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Study of the Physico – Chemical Properties of Alkyd Resin Produced from Shea Butter Oil

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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

Alkyd resin was prepared from shear butter oil (SBO) using the two step procedures; alcoholysis and polyesterification. The oil and alkyd resin were characterized and evaluated by standard methods, for their physico-chemical properties. The analysis showed that the alkyd resin based on Shea butter oil recorded average acid value of 15.2 (mgNaOH/g), iodine value (98.0 mg/g), P^H value 6.7, density 1.36 g/cm^3, formaldehyde value of 0.05 ppm, saponification value 842.0 mgKOH/g, and Drying time 6000 seconds. The FT-IR studies showed that, the ester formation is indicated by C = O band at 1723 cm^{-1} and C – O band at 1201 cm^{-1}, and the aromatic C = C double bond at 1617 cm^{-1} band.

Keywords: Alkyd resin; shear butter oil; alcoholysis; polyesterification.

1. INTRODUCTION

Alkyd resins are widely used in the coating and paint industry and they have become essential raw materials which are used in the production of metals, wood and wood-based materials like furniture and floors, cement, cement-lime and gypsum plasters. An important property of alkyd resins is their ability to cure. It is conditioned by the presence of unsaturated bonds in the fatty

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acid chains, but above all by the presence of free functional (hydroxyl and carboxyl) groups, both in the cap groups and in the side groups of the formed structures. The distribution and the number of hydroxyl groups in polyols are critical for the drying properties of the resin, for the structure of the obtained polymer, and thus also for the properties of the resin. Esters of alcohols with four or more hydroxyl groups give coatings with good performance properties [1]. Oils from various seeds have been used in the synthesis of different kinds of polymeric resins like alkyds [2]. Certain fats and oils possess the ability when spread and exposed to air to slowly absorb oxygen forming dry, tough, transparent and durable films. These drying oils contain variety of polyunsaturated fatty acids such as linoleic and linolenic acid and their triglycerides [3].

Shea butter is an off- white or ivory-colored fat extracted from the nut of the African shea tree (*Vitellaria paradoxa*). Shea butter is a triglyceride (fat) derived mainly from stearic acid and oleic acid [4].

In this work, Shea butter oil (SBO), glycerol and maleic anhydride were employed in the synthesis of alkyd resin using alcoholysis and polyesterification method with calcium oxide as the catalyst. Physicochemical properties of the oil and the Alkyd were then investigated using standard methods.

2. MATERIALS AND METHODS

2.1 Materials and Chemicals

Shea butter oil (SBO)/*Butryrospermumparkii*., glycerol, maleic anhydride. (Sasol-Huntsman Germany), nitrogen gas.(BOC Gas Company Kaduna), xylene, catalyst (calcium oxide), methanol, carbon tetrachloride, hexane, ethanol, toluene, acetone, nitrocellulose, white spirit, methyl ethyl ketone, Wij's reagent, sodium thiosulphate, indicators, chloroform, petroleum ether.

2.2 Apparatus and Equipment

Four-necked round bottom flask, reflux condenser, thermometer, heating mantle, mechanical stirrer, beakers, viscometers, refractor meter, fourier transform spectrophotometer (FT-IR).

2.3 Preparation of Alkyd Resin

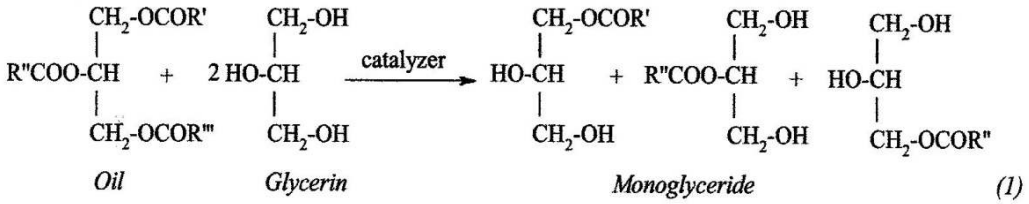
The African Shea butter oil used in this study was obtained from Samaru market, SabonGari L.G.A. Kaduna State Nigeria. Alkyd resins were synthesized by alcoholysis – polyesterification of Shea butter with maleic anhydride and glycerol. The reaction was carried out in two stages.

