

Determination of the Physico-Chemical Properties of Alkyd Resin Produced from Natural Shea Butter Oil

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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), PH value 6.7, density 1.36 g/cm³, 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 acid chain, 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)/ *Butyrospermumparkii*., 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, Wj'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, Sabon Gari 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°C 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.

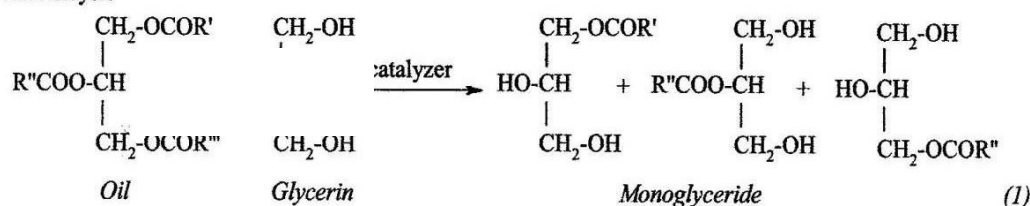
The mixture was cooled down to 150 °C, and 50g of maleic anhydride, 10g of glycerol, were added, 10 ml of xylene was introduced

to aid distilling off water molecules by azeotropic method. The temperature was gradually increased and maintained between 180°C – 220 °C. The reaction continued in this condition for 5 hours.

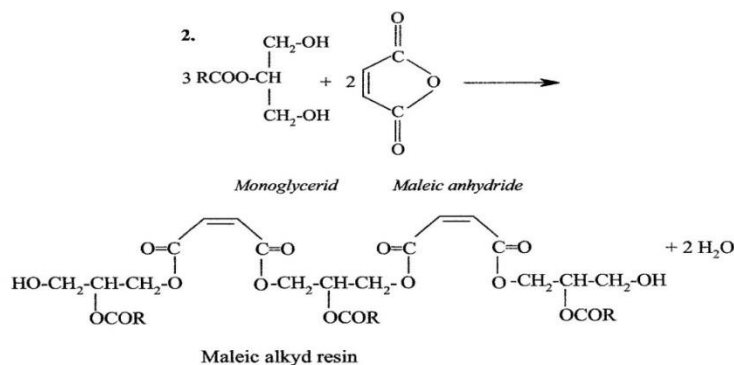
Samples of the alkyd resin were synthesised (as above) at temperatures of 190 °C, 200 °C, and 220 °C with each product examined and bleached. Viscosity and acid value were also monitored using Brookfield viscometer and titrimetric method respectively. The discharged viscosity was 5cps while the acid value was 10 mg/KOH/g Finally, the polymerization was terminated by cooling to about 150 °C. Thinning was done by addition of 10ml white spirit gradually under constant stirring for the reaction to be homogeneous (Ogunniyi and Odetoye 2007).

The main reactions that happened are:

a. Alcoholysis



Scheme 2.1. Alcoholysis reaction of the oil with glycerol



Scheme 2.2 Esterification of Monoglyceride with Maleic anhydride.

3.0 Physico – chemical the Shea butter oil and the alkyd resin produced.

3.1.1. Determination of the Density of the Oil:

The density of the oil was determined by the standard volume / weight measurements using a 25 cm³ standard specific gravity (density) bottle, which was filled while kept in a thermostatically controlled water bath set at 25 °C.

3.1.2 Determination of Colour of the Oil: The samples colour was determined by its physical appearance viewed under light.

3.1.3. Determination of Viscosity of the Oil: The viscosity of the Shea butter oil was determined using the NDJ-85 digital rotary viscometer at National Research Institute for Chemical Technology (NARICT), Basawa, Zaria Kaduna State. The sample was run at 26.7 °C 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. For the Alkyd resin the Brookfield Synchro-lectric viscometer of the Chemical Engineering Dept.

ABU Zaria was used. 25ml of the sample as poured into the beaker, with the spindle allowed to rotate from the starting point at temperature of 25 °C

3.1.4. Determination of Melting and Boiling Points: The melting and boiling points were determined using the recommended method (AOAC, 1997). 2 g was taken into a beaker with a thermometer in place and heated on a heating mantle and was observed for melt as it changed to liquid, till it started boiling, both temperatures were noted and recorded.

