ NaI + \text{starch} + Na_2S_2O_6 \quad (3)$$

The expression below was used to calculate the iodine value:

$$\text{Iodine value} = \frac{(B-S)\times M\times 12.69\times 100}{\text{Weight of oil}}$$

Where: ICl is the iodine monochloride solution (Wijs''s reagent); B is blank titre of sodium thiosulphate solution (no oil present); (B-S) is sodium thiosulphate solution; M is molarity of ($Na_2S_2O_3$). Starch was used to indicator.

2.3.6. Determination of Density or Specific Gravity

A measuring cylinder (10ml) was weighed and recorded as W_1. The sample was poured into the cylinder to the mark of 10ml, weighed and recorded as W_2, mass of sample becomes the difference between the two. Density is the ratio of mass per unit volume.

2.3.7. Synthesis of Alkyd Resin

The mango seed oil was measured (100 ml) and poured into the three necked round bottomed flask and heated to 80°C to reduce water molecules or moisture. The modification of the oil to alkyd resin was carried out in two stages:

Alcoholysis Stage

5 g of glycerol was added to 10 g of the mango seed oil (MSO) in the three-necked flask in the presence of 0.2 g of lead oxide (catalyst) to form a monoglyceride. This was heated to a temperature of 230°C, and allowed for an hour for

completion. Solubility of 1 ml of the heated sample in 3 ml methanol showed a completion of alcoholysis reaction to form monoglyceride. The sample mixture was allowed to cool to 140°C, before the esterification stage.

Esterification Stage

2.8g of phthalic anhydride, as well as 2 mls of xylene, were added to the already formed monoglyceride mixture. The mixture was heated to 230°C for 4hrs to remove water molecules. The acidity value of the mixture determines the end of the reaction. Small quantity of the mixture was removed at intervals of 1 hour, to check the pH drop. The reaction was stopped by dipping the reaction system in cold water. Afterwards the already formed alkyd resin was allowed to cool. Some properties of alkyd resin was tested and compared to the commercial grade.

2.3.8. Physico-Chemical Characterization of the Alkyd Resin

i. Determination of refractive index

Refractive index of the alkyd was determined at 30°C using Abbe's refractometer. The machine was tuned. The cell was rinsed with water, followed by acetone and allowed to dry by draining it. The oil was added into the cell and the reading taken respectively.

ii. Determination of viscosity

Viscosity of the alkyd resin was determined at 30 °C using a viscometer which was rinsed with organic solvent (acetone) and allowed to dry. A small quantity of the oil sample was added into the viscometer with micro pipette to fill the mark. At the release of each sample, a stop watch was put on and the time taken for the oil to flow from the point of origin to the calibrated mark was noted. The reading of the calibrated mark reached at an interval of time was taken and multiplied by a constant factor ($0.2483m^2/s$) to obtain the sample's viscosity.

iii. Test on drying process

The solution of each of the alkyd resin in xylene together with the drying agents (cobalt napththanate) were applied on a clear glass panel and dried at room temperature to study the film performance property. The drying process was monitored in terms of the time of set-to-touch, surface-dry and dry-through, according to [6].

iv. Determination of chemical resistance test according to [28].

It was determined under the room temperature, by coating the ends of the glass panels with wax. The panels were then dipped into water, 3% (V/V), H_2SO_4 and 1% (W/V) NaOH and were examined for the change in appearance at intervals of time and noted at the first noticeable effect or attack on the coated glass.

3. Results and Discussion

The results on the characterization of the mango seed oil *(Dasheri or German Specie),* as stated on Table 1, show that the oil is a non-drying oil, based on its saponification value of 218.383 mg KOH/g. [23], reported that, long chain fatty acid exhibits low saponification value, due to the presence of few number of carboxylic functional groups per unit mass. The saponification value of our mango seed oil is considered good as compared to that of breadfruit seed oil (161.30mg KOH/g) and Soya bean seed oil (191.50 mg KOH/g), according to [13]. This property makes mango seed oil (Dasheri or German specie) good in soap or detergent formulation, and in bio-resin formulation. Saponification value also helped us to determine the molecular weight of fatty acid. High saponification value had been proved to be better in formation of bio-resin, than those with low value.

