

COMPARATIVE ANALYSIS OF ALKYD RESINS PRODUCED FROM CASTOR AND SOYBEAN SEED OILS

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Abstract: Studies were carried out on the production of alkyd resin from castor and soybean seed oils respectively. Castor and soybean oils extracted from their respective seeds were refined to remove any unwanted materials and then characterized to determine their physicochemical properties such as acid value, iodine value, saponification value, etc. The results showed that the relatively high iodine content of soybean makes it naturally a suitable vegetable oil for the synthesis of quality alkyds, while the low iodine content of castor oil indicated that it is a non-drying oil and therefore not suitable for the production of quality alkyds. In this present study, castor oil was severely dehydrated to increase its degree of unsaturation as indicated by its high iodine content after dehydration; thus making it suitable for the synthesis of quality alkyds. Monoglyceride method was used in the synthesis of medium oil alkyd resins of 50% oil length from appropriate amount of glycerol and each oil via alcoholysis, followed by addition of phthalic anhydride by esterification. The produced resins were characterized using standard methods, tested for their coating properties such as drying time, chemical resistance, solubility in different solvents, etc. The resins were equally analyzed using Fourier Transform Infrared Spectroscopy (FTIR) to determine their structural functionalities. The results obtained showed that both soybean and dehydrated castor oil modified resins have desirable qualities, though soybean oil modified resin has preferred coating properties.

Keywords: Alkyd Resin, Soybean Oil, Castor Oil, Monoglyceride Method, Esterification.

1. INTRODUCTION

For several years, varieties of vegetable and non-edible oils have been used in paint industry as they are able to dry quickly (Ezeagba et al., 2014). The fatty acid esters derived from the triglyceride vegetable oils are attractive sources of raw materials for polymer synthesis (Shabeer et al., 2007). Moreover, vegetable oils are renewable resources; and the fast rising of petrochemical prices have increased interest in polymers made partially from renewable resources (Issam and Cheun, 2009).

Ezeagba et al. (2014), noted that the most important constituent of all conventional paints is the 'binder,' commonly called vehicle. Gabriel et al. (2013), reported that alkyd resin is the polymer binding material in paints, which is responsible for the formation of continuous film that adheres to the substrate and holds the other substances together. They are the essential raw materials in surface coatings. The demand for alkyd resins in Nigeria surface coating industry has increased tremendously (Aigbodion and Okieimen, 2001). Thus, alkyd resin has surpassed all other synthetic resins in terms of low cost, versatility, etc. Majumdar et al. (1998), had opined that the acceptance of alkyds as vehicles in surface coatings is mainly due to their film hardness, colour, durability, gloss retention, abrasion resistance, compatibility with many polymers and other resins, as well as other desirable qualities imparted on them through modification of the drying oils. By definition, alkyd resins are polyester polymer of fatty acids, derived from the poly condensation of a polybasic acid (e.g. phthalic anhydride), and a polyhydric alcohol (e.g. Glycerol), modified with monobasic fatty acid, or its triglyceride oil (Kyenge et al., 2012).

Alkyd resins are modified with drying oils to give them special drying properties. Hlaing and Mya, (2008) reported that the commonly used vegetable oils in alkyd resin production are linseed oil, tall oil, soybean oil and dehydrated castor oil. In this research, the use of dehydrated castor oil and soybean oil in the production of alkyd resin was investigated. Castor oil, valued as a non-drying oil has variety of uses like being used as a lubricant, food additives, preservatives, and can be imparted excellent drying properties through severe dehydration (Spyros, 2004). Thus the most important use of castor oil is in the form of dehydrated castor oil (Hlaing and Mya, 2008). Onukwuli and Igbokwe (2008), reported that dehydrated castor oil is known for its non yellowing film formation, outstanding colour retention, high degree of unsaturation, flexibility and adhesion in protective coatings.

Soybean oil is one of the most readily available and widely used vegetable oil in the world. For many years, soybean oil has been a major ingredient in making alkyd resins (Kyenge et al., 2012), due to its high iodine value; which makes it valued naturally as semi-drying oil. Furthermore, the level of unsaturation in soybean oil is high because it is composed primarily of polyunsaturated Linolenic acid (C 18:3) and Linoleic acid (C 18:2), as well as monounsaturated Oleic acid (C 18:1) (Ivanov et al., 2010). This high degree of unsaturation enables it to easily polymerize into useful materials (Shabeer et al., 2007).