3.1.5. Determination of Specific Gravity/Density: 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.

$$S.G. = \frac{W_1 - W_0}{W_2 - W_0} \quad 2.1$$

where; S.G = Specific Gravity.

$W_1 - W_0$ = Mass of substance.

$W_2 - W_0$ = Mass of equal volume of water.

3.1.6. Refractive index: 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.

3.1.7. Solubility with Suitable Solvents: About 2 g of the sample was poured into a 25 ml capacity measuring cylinder. About 5ml each of the solvent was added separately and shaken vigorously and the mixture was allowed to stand for few minutes at room temperature and observed.

3.1.8. Solidification Time: Equal volume of the sample were collected and heated to their melting state, after which they were allowed to solidify at room temperature and where tilted to notice the flow. The time at which there was no flow indicated the solidification time. The BSI specification 1449 standard was used.

3.1.9. Drying Time: In accordance to American standard for testing material (ASTM, D523-1939)

Aluminum plates were cleaned by ethanol to ensure that there was no contaminant present to affect the test result. The sample was then applied and the time of drying noted under sunlight.

3.1.10 Determination of Moisture Content: The mass of an empty beaker was taken using an electronic scale (W_0). 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°C for one hour; the beaker was reweighed (W_2) with the difference in weights noted. The moisture content was calculated according to the equation below.

% Moisture content

$$= \frac{W_1 - W_2}{W_1 - W_0} \times 100 \quad 2.2$$

3.1.11. Gas Chromatography- Mass Spectrometry Analysis (GC-MS): The GCMS of the oil sample was determined to give the molecular weight of the compound present in the oil compared with the expected result from the literature. (GCMS-QP2010 plus Shimadzu Japan) at NARICT, Basawa Zaria was used for running this test.

3.2 Chemical Characterization of the oil and Alkyd Resins.

3.2.1. Determination of Peroxide Value: The method employed is AOAC method 920:158.2 g sample was weighed into a 250cm³ conical flask. 1g of powdered potassium iodine (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 cm³ of 5% KI was then added and the solution was titrated with Na₂S₂O₃ using starch indicator. The peroxide value of the oil was calculated using the formula below.

$$P.V. = \frac{(S-B) \times 1000 \times N}{W} \quad 2.3$$

where S= titre value for sample.

B=titre value for blank.

N=normality of Na₂S₂O₃.

W=weight of the sample.

P.V= Peroxide Value

3.2.2 Determination of Acid Value (A.V): The Acid values of the various samples were determined by ASTM method (ASTM-D97400). 2 g of the sample was weighed into 100 ml 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.

$$A.V = \frac{A \times M \times 40}{W} \quad 2.4$$

where; M= the concentration of NaOH.

A=ml of 0.1MNaOH used.

W=weight in grams of the sample.

A.V= Acid value.

3.2.3. Determination of Saponification: An American standard for testing material (ASTM) method (D5558-95) was used for the determination. 2g sample was weighed into a 200 ml quick fit conical flask. 25 ml of ethanolic KOH solution (0.5M) was added. A blank was 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. 1ml of phenolphthalein indicator was added and the solution was immediately titrated with standard 0.5MHCl solution until when the pink colour changed into colourless. The saponification value calculated is expressed as mg KOH per g oil.

$$S.V = \frac{(S-B) \times N \times 56.1}{W} \quad 2.5$$

where; S.V= Saponification value.

S= ml of HCl required by sample.

B= ml of HCl required by blank.

N= normality of HCl 56.1- Molar mass of KOH.

W = weight in grams of sample.

3.2.4. Determination of the Iodine Value:

About 0.2 g of the oil sample was weighed into 100ml conical flask, 20ml 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 stopped, the solution swirled gently, a 20 ml of 10 % (m/v) potassium iodide (KI) solution was added followed by 100ml of distilled water and solution was titrated with $Na_2S_2O_3$ solution (0.1M), adding 1ml 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 using (AOAC.1997) from the formula expressed as;

$$I.V = \frac{(B-S) \times N \times 12.69}{W} \quad 2.6$$

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.