Iodine value of our research mango seed oil (MSO) is 50.91, as stated in Table 1. The iodine value or number is a measure of the level of unsaturation or saturation of an oil. Low iodine value in oil means, high content of saturated fatty acids, while, high iodine values result to high content of unsaturated fatty acids. Studies had shown that oils with iodine values greater than 130 are drying-oil, those between "115 —130" are semi-drying oil, while, those with values less than "115" are non-drying oil, [22]. Iodine value of seed oils had been described have high degree of unsaturation, and C=C carbon-carbon double bond in the molecule. According to [16], the double bonds in the oil could be used as reactive sites in the epoxidation reaction in the synthesis of bio-resin production. However, considering the abundance of the mango seed wastes and comparative low cost of them extraction, the low iodine level of the extracted oils may be tolerated since the minimum iodine value of oil for bio-resin production is 50 mg iodine/100g oil. It is also a good parameter to determine rancidity of an oil, and confirms that the sample oil will possess a long shelf life.

The specific density of mango seed oil is 1.09 g/ml at 25°C, which is within the range of most seed oils, as the value is in conformation to the values of [3], who observed the densities of different species of mango seed oil to be within 0.80 to 1.00 g/ml at, 25°C. They concluded that the differences in the density of most seed oil are due to their various chemical composition. The density of our sample oil is slightly higher, but, compared favorably with the densities of palm oil (0.931g/ml), Hemp seed oil (0.893/ml) and soya bean oil (0.908g/ml) as reported by [17]. Mango seed oil (MSO), like other renewable oils has numerous physicochemical properties that will give an insight into their suitability in bio-resin production. The values imply that the oil is denser than water due to the absence of heavy elements or hydroxy groups in it [26]. According to [14], value of relative density of any oil gives an idea about its heaviness (dense nature) when compared with that of distilled water.

Acid value, acid number, or neutralization number, used to describe the mass of potassium hydroxide (KOH), needed to neutralize 1 gram the test sample. The lower the acid value, the fewer the free fatty acids it contains which makes it less exposed to the phenomenon of rancidification [5]. [17] described that, acid value of any oil helps to determine the tendency of spoilage and shelf life of the oil. MSO gave an acid value of 3.31%, which is higher than the palm oil (2.86 mgKOH/g oil), hemp seed oil (2.15 mgKOH/g oil) and soya bean oil (1.05 mgKOH/g oil) as reported by [25, 16]. The high acid value indicates that Mango seed oil has higher free fatty acids and carboxylic acid group compared to others, which, implies that the oil is saturated [14].

The percentage yield of MSO is 6.37% of MSO. This value was considered good due to the fact that they were picked from waste deposit. They would have been discarded by consumers and users, therefore, the low yield would be tolerated, because, the seeds are abundantly available.

Table 1. *The Physicochemical Properties of Mango Seed Oil.*

Parameter	German Mango Oils
Yield (%)	6.37
Saponification value (mgKOH/g)	218.38
Iodine value (wijs)	50.91
Peroxide value	15.71
Density (g/ml)	1.09
Free fatty acid (%)	1.62
Acid value (%)	3.31

NB: % = percentage; g/ml = gram per millilitre; mgKOH/g = milligram potassium hydroxide per gram

3.1. Results on Physico/Physiochemical Properties of Mango Seed Oil Alkyd Resin (MSOAR) as Compared to Commercial Alkyd Resin (CAR)