The first step is alcoholysis of the oil with glycerol in the presence of CaO catalyst. The oil, 100 g; 20 g of glycerol and 0.1 g of calcium oxide was charged into a liter 4-necked glass flask equipped with Dean and Stark apparatus connected to a reflux condenser with nitrogen gas. The mixture was then heated up to 180℃ for one hour under constant mechanical stirring. The completion of the reaction was verified by mixing one part of reacting mixture in three part of methanol at room temperature. The mixture was completely dissolved in methanol and gave a clear liquid indicating that the monoglycerolysis was over.

The mixture was cooled down to 150℃, and 50 g of maleic anhydride, 10 g of glycerol, were added, 10 ml of xylene was introduced to aid distilling off water molecules by azeotropical method. The temperature was gradually increased and maintained between 180℃ - 220℃. The reaction continued in this condition for 5 hours.

The main reactions that happened are:

a. Alcoholysis



Oil *Glycerin* *Monoglyceride* *(1)*

Scheme 1. Alcoholysis reaction of the oil with glycerol

Samples of the alkyd resin were synthesised (as above) at temperatures of 190℃, 200℃, and 220℃ with each product examined and bleached. Viscosity and acid value were also monitored using Brookfield viscometer and titrimetric method respectively. The discharged viscosity was 5cps while the acid value was 10 mg/KOH/g.

Finally, the polymerization was terminated by cooling to about 150℃. Thinning was done by addition of 10 ml white spirit gradually under constant stirring for the reaction to be homogeneous.

2.4 Physical Characteristic of the Oil Samples and Alkyd Resin

2.4.1 Determination of specific gravity/ density

The density of the oil was determined by the standard volume per weight measurements using a 25 cm^3 standard specific gravity (density) bottle, which was filled while kept in a thermostatically controlled water bath set at 25℃.

For the alkyd resin, standard procedure of American Oil Chemistry Society (AOAC, 1997) was used. A cleaned dried 25 ml wide mouth pycnometer was weighed (w_0). It was then filled with the sample and reweighed again (w_1). Sample was replaced with water and weighed to give (w_2)

The specific gravity of the sample was calculated using the formula.

$$S.G.= \frac{W1-W0}{W2-W0} \quad (1)$$

Where;

S.G =Specific Gravity.
w_1-w_0 = Mass of substance.
w_2-w_0 = Mass of equal volume of water.

2.4.2 Determination of colour

The sample(s) colour was determined by its physical appearance viewed under light.