3.2.5 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.

3.2.6. pH Determination: The sample 2g was poured into a clean dry 25 ml beaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly .It was then cooled to 25°C. The pH electrode was standardized with buffer solution and the electrode immersed into the sample and the pH value was read and recorded.

3.2.7. 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. All spectra were recorded over the range with results shown in Figure 4.10 and 4.11 at the Appendix.

3.2.8 Resistance to liquid medium: The alkyd resin samples were tested in various liquid medium such as acid, brine, water and alkali to measure its degree of aggressiveness when in contact. 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 (ASTM-D97400).

4.0 Results and Discussion

4.1 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.

4.2 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 resins 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 4.1 showed that the alkyd was set to touch after 1 hour: 30 minutes 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. Based 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 4.1) 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 4.1) 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 4.1 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; the iodine value of SBO was measured to be 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 alkyd resin iodine value increases considerably 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].

4.2 Fourier Transform Infrared (FT-IR) Spectroscopy: The FT-IR spectrum of the Alkyd resin is shown in Fig. 4.4. 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 4.1 CHEMICAL CHARACTERIZATION OF SYNTHESISED ALKYD RESIN

CHEMICAL CHARACTERIZATION											
NO	ANALYSIS	OF	THE	BLEACHED	BLEACHED	SAMPLE	3	SAMPLE	2	SAMPLE	1

SAMPLES	HYDROGEN PEROXIDE SAMPLE 5 220°C	ACTIVATED CHARCOAL SAMPLE 4 220°C	220°C	200°C	190°C
1 ACID-VALUE mgNaOH/g	16.0	22.2	15.0	3.9	8.9
2 IODINE VALUE mg/g	108	112	98	94	89
3 pH VALUE.	1.8	2.3	2.0	2.1	2.4
4 FORMALDEHYDE EMISSION.(PPM)	0.07	0.03	0.05	0.05	0.03
5 SAPONIFICATION VALUE. mgKOH/g	877.5	867.0	842.0	821.4	823.0

TABLE 4.2 PHYSICAL CHARACTERIZATIONS OF SYNTHESISED ALKYD RESINS

NO	ANALYSIS OF THE SAMPLES	BLEACHED HYDROGEN PEROXIDE SAMPLE 5 220°C	BLEACHED ACTIVATED CHARCOAL SAMPLE 4 220°C	SAMPLE 3 220°C	SAMPLE 2 200°C	SAMPLE 1 190°C
1	DENSITY	1.23	1.49	1.36	1.18	1.68
2	MELTING POINT	55-60°C	55-70°C	55-65°C	55-60°C	55-63°C
3	COLOUR	GOLDEN YELLOW	BLACK	MILKY YELLOW	MILKY YELLOW	LIGHT YELLOW
4	DRYING-TIME UNDER(SUN) @39°C (S)	5400	3600	6000	6000	4800
5	REFRACTIVE INDEX @25°C	1.496	1.593	1.630	1.630	1.63
6	VISCOSITY CP	15.5	7.5	24.0	32.5	46.0

TABLE 4.3 SOLUBILITY TESTS OF THE ALKYD RESINS

ANALYSIS OF THE SAMPLES	BLEACHED HYDROGEN PEROXIDE SAMPLE 5 220°C	BLEACHED ACTIVATED CHARCOAL SAMPLE 4 220°C	SAMPLE 3 220°C	SAMPLE 2 200°C	SAMPLE 1 190°C
XYLENE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE
	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE
METHYLETHYL KETONE	LIMITED SOLUBILITY	LIMITED SOLUBILITY	LIMITED SOLUBILITY	LIMITED SOLUBILITY	LIMITED SOLUBILITY
ETHANOL	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE
	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE	INCOMPLETE SOLUBLE
CARBON TERACHLORIDE	INSOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE
HEXANE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE
	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE	SOLUBLE
TOLUENE					
METHANOL					
NITROCELLULOSE					
WHITE SPIRIT					

TABLE 4.4 RESISTANCE OF ALKYD RESIN

ALKALI (0.1M KOH)	FILM REMOVED
ACID (0.1M H ₂ SO ₄)	FILM NOT REMOVED

BRINE (5% W/W NaCl)	FILM NOT REMOVED
WATER	FILM NOT REMOVED

FT-IR INTERPRETATION OF ALKYD RESINS

TABLE 4.5 FT-IR absorptions group.