This work tried to compare the alkyd resins produced from soybean and dehydrated castor oils.

2. MATERIALS AND METHODS

2.1 Materials:

Castor seeds were obtained from Nkwo Ihuokpara in Nkanu East Local Government Area of Enugu State, and soybean seeds, bought from Ogbete main market Enugu. Phthalic anhydride with assay 99.7%, glycerol with assay 99.5%, xylene with assay 98%, from BDH chemicals Ltd, Poole, England; Calcium hydroxide[Ca(OH)₂], anhydrous methanol, Nitrogen (analytical grade) ethanol, sodium hydroxide(NaOH). The equipments include heating mantle, electronic weighing balance, mechanical stirrer, thermometer, 3 neck round bottom flask, dean and stark apparatus, general laboratory glass ware, fume cupboard.

2.2 METHODS:

2.2.1 Extraction of Soybean and Castor Oils:

The purchased soybean and castor seeds were first cleaned to remove the damaged seeds and unwanted materials, and the good seeds washed thoroughly with clean water, sun dried, cracked and de-hulled. The de-hulled soybeans and castor beans were ground into powdery form using grinder in order to weaken the cell wall to release soy and castor fats; and make extraction easier. The oils were extracted by the solvent method using n-hexane (Akpan et al.).

2.2.2 Refining of the Extracted Oils:

The extracted crude oils passed through refining and treatment processes, such as degumming, neutralization, and bleaching, to reduce the FFA content, remove trace elements, unwanted materials like chlorophyll and other impurities.

2.3 DEHYDRATION OF CASTOR OIL:

The refined castor oil was mixed with 0.5g of H₂SO₄ in a conical flask and poured into a three (3) neck round bottom flask, where it was heated to 250°C. The 3 neck round bottom flask was fitted with a reflux condenser, Mercury in glass thermometer and a Nitrogen opening from a Nitrogen source (Nitrogen is analytical grade). The temperature was maintained for one hour, after which the source of heat was removed. The dehydrated oil was then allowed to cool.

2.4 PREPARATION OF ALKYD RESIN:

Monoglyceride method was used in the preparation of medium oil alkyd resins with 50% oil length from Glycerol, Phthalic anhydride and Triglyceride oils (Castor and Soybean oils respectively) using Calcium hydroxide [Ca(OH)₂] as catalyst.

The preparation was carried out in a fume cupboard, using a heating mantle and 3 neck round bottom flask fitted with a mechanical stirrer, a Nitrogen inlet and a thermometer pocket. Xylene was also employed as the Azeotropic Solvent to remove any water formed. The reaction involved two stages: Alcoholysis stage and Esterification stage

ALCOHOLYSIS:

Alcoholysis of soybean oil and dehydrated castor oil was done with calculated amount of glycerol to produce monoglyceride from each oil. A measured quantity of the oil was poured into the three neck round bottom flask and heated to 120°C to remove any moisture present. A heating mantle was used to carry out the heating. 0.2g of Calcium hydroxide (catalyst) was added to the heated oil, and the mixture stirred; after which a measured quantity of glycerol was added, and the temperature was increased to a range of 230-250°C. After 30 minutes, a small quantity of the aliquot was taken to check its solubility in Methanol. Alcoholysis was completed when the sample of the mixture formed became soluble in 1 to 3 volumes of anhydrous methanol; giving a clear solution. After this, the reaction temperature was cooled to 140°C to allow smooth introduction of the esterification stage.

ESTERIFICATION:

Here, a measured quantity, each, of phthalic anhydride and xylene, was added into the flask containing the monoglyceride mixture (this was introduced gently). The mixture was continuously stirred and heated with a heating mantle. The temperature was increased and maintained at the range of 230-250°C. Sampling was carried out at periodic intervals to check the drop in acid value of the reaction mixture. The reaction was discontinued as soon as the acid value of the mixture formed dropped to about 10mgKOH/g or less.

2.5 TESTING:

FTIR Analysis: The structural functionalities of the oils and the alkyd resins produced from each oil sample were analyzed using Fourier Transform Infrared Spectroscopy.