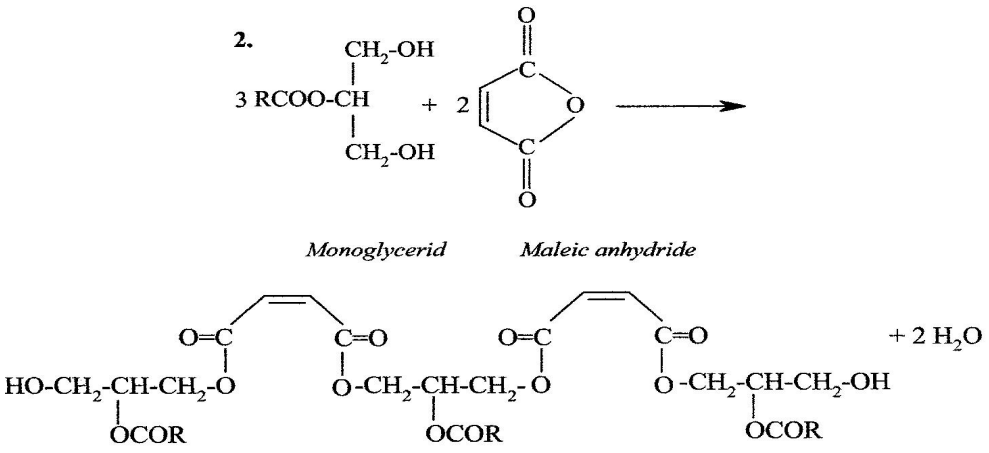
2.4.3 Measurement of viscosity

The viscosity of the Shea butter oil was determined using the NDJ-85 digital rotary viscometer at National research Institute of Chemical Technology (NARICT), Basawa, Zaria Nigeria. The sample was run at 26.7℃ test efficiency of 24.8%, time of spindle rotation 5 min, at 60 rpm. The reading was taken under laminar flow condition and reported in Mpa.s.

For the alkyd resin the Brookfield Synchro-lectric viscometer was used at the Chemical Engineering Department, Ahmadu Bello University, Zaria, Nigeria. 25 ml of the sample was poured into the beaker, with the spindle allowed to rotate from the starting point at temperature of 25℃.

2.4.4 Determination of melting / boiling point

The melting and boiling points of the oil and alkyd resin samples were determined as follows; 2 g of a sample was put into a beaker with a



Scheme 2. Esterification reaction

thermometer in place and heated on a heating mantle and observed as it melts to liquid, untill it started boiling, both temperatures were noted.

2.4.5 Refractive index

The refractive index of the samples were measured using the refractometer at Chemical Engineering department of Kaduna Polytechnic, Kaduna Nigeria.

The refractor meter was turned on and the light in the clear reading section came up. The system was allowed to warm up for about 15 minutes after which the upper prism case was opened. A drop of the sample was placed on the sample stage and closed. The prism knob was then adjusted until the critical index was observed i.e. the region of dark and bright was divided into 2 equal parts. The scale reading was taken as the refractive index.

2.4.6 Solubility in different solvents

The solubility of the SBO and the alkyd resin in various solvents were determined as follows; About 5 g of the samples was each poured into a 25 ml capacity measuring cylinder. About 5ml each of the solvents was added separately and shaken vigorously, the mixture was then allowed to stand for few minutes at room temperature and observed.

2.4.7 Solidification time

The BSI specification 1449 standard was used in the measurement of solidification time. Here equal volume of the samples were collected and heated to their melting state, after which they were allowed to solidify at room temperature and were tilted so as to notice their flow. The time at which there was no flow indicated the solidification time.

2.4.8 Drying time

ASTM, D523-1939 was employed in this test. Aluminum plates were cleaned by ethanol to ensure that there was no contaminant present to affect the test result. The sample(s) was then applied and the time of drying noted under sunlight.

2.4.9 Determination of moisture content

The mass of an empty beaker was taken using an electronic scale (w_0). Then, 5 g of the sample was added into the beaker and reweighed (w_1). The beaker was placed on a hotplate and heated at a temperature of 100℃ for one hour; the beaker was reweighted (w_2) with the difference in weights noted. The moisture content was calculated according to the equation below.

$$\% \text{ Moisture content } = \frac{w1-w2}{w1-w0} \times 100 \qquad (2)$$

2.5 Chemical Characteristics of the Shea Butter Oil and Alkyd Resin

2.5.1 Determination of peroxide value

The method employed is AOAC (920:158) method. 2 g of each of the samples were separately weighed into a 250 cm^3 conical flask. 1g of powdered potassium iodide (KI) and solvent mixture (2:1 of glacial acetic acid and trichloromethane) were then added, the solution was then placed on a water bath for few minutes to dissolve properly. 20 cm^3 of 5% of KI was then added and the solution was titrated with $Na_2S_2O_3$ using starch indicator.