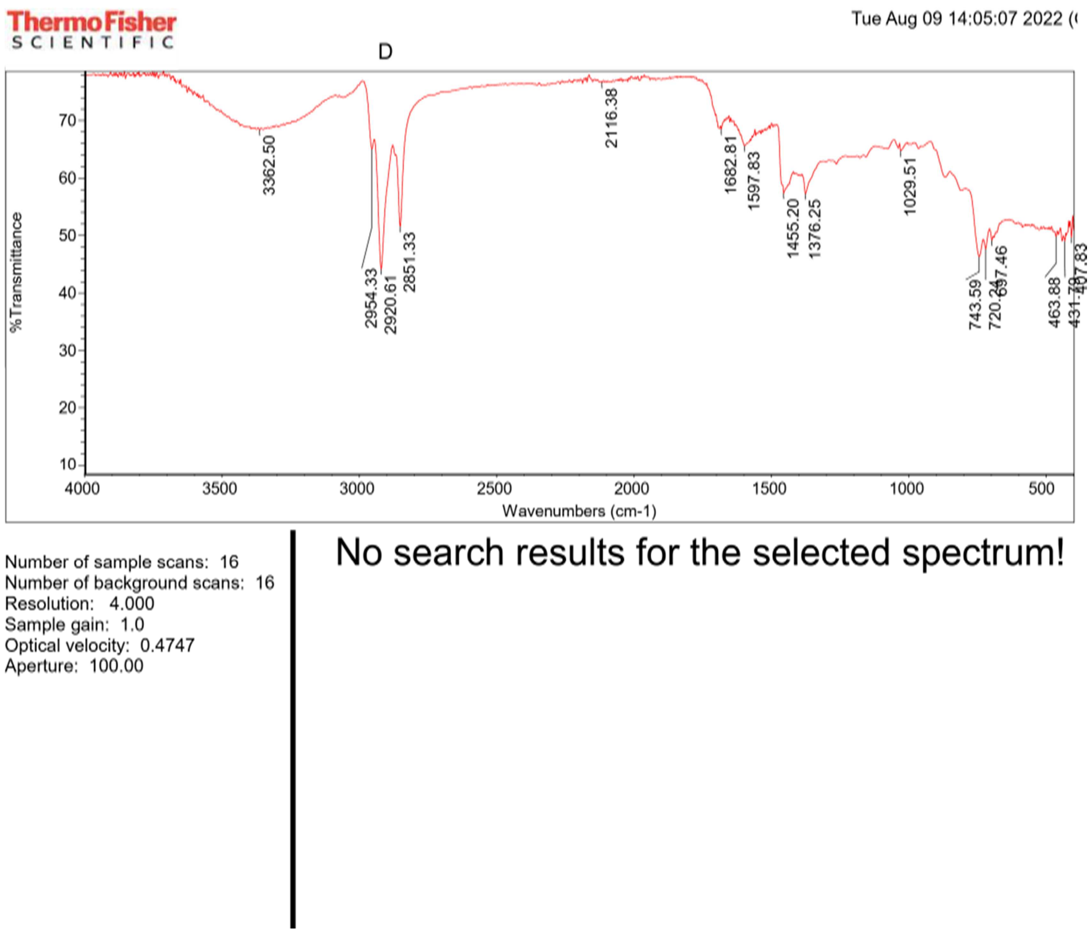
Results of some physicochemical tests carried on the mango seed oil modified alkyd resin and a branded commercial alkyd resin, to compare their properties are stated in Table 2.

The colour of the MSOAR which was tested with Gardner Scale gave the value of 14 as compared to 11 of the commercial grade alkyd resin (CAR). The differences in the colours of MSOAR and CAR, from yellow to dark orange could be attributed to the differences in their compositions and reaction system. This also shows that the sample bio-resin has the ability to reflect the colour of a coated or painted surface when applied.

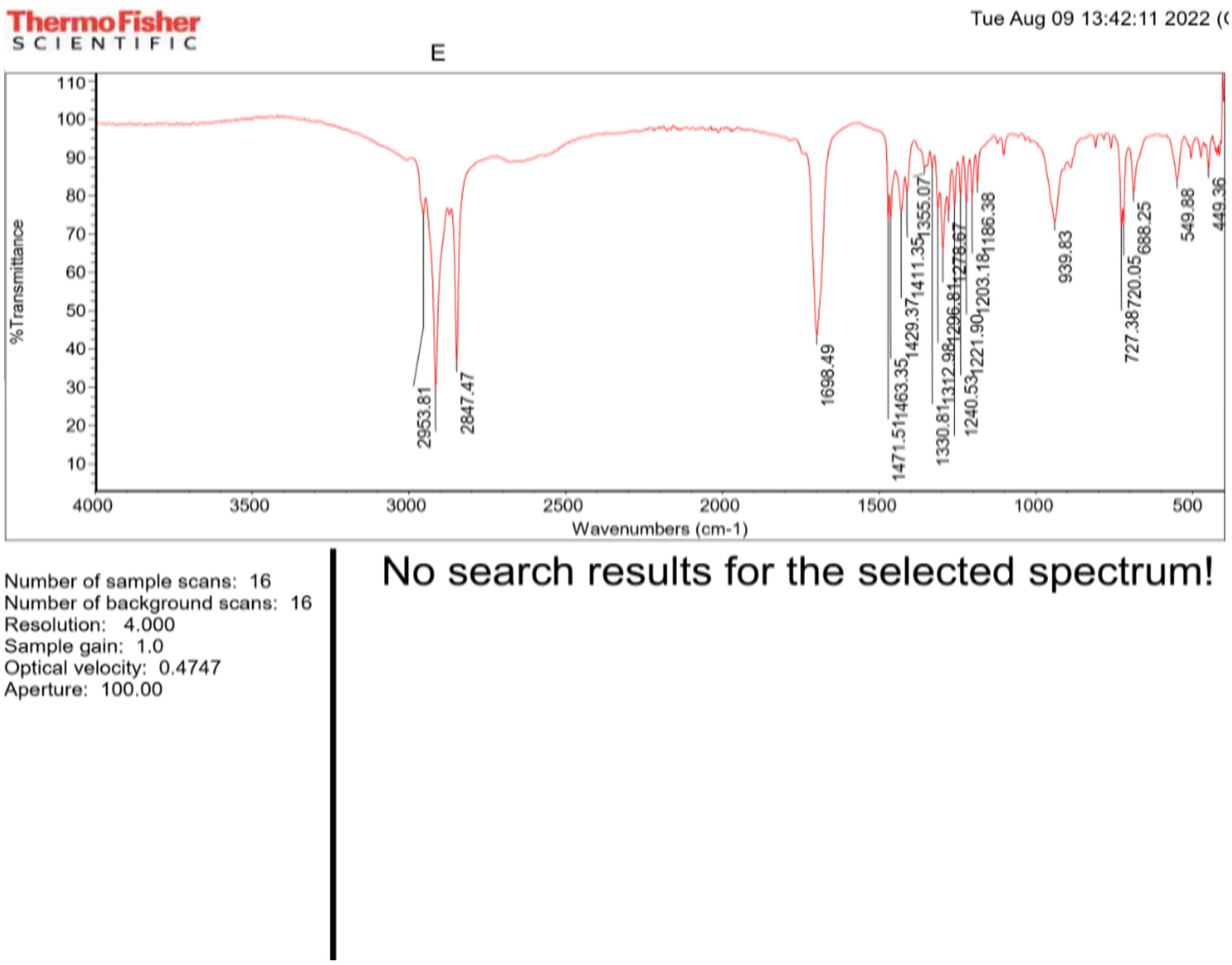
Table 2. *Results of Physicochemical Properties of MSOAR/CAR.*