The FTIR spectra of all the samples were recorded with Buck Scientific Infra-red Spectrophotometer (Model: M530) over a wavelength range 4000-500cm⁻¹. Procedure: 1g of each sample was mixed with 0.5g of KBr; after which 1ml of Nujol (a solvent for preparation of sample by Buck 530 IR – Spectrophotometer) was pipetted into the sample mixture with the aid of a syringe to form a paste before introducing into the instrument sample mould and allowed to scan at a wavelength of 600 – 4000nm to obtain its spectra wavelength

Solidification Time: 5g of each sample was collected and heated with a laboratory heating mantle, to melting point and allowed to cool and then solidify at room temperature. The time at which each solidified without flowing indicated the solidification time.

Drying Time: Mild steel sheets (3cm x 3cm x 1mm) were cleaned with ethanol to ensure there were no impurities. Each test sample was applied at the surface of each sheet. The time taken for the samples on the sheets to dry at room temperature is the drying time.

Set to Touch: This is the time it takes for the alkyd resin applied on a material to dry to an extent, such that there is no physical deformation on the surface when touched.

Solubility: The solubilities of the alkyd resins produced were tested and determined in four different solvents (Xylene, Ethanol, Methanol and n-Hexane). 5g of each sample was weighed into conical flasks. 5mls of each of the solvents was added separately and the mixture shaken vigorously. Each mixture was allowed to stand for 1hour at room temperature and the solubilities were observed and recorded.

Resistance to Solvent Medium: The effects of acid, brine, water and alkali on the alkyd resin were investigated. Mild steel sheets (3cm x 3cm x 1mm) were cleaned with ethanol to ensure the absence of impurities. Test samples were applied to them and replicates immersed in dilute alkali 0.1M KOH, acid 0.1M H₂SO₄, Brine (5% w/w NaCl) and distilled water separately. The metal sheets were removed after 30minutes and examined for chemical resistance.

3. RESULTS AND DISCUSSIONS**3.1 Characterization of Crude, Refined and Dehydrated Castor Oils:**

The physicochemical properties of crude, refined and dehydrated castor oil were determined according to ASTM standards and the results presented in Table 1

Table 1: The Physicochemical Properties of Crude, Refined and Dehydrated Castor Oil

Properties	Crude Castor oil	Refined Castor oil Castor Oil	Dehydrated	ASTM Range (1996)
Acid Value (mgKOH/g)	1.150	0.87	4.51	0.4 – 4.0
Saponification (mgKOH/g)	189	182	47	175 – 187
Iodine Value (gI ₂ /100g)	82.5	83	130.10	82 – 88
Moisture (% w/w)	7.2	6.6	Nil	6.3 – 8.8
Kin. Viscosity (mm ² s ⁻¹)	442.2	438.6	436.4	
Specific Gravity	0.9634	0.9633	0.9633	0.957– 0.968
Refractive Index	1.474	1.4762	1.4761	1.476-1.479
% Oil Yield	46.5	48	48	46-55
Colour	Amber	Amber	Amber	amber
% FFA Content	19	0.75	-	0.3 – 1.0

The table above shows that refining the oil has significant effect in reducing FFA content of the castor oil. The physicochemical properties such as viscosity, saponification value, specific gravity and iodine value of the crude and refined oils are nearly the same. The iodine value is a measure of unsaturation in oils, therefore the low iodine value of refined and crude castor oil shows that they have low level of unsaturation and are unsuitable for paint formulations or making of vanishes (Kyari, 2008).

Comparing the physicochemical properties of refined and dehydrated castor oil, it was clearly seen that the process of dehydration had little or no effect on properties of the oil such as density, refractive index, colour, viscosity but had significant effect on properties such as saponification value, acid value, pH value and especially iodine value. Dehydrated castor oil has low saponification value; indicating it is not suitable for making soaps and lubricants. The high iodine value of dehydrated castor oil simply indicates that its level of unsaturation increased during dehydration, thus making it suitable for alkyd resin modification.