The peroxide value of the oil was calculated using the formula below:

$$\text{P V} = \frac{(S-B)\times 1000\times N}{W} \qquad (3)$$

Where

S= titre value for sample.
B=titre value for blank.
N=normality of $Na_2S_2O_3$.
W=weight of the sample.
P.V= Peroxide Value

2.5.2 Determination of acid value (A.V)

The acid values of the various samples were determined by ASTM-D97400 method. 2g of the sample was weighed into 100ml beaker, 5ml ethanol was added and heated on a water bath to dissolve. The solution was titrated against 0.1MNaOH using 3-4 drops phenolphthalein as indicator, and shaken constantly until a pink colour persisted. The acid value is a conventional expression of the percentage of free fatty acid.

$$\text{A.V} = \frac{A\times M\times 40}{W} \qquad (4)$$

Where;

M = the concentration of NaOH.
A = ml of 0.1MNaOH used.
W = weight in grams of the sample.
A.V= Acid value.

2.5.3 Determination of saponification value of the sample

ASTM-D5558-95 method was used in this determination. 2 g of each of the samples was individually weighed into a 200 ml quick fit conical flask. 25 ml of ethanolic KOH solution (0.5 M) was added. A blank is also prepared by putting 25 ml of the ethanolic KOH in a similar flask. The flask and the content were refluxed for one hour in a water bath, with occasional swirling from time to time. The flask was allowed to cool a little. 1 ml of phenolphthalein indicator was added and the solution was immediately titrated with standard 0.5 MHCl solution until when the pink colour changed into colourless.

The saponification value calculated is expressed as mg KOH per g oil.

$$S.V = \frac{(S-B)\times N\times 56.1}{W} \quad (5)$$

Where;

S.V= Saponification value.
S= ml of HCl required by sample.
B= ml of HCl required by blank.
N= Molarity of HCl 56.1- Molar mass of KOH.
w = weight in gramsof sample.

2.5.4 Determination of the iodine value

The iodine value was determine using [5]. About 0.2 g of the oil sample was weighed into 100 ml conical flask, 20 ml of carbon tetrachloride CCl_4 was added to dissolve the sample followed by exactly 25 ml of the Wij's reagent that was previously prepared. The flask was immediately glass Stoppered, the solution swirled gently, a 20 ml of 10% (m/v) potassium iodide (KI) solution was added followed by 100 ml of distilled water and the solution was titrated with $Na_2S_2O_3$ solution (0.1 M), adding 1 ml of 1% (m/v) starched solution when a yellow – green colour was obtained and the titration continued until the colour just disappeared after vigorously shaking. A blank determination was carried out simultaneously without the sample under the same conditions. The iodine value was calculated from the formula expressed as;

$$I.V = \frac{(B-S)\times N\times 12.69}{W} \quad (6)$$

Where

I.V= Iodine value.
B = blank titration.
S = sample titration.
N = normality of $Na_2S_2O_3$.
W=weight in grams of sample.
12.69 = atomic weight of iodine.

2.5.5 Determination of formaldehyde emission using UV-Spectrophotometer

To determine any possible absorbance by formaldehyde, deionized water was used as the blank. The cuvette was rinsed several times with tap water followed by deionized water, it was then filled with deionized water, and placed in the holder,and the spectrophotometer was blanked at 563 nm. The sample was then put into another cuvette and the absorbance was noted at the same wavelength of 563 nm, with concentration recorded.

2.5.6 P^H determination

The sample 2 g was poured into a clean dry 25 ml beaker and 13 ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled to 25℃. The P^H electrode was standardized with buffer solution and the electrode immersed into the sample and the P^H value was read and recorded.

2.5.7 Fourier transform infrared spectroscopy analysis (FT-IR)

The chemical composition of the SBO and the alkyd resins produced at various temperatures was confirmed by Shimadzu FT-IR 8400s Fourier Transform spectrometer at NARICT Basawa Zaria. The FT-IR equipment was operated with wavelength range of 4500-500 cm^{-1}.