Parameters	Sample Alkyd Resin	Commercial Alkyd Resin
Acid value (mg KOH/g)	10.20	10.40
Colour (Gardener Scale)	14	11
Viscosity	448cP	457cP
Gloss level	Standard	Standard
Saponification value (mg KOH/g)	333	375

The value for the viscosity of MSOAR gave 448cP as against the CAR with 457cP, as shown in Tale 2. The standard viscosity of most bio-resins fall within the range of 100 cP to 1000cP. This value shows that mango seed oil alkyd resin is a quality liquid resin which will be suitable for surface gluing, bonding and coating.



(a)



(b)

Figure 4. *a. FITR Absorption of the Mango Seed Oil; b. FITR Absorption of Mango Seed Oil Modified Alkyd Resin.*

3.2. Result of the Fourier Transform Infra-Red Spectroscopy

Fourier transform Infra-red spectroscopy (FTIR) is a technique used for both quantitative and qualitative identification of compositions of organic or inorganic samples. It is used to detect the functional groups and characterise the bonds between atoms by their infrared absorption of spectrum in a specific wavelength. The spectra produced profile of distinctive finger-prints used to determine the functional groups or bonds of the resin sample.

Some of the characteristic peaks in the Infra-Red (IR) spectra of the mango oil and the alkyd resin is represented in the above Figures 4a and 4b respectively. The formation of ester bonds is indicated by the presence of a strong peak at 11698 cm^{-1} for the C═O stretching as well as, 1240 cm^{-1}, 1221 cm^{-1}, 1188 cm^{-1} for C—O stretching frequency. This is in conformation to the findings of absorption wavelength of alkyd resin from Albizia lebbeck (Frywood) seeds, according to [23]. They discovered the peaks at the ranges of 2930.30 to 2933.83 cm^{-1} for the alkyd resin and concluded that the height of 1727 cm^{-1} was due to the C═O stretch of unsaturated cyclic ester of monoglycerides. Aromatic carbon, C═C stretching of 939 cm^{-1}, indicates the presence of phthalic systems in the resin. A complete disappearance of the peak at 2920 per centimetre in the alkyd resin, and the appearance of the peak at 1240, 1221, and 1188 per centimetre indicates that a modification of oil took place.

4. Conclusion and Recommendation

Characterization and physicochemical properties, such as, free fatty acid, saponification value, specific density, peroxide value, etc, had shown that the mango seed oil is a non-drying oil and is very suitable for bio-resin formulation. The percentage yield is commercially considerable, due to its affordability, availability, and sustainability. The use of mango seed oil from fibrous, sweet mango specie in the alkyd resin formulation will reduce the over-dependency in synthetic alkyd resin from petrochemical source materials, will not only improve the economy, control accumulation of municipal waste, but enhance our healthy condition. The quality tests like hardness, drying test, glossy test, etc, could confirm, the alkyd resin suitable for surface coating and adhesive purposes.

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