3.2 Characterization of Crude and Refined Soybean Oils:

The physicochemical properties of crude and refined soybean oil were determined according to ASTM standards and the results presented in Table 2

Table 2: Results on Characterization of Crude and Refined Soybean Oil Samples

Properties	Crude Soybean Oil	Refined Soybean Oil
Acid Value (mgKOH/g)	1.01	0.341
Saponification (mgKOH/g)	195	189
Iodine Value (gI ₂ /100g)	127.9	129
Moisture (% w/w)	0.8	0.8
Kin. Viscosity (mm ² s ⁻¹)	49.2	46.01
Specific Gravity	0.924	0.923
Refractive Index	1.469	1.472
% Oil Yield	47.5	49
Colour	dark yellow	faint yellow
% FFA Content	16.0	0.58

Just like the case of castor oil, refining crude soybean affected mostly properties like acid value, FFA content. The level of unsaturation in soybean oil is high as indicated by its high iodine content. Thus soybean oil is naturally suitable for the modification of alkyd resin for use in paint and varnish industry as noted earlier by Kyenge et al., 2012.

3.3 Characterization of Alkyd Resins Produced:

The physicochemical properties of alkyd resin produced from castor and soybean oils were determined and the results presented below:

Table 3: Physicochemical properties of Castor Oil Modified and Soybean Oil Modified Resins

Properties	Castor Oil Modified Resin	Soybean Oil Modified Resin
Acid Value	8.60	5.30
Saponification Value	636.42	638.41
Peroxide Value	Nil	Nil
Iodine Value	24.65	28.46
Specific Gravity	0.9728	0.9822
Refractive Index	1.6779	1.6767

The physicochemical properties above were determined at the same reaction conditions. The properties are within the range specified by ASTM standards for quality alkyd resins. However soybean oil modified resin has lower acid value than castor oil modified resin at the same reaction conditions. Thus the rate of decrease in acid value is faster in soybean oil modified resin than castor oil modified resin. In other words, at the same reaction time, temperature and dosage of phthalic anhydride, soybean oil yields alkyd resin faster than castor oil.

3.4: COATING PROPERTIES OF THE ALKYD RESIN:

The results from the coating properties determined are presented in Tables 4-6

Table 4: Film Properties of Oil Modified Alkyd Resin (% Removal) at 240° C

Coating Properties	Castor Oil Modified Resin	Soybean Oil Modified Resin
Solidification Time(hrs)	15	14
Set to Touch(hrs)	1 ¹ / ₂	1
Drying Time(hrs)	11 ¹ / ₂	10

Table 5: Solubility of Alkyd in Different Solvent Media after 1hr

Mediums	Soybean oil modified alkyd film	Castor oil modified alkyd film
Ethanol	Sparingly soluble	Sparingly soluble
Xylene	soluble	soluble
Methanol	soluble	soluble
N-Hexane	Partially soluble	Partially soluble

Table 6: Chemical Resistance of Alkyd

Mediums	Soybean oil alkyd film	Castor oil alkyd film
Distilled Water	Excellent (film not removed)	Excellent (film not removed)
Acid (0.1M H ₂ SO ₄)	Excellent (film not removed)	Excellent (film not removed)
Alkali (0.1M KOH)	Good (film removed)	Good (film removed)
Brine (5%w/w NaCl)	Excellent (film not removed)	Excellent (film not removed)

The chemical resistance of the alkyd resin film in different media shows that both alkyd resins produced from castor and soybean oil are unaffected by acid, brine and water. However, they are susceptible to alkali mediums, that is, they show poor resistance to alkali. This poor resistance to alkali medium is mainly due to the presence of alkali hydrolysable ester groups in the alkyd resins (Ikhuria et al., 2011)

Generally, the chemical resistances of the alkyd resins as well as their solubilities in different organic solvents are found to be independent of the reaction processing time, reaction temperature, as well as the dosage of phthalic anhydride used. However, the rate at which the alkyd resin dissolves in the solvent medium depends on the nature of the medium and can be increased by vigorous shaking, and by allowing the alkyd to stay longer in the solvent.

Drying time and solidification time are other important properties of alkyd resins, which are very critical to their application as binders in paint. Drying time determines the ability of the alkyd to dry hard and form durable films through the process of autoxidation. It is related to the amount of double bond present in the oil as measured by the iodine value (Muizebelt et al., 1998). Thus, table 5 showed that soybean oil modified alkyd resin has relatively lower drying time, set to touch and solidification time than castor oil modified alkyd resin, because of the higher number of double bonds in the fatty acid methyl esters present. However, the drying time, solidification time and set to touch, are relatively affected by changes in temperature.