The sample was characterized using KBr disc sampling method. The discs were prepared with SBO and the Alkyd resin then compressed into a disk and analyzed with the spectrophotometer. The spectra were recorded over the range with results shown in Fig. 3.1.

2.5.8 Resistance to solvent medium

The effect of acid, brine, water and alkali on the alkyd resin samples was investigated. The test painted panel were immersed in the solution mixture of alkali 0.1M KOH, acid 0.1 M H_2SO_4,Brine 5% w/w NaCl and water (cold).The panel were removed after 30 minutes immersion dried and examined for resistance using ASTM method.

3. RESULTS AND DISCUSSION

3.1 Physical Characterization of Shear Butter Oil (SBO) and Alkyd Resin

The shear butter oil (SBO) has a milky cream appearance, with refractive index of 1.471 showing that it is slightly thinner than most drying oils whose refractive indexes were between 1.475 and 1.485 (Duel, 1951). However, converting the SBO to Alkyd resin, the refractive index becomes higher (1.630) in magnitude as expected, and hence relatively thicker. The shear butter oil is less dense than water with specific gravity of 0.929, but the density of the alkyd resin is higher than that of water (1.18 – 1.68) depending on the processing temperature and treatment.

3.2 Chemical Characterization of Shear Butter oil (SBO) and Alkyd Resin

The saponification value of the oil was 153.896 mg KOH g^{-1} which is lower than the values obtained for some vegetable oils ranging from 188-196 mg KOH g^{-1}. This probably indicates that the SBO contains low proportion of lower fatty acids.

Converting the SBO to alkyd resin tremendously increases the saponification value (821-877 mg KOH g^{-1}).

Drying and Solidification time. Another important property of alkyd reins which of course, is the most critical to their application as binder is the drying schedule. This is the ability of the alkyd to dry to hard and durable film by the process of autoxidation. This is related to the amount of the double bond present in the oil as measured by the iodine value [6]. The result in Table 3.1 showed that the alkyd was set to touch after 1 hour: 30 miuntes to 2 hours. Complete surface drying takes 8-9 hours.These medium oil alkyd gives slower initial drying due to thermosetting of the oil with gloss retention. Base on this property of the alkyd resin it could be used as binder in surface coating formulation.

Formaldehyde emission value: The low value of the resin (Table 3.2) is attributed to its eco-friendly in binder. High threshold level that becomes harmful during exposure to man (Daniel, 1964)

Acid value of 7.854 mg/KOH/g was obtained (Table 3.2) from the Shea butter oil when compared with those reported by FAO/WHO Alimentation Commissions (CAC) International standards for edible oil like coconut oil (7.50 mg/KOH/g), groundnut oil (4.00 mg/KOH/g) and olive (17.00 mg/KOH/g), cotton seed (0.90 mg/KOH/g), soya beans (3.40 mg/KOH/g). The lower the acid value of an oil the fewer the free fatty acids it contains which makes the phenomenon of rancidification less (Aigbodion et al. 2001).Lower acid value implies a rather stable oil at the extraction temperature. The five alkyd samples produced with maleic anhydride as shown in Table 3.4 had relatively low acid value, which is an advantage, since higher values, would contribute to corrosion. The variation in acid value of the samples may be attributed to changes in temperature, bleaching agents and acid value of maleic hydride (1144.2 mgKOH/g). A high acid value of oil could be due to hydrolytic reaction during processing of the oil as result of enzymatic action in the oil bearing seed [7].