SAMPLE	BOND	2853cm ⁻¹ 2962cm ⁻¹ C-H	1300cm ⁻¹ -1440cm ⁻¹ C-O	3590cm ⁻¹ - 3650cm ⁻¹ O-H.	1620cm ⁻¹ 680cm ⁻¹ C=C.	1710cm ⁻¹ 1780cm ⁻¹ C=O.	1710cm ⁻¹ -1750cm ⁻¹ COOR.	1020cm ⁻¹ - 1275cm ⁻¹ C-O-C	650cm ⁻¹ - C-C
	VIBRATION	Straight stretching				Stretching.			
SBO	FREQUENCY CM ⁻¹	2863-2926	1363-1453	3445-4334	-	1745	-	-	707
1	CM ⁻¹	2926	1363-1448	3444-4340	-	-	1733	-	-
2	CM ⁻¹	2926	1395	3411-4338	1647	1723	1723	1039	-
3	CM ⁻¹	2929	1439	3417-4344	1636	1729	1729	-	-
4	CM ⁻¹	2931	1388-1439	3409-4338	1645	1729	1729	-	-
5	CM ⁻¹	2875-2930	1389-1450	3403-4341	1647	1726	1726	1042-1287.	-

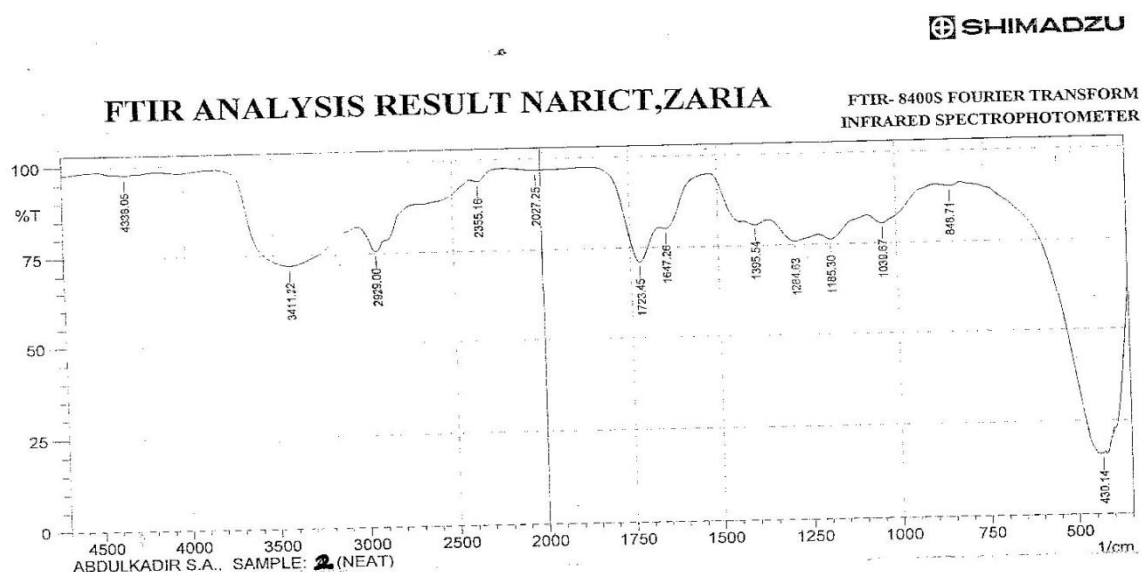


FIGURE: 4.6 FT-IR ABSORPTIONS

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 4.4.

5.0 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.

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