3.5: FTIR SPECTRA ANALYSIS:

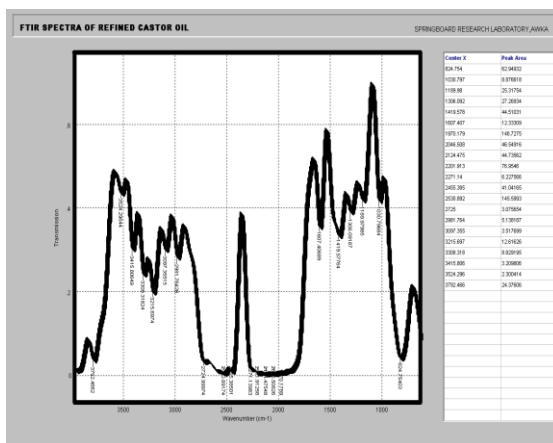


Fig 1: FTIR Spectra of Refined Castor Oil

The FTIR spectra of the refined castor oil shows the broad band shoulder around 3097.355cm⁻¹, 3308.318cm⁻¹, 3415.806cm⁻¹, 3524.296cm⁻¹, 3792.466cm⁻¹ OH stretch of phenolic compounds of alcohol. The medium band around 2046.508cm⁻¹, 2124.475cm⁻¹, 2201.913cm⁻¹ and 2271.140cm⁻¹ was attributed to C=O vibration of carboxylic and carbonyl compounds. The absorption band around 2455.385cm⁻¹ was due to CN stretch of unsaturated nitrile compound, while the peak values at 1607.407cm⁻¹ and 3215.697 cm⁻¹ correspond to the N-H of amine compounds (Gan and Tan, 2001).

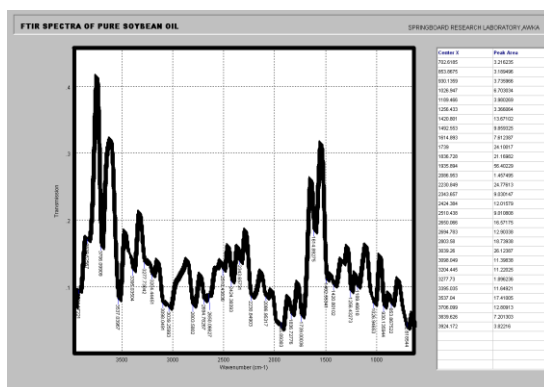


Fig 2: FTIR spectra of Refined Soybean Oil

The FTIR spectra of the soybean oil shows the broad band shoulder around 3039.260cm⁻¹, 3098.049cm⁻¹, 3395.035 cm⁻¹, 3537.040 cm⁻¹, 3839.626 cm⁻¹ and 3924.172cm⁻¹ OH stretch of 1^o, 2^o, and 3^o alcohol respectively. The medium bands around 1259.433cm⁻¹, 1614.893 cm⁻¹, 3204.44cm⁻¹ and 3706.099cm⁻¹ are due to NH stretch of 1^o, 2^o and 3^o amine compounds. The absorbance around 2086.953 cm⁻¹, 2230.849 cm⁻¹, 2343.384 cm⁻¹ are thus assigned to CO anti-symmetric stretch of carboxylic and carbonyl compounds, while the peak value at 2424.384 cm⁻¹, 1970.179cm⁻¹ and 2981.764 cm⁻¹ corresponds to C≡N and SCN anti-symmetric stretch of nitriles and thiocyanate compounds respectively. The absorption band at 1836.728cm⁻¹ was due to cyclic compound of methyl ester unsaturated fatty acid, (Uzoh, 2013).

FTIR Spectra of Alkyd Resin Produced from Castor and Soybean Oils

This study showed the functional groups present in alkyd resin produced from castor and soybean oils respectively.