Iodine value; Table 3.2 showed the iodine value of SBO as 92.214 mg/g. The iodine value is a vital parameter employed in ascertaining the suitability of oil for alkyd synthesis. It shows the level of unsaturation of the oil. This result is indicative of the fact that SBO is quite suitable in alkyd synthesis as its level of unsaturation will accommodate the cross-linking reaction for alkyd to form dry, hard, solid film [8]. The iodine value obtained for Shea butter oil above, classified the oil as a non-drying oil. Non-drying oils have iodine values less than 100 [9], The iodine value of Shea butter oil is lower than those of sunflower 110-143, Soya beans 120-143, and Rubber seed oil 134.51 [10]. The low iodine value for Shea butter oil indicates that the oil is rich in saturated fatty acids, which ensures stability against oxidation and rancidification of food prepared with the oil [11]. The alkydresin iodine value increases considerable as compared to that of the crude oil, it could be as a result of dimerization and polymerization reaction at the reaction double bond of the maleic anhydride yielding to a stereo structure of the alkyd molecular bigger [12].

3.3 Fourier Transform Infrared (FT-IR) Spectroscopy

The FT-IR spectrum of the Alkyd resin is shown in Fig. 3.1. The ester formation is indicated by C = O band at 1723 cm^{-1} and C – O band at 1201cm^{-1}. The band at 1617 cm^{-1} is due to the aromatic C = C double bond, and the C – H band is prominently shown at 2929 cm^{-1}. Similar results were obtained by other researchers; Isaam and Cheun, 2009.

Table 3.1. Physical characterization of Shear butter oil (SBO) and Alkyd resin

S/No	Characterization	Sample 5 bleached with hydrogen peroxide (220°C)	Sample 4 bleached with activated charcoal 220°C	Sample 3 220°C	Sample 3 200°C	Sample 3 190°C	SBO
1	Relative Density	1.23	1.49	1.36	1.18	1.68	0.929
2	Colour	Golden yellow	Black	Milky yellow	Milky yellow	Light yellow	Milky cream
3	Viscosity (cps)	15.5	7.5	24.0	32.5	46.0	124.5
4	Melting point	55-60°C	55-70°C	55-65°C	55-60°C	55-64°C	55°C
5	Refractive Index	1.497	1.593	1.630	1.630	1.630	1.471
6	Solidification time	5400	3600	6000	6000	4800	
7	Moisture content						9%

Table 3.2. Chemical characterization of Shear butter oil (SBO) and Alkyd resin

S/No	Characterization	Sample 5 Bleached with hydrogen peroxide (220°C)	SAMPLE 4 Bleached with activated charcoal 220°C	Sample 3 220°C	Sample 3 200°C	Sample 3 190°C	SBO
1	Acid value (mgNaOH/g)	16.00	22.20	15.00	3.90	8.90	7.85
2	Peroxide value (mg/g)						6.000
3	Saponification value (mgKOH/g)	877.5	867.0	842.0	821.4	823.0	153.9
4	Iodine value (mg/g)	108.0	112.0	98.0	94.0	89.0	92.2
5	Formaldehyde emission (PPM)	0.07	0.03	0.05	0.05	0.03	
6	pH value	6.8	6.3	6.7	6.5	6.4	

Table 3.3. Solubility test of SBO and the Alkyd resin

S/No	Solvent	Sample 5 Bleached with hydrogen peroxide (220°C)	Sample 4 Bleached with activated charcoal 220°C	Sample 3 220°C	Sample 2 200°C	Sample 1 190°C	SBO
1	Carbontetrachloride	Partially soluble	Partially soluble	Partially soluble	Partially soluble	Partially soluble	Partially soluble
2	Chloroform						Soluble
3	Ethanol	Limited solubility	Limited solubility	Limited solubility	Limited solubility	Limited solubility	Soluble
4	Hexane	Partially soluble	Partially soluble	Partially soluble	Partially soluble	Partially soluble	Soluble
5	Methanol	Soluble	Soluble	Soluble	Soluble	Soluble	
7	Nitrocellulose	Soluble	Soluble	Soluble	Soluble	Soluble	
8	Petroleum ether						Partially Soluble
9	Toluene	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	
10	Water						Insoluble
11	White Spirit	Soluble	Soluble	Soluble	Soluble	Soluble	
12	Xylene	Soluble	Soluble	Soluble	Soluble	Soluble	

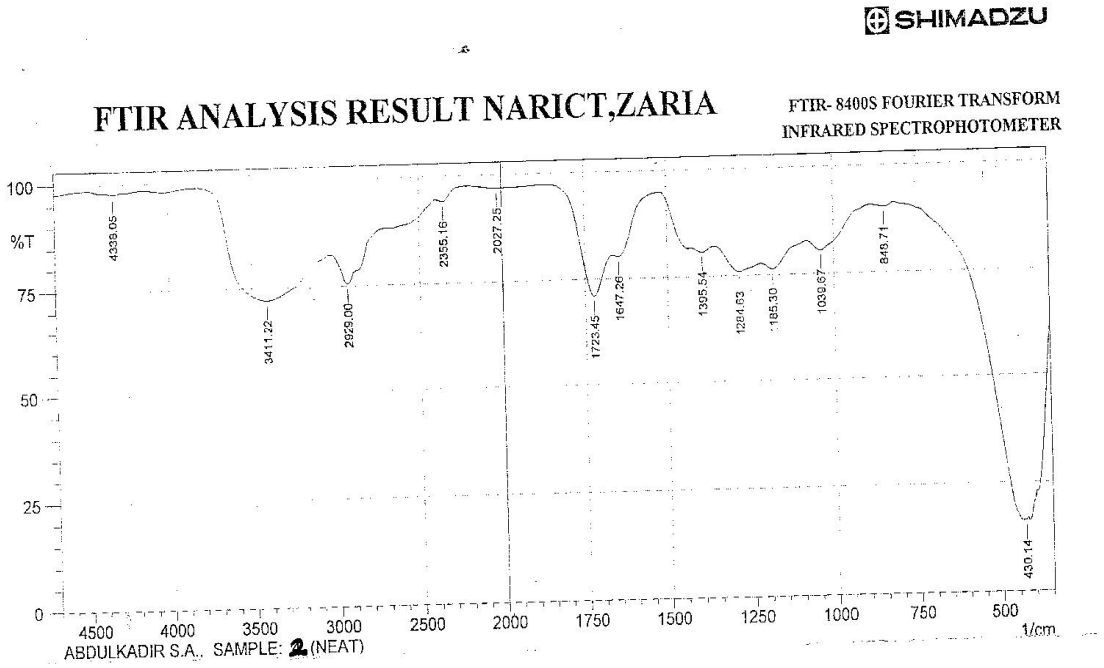


Fig. 3.1. FT-IR spectrum for Alkyd resin based on shear butter oil

Table 3.4. Resistance of Alkyd resin to some agencies

S/No	Agencies	Remarks
1	Alkali (0.1m KOH)	Film removed
2	Acid (0.1M H_2SO_4)	Film not removed
3	Brine (5% w/w NaCl)	Film not removed
4	Water	Film not removed

However in this work, the band indicated at 3411 cm^{-1} could be due to the residual O – H bond after the esterification reaction.

The solubility of the Alkyd resin was tested and it was found to be soluble in solvents such as Methanol and Xylene, partially soluble in Hexane and Carbontetrachloride. It was however found insoluble in Toluene.

The alkyd resin based on SBO was shown to be resistant to Acid and Brine solution but susceptible to Alkali, as indicated in Table 3.4.

4. CONCLUSION

The physico-chemical characterization of the Shea butter oil indicated that it is non-drying in nature based on the iodine value, and it is suitable for alkyd resin preparation. The extent of polymerization of the alkyd resin synthesized reveals the formation of high molecular weight, and exhibited excellent resistance to acid, brine, and cold water, but fairly resistance to alkali. Its resistance to different service media is consistent with observable resistance of oil-paint to these media when applied as surface coating.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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