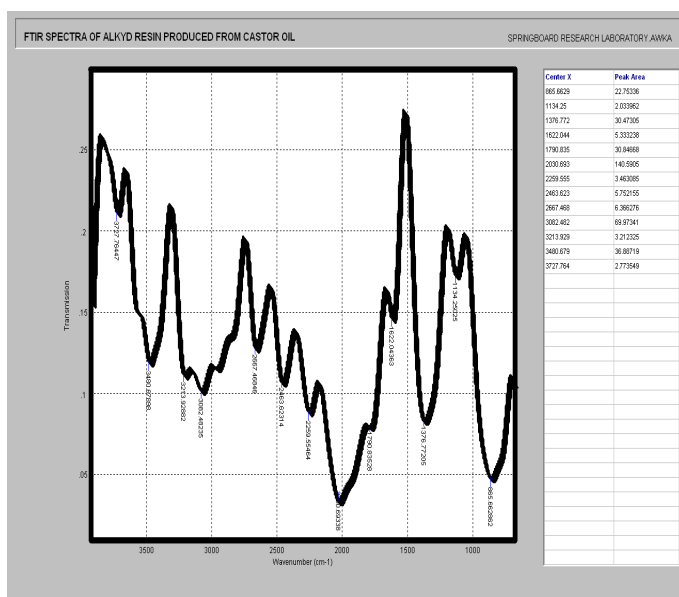


Fig 3: FTIR of Castor Oil Modified Alkyd Resin

Figure 3 shows the spectra and functional compounds in castor oil modified alkyd resin. The broad bands around 3082.482⁻¹, 3480.679⁻¹ and 3727.764cm⁻¹ were assigned to hydroxyl (OH) group of unsaturated fatty acid in castor oil modified alkyd resin. The peak value at 1790.835cm⁻¹ corresponds to cyclic ester of saturated fatty acid. Peaks due to free carboxyl and carbonyl compounds were observed around 2030.693cm⁻¹, 2259.555 cm⁻¹ and 2667.468cm⁻¹ respectively (Bellamy, 2005). The medium band around 2463.623cm⁻¹ was assigned to CN anti-symmetric stretch of nitrile compound.

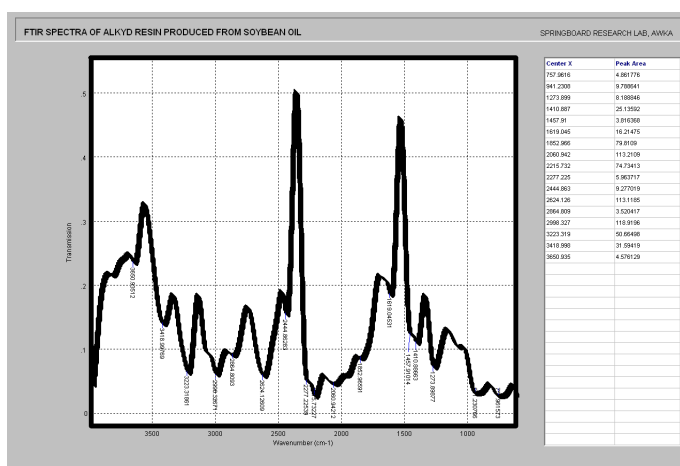


Fig 4: FTIR of Soybean Oil Modified Alkyd Resin

The FTIR spectra of the mixture shows the broad band shoulder around 3418.998cm^{-1} and 3650.935cm^{-1} , corresponding to 2° and 3° alcoholic (OH) hydroxyl compound of the unsaturated fatty acid in the produced resin (Uzoh, 2013). The carboxyl and carbonyl (C=O) are found at 2060.942cm^{-1} , 2215.732cm^{-1} and 2624.126cm^{-1} respectively. Alkyne compound of nitriles (C≡N) and thiocyanate (SCN) are attributed to high saturated compounds found around 2444.863cm^{-1} and 2998.327cm^{-1} respectively. The absorption band around 1273.899cm^{-1} , 1619.045cm^{-1} and 3223.319cm^{-1} are due to NH stretch of 1° and 2° amine compounds. The aromatic ring ester at medium band shoulder of 1852.966cm^{-1} helps to confirm the esterification reaction of the alkyd resin (Grasselli et al., 2001).

4. CONCLUSIONS

Both dehydrated castor and soybean seed oils have excellent properties to be considered as suitable drying oils for the synthesis of quality alkyds. Both castor oil modified and soybean oil modified resins exhibited excellent coating properties such as their solubilities in different solvent media, drying time, set to touch and resistance to chemical medium. However, they show poor resistance to alkali medium. At the same esterification reaction condition, the acid value of soybean oil modified resin was lower than castor oil modified resin. This simply indicated that the rate of decrease in acid value of alkyd resin synthesized from soybean oil is faster; implying that soybean oil has desirable drying qualities for surface coatings than castor oil due to its high iodine content and presence of more unsaturated fatty acid compounds, as shown by the FTIR analysis.

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