Extraction of Oil from Soyabean (*Glycine max*) and its Subsequent Use for the Preparation of Short Oil Alkyd Resin

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Abstract: The extraction of oil from Soyabean (*Glycine max*) seeds was carried out and subsequently used for the formulation of short oil alkyd resin. The resin used had dark brown in colour with a pungent choking smell. It was viscous and dragged on brush, below 20°C. It had a pH of 3.8 indicating that it was acidic. The results showed that the percentage oil length yield was 40.67 which were in the category of short alkyd resin, within the limits of experimental errors. The result obtained indicated an acid value of 9.93 mgKOH/g, a hydroxyl number of 50.60 and percentage solid content of 49.5 for the short oil alkyd resin. The formulated sort oil alkyd resin had a smooth surface with spots on dispersion and had fast drying time. The resin is a good adhesive and is recommended for use for storing enamels, wood finishes, decorative coatings and in paint industries in Nigeria and in international trade.

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1. Introduction

A resin can be defined as a thick sticky liquid produced chemically, used as a binder, it's essential function is film formation or to reinforce the oil films and give additional gloss to surfaces.

It has the ability to pass from a low viscosity liquid film to a more or less hard plastic one (Santos and Elliot, 2004). It also has the ability to bind particles of pigments together and retain them in the film.

The choice of binder plays the major part in deciding the properties of the film, though these properties may be modified to a considerable extent by the use of pigments. Among the more important film properties are adhesion, gloss and mechanical properties.

Any natural or synthetic organic compound consisting of a non-crystalline (amorphous) solid or viscous liquid substance or mixture is referred to as a resin. Natural resins are usually transparent or translucent, yellow to brown and can melt or burn. Most are exuded from trees especially coniferous plants; the fluid secretion material that can be worked.

Natural resins have been used in perfumes and medicines (e.g. balsams), in paints and varnishes. (E.g. turpentine and shellac the latter derived from the secretion of an insect), and in decorative ware (e.g. amber, oriental lacquer).

Synthetic resins are all plastics; the term resin; though still used in the modem industry, date the years when synthetics began to replace natural resins. Thermoplastic resins are plastics such as polyethylene that can be shaped repeatedly on heating.

Whereas, thermosetting resins are plastics such as epoxy that cannot be reshaped (Aigbodion and Okiemen, 2001). However, Oil modified resins are combinations of oil, and resins usually referred to as short, medium or long oil resins depending on the relative fraction of drying oil component in the resin, derived from natural sources.

Those of synthetic oils are oil modified resins. Resins are added to painting media primarily to offer a paint film that can be worked over within hours or, at maximum.

The next day because when the solvent evaporates, which happens within several hours, the resin hardens, holding the drying oil in places until it can oxidize and solidify, also to dilute paint for glazing without over thinning and dry with greater clarity than draying oils, providing brilliance to paint films.

Resins are also the primary material used for varnishing painting. Today, the five most commonly used resins are copal, dammar, mastic, Venice, turpentine and alkyd (Dulta *et al.*, 2004). The resin of this project interest is the alkyd resin.

1.1 Alkyd Resin

Alkyd resin is the category of resins that are made from mixtures of dibasic acids and polyhydric alcohols (Pearson, 1976). There are many alkyd resins and each manufacturer picks a favourite and keeps it a secret. The quality and concentration of an alkyd resin or medium can vary a great deal from manufacturer to manufacturer.

The selected resin is chemically combined with non-yellowing oil, such as safflower, coconut, soyabean oils, producing a workable, fast drying medium. Most manufacturers add driers to speed up the drying time and silica to give extra body.

Alkyd resin can exhibit a thixotropic effect. Thixotrophy is an unusual phenomenon where a gel or paste suddenly loses its plasticity when disturbed or moved mechanically, resulting in a liquid (Pearson, 1976).

The opposite may also occur where a liquid, left undisturbed, forms into a gel. This effect can be used to advantage during glazing by helping to prevent the glaze from spreading uncontrollably over the printed surface.

However, this characteristic is undesirable with impasto techniques. Alkyd resins should not be over thinned, so add no more 25% thinner to resin. The alkyds comprise one of the most versatile groups of synthetic resins.

Alkyd coatings are typically sold in three classes; long, medium and short. These terms represent the relative fraction of drying oil alkyds have a high percentage of drying oil (75% and above) content and are generally sold as medium duty coatings for the consumer market.

Medium oil alkyds have less drying oil and have a higher percentage of large molecular height polyester backbone. They dry slower and are employed as high gloss coatings and wood finishes (50-65%).

Short oil alkyds have a very low percentage of drying oil (40 – 45%) content in relation to the base polyester polymer or backbone chain. These coatings will not air dry or harden unless heated. Short oil alkyds are employed as baking enamels for finished metal products.

The term alkyd resin was invented by Kience in 1927 to describe the “complexes resulting from the reaction of polyhydric alcohols and resinfying carboxylic organic acids” (Nwanorh, 2004) such as polybasic acids and their anhydride.

The word is a combination of the first two letters of alcohol and of last three acids with charged spelling naming alkyl. They are class of polyester coatings derived from the reaction of an alcohol and an acid or acid anhydride, hence, the term alkyd from “alcohol and acid anhydride and are dominant resin or binder in most oil based coatings sold to the consumer market (Irving, 1962).

It is valued for its chemical constituents and uses. Fossilized resins are the source of amber. The term is also used for synthetic substances of similar properties. Resins have a very long history and are mentioned by both ancient Greek Theophrastus and Roman Pling, especially as the formerly known frankincense and myrrh.

They were highly prized substances used for many purposes especially perfuming and as incense in religious rites. Resins are viscous liquid, composed mainly of volatile fluid, terpenes with lesser components of dissolved non-volatile solids which make resin thick and sticky.

The most common terpenes in resin are the bicyclic terpenes alpha-pinene, beta-pinene, the monocyclic terpenes limonene and terpinslens, and smaller amounts of the tricyclic delta-cadiene. Some resins also contain a high proportion of resin acids. The individual components of resin can be separated by fractional distillation (Iheonu, 1999).

Smith, in 1901, prepared the first phthalic anhydride resin, but it was not until 1912 that a concerted effort was made to prepare useful alkyd resins that might have commercial value. World War 1 however served to assure alkyd resins a permanent and important place in the protective-coating field.

The cheap production of phthalic anhydride by the Gibbs process of oxidizing naphthalene, the development of the low viscosity cellulose nitrate solution and the manufacture of many new industrial solvents, all contributed to the initial success of these resins. The full potentialities of alkyd resins were not realized, however, until 1927 when interest development in them as baking and air-drying finishes.

Kience at this time filed a patent, which was granted, covering the preparation of drying oil-fatty acid modification. Only the broad claims of this patent were later declared invalid.

These resin of Kience’s were truly varnishes in themselves, they dried to harden coatings in much shorter period of time than comparable oleoresins varnishes and the xilma were superior to those of the natural resin vehicles used therefore.

The sudden increase in production of alkyd resins that followed has steadily continued and the peak in production has probably accounted for more than three-fourths of all of the resins used in the manufacture of protective coatings. This was despite a large demand, for other purposes for the raw materials used in the manufacture of alkyds.

2. Materials and Methods

2.1 Sample Collection and Preliminary Preparation

Soya-beans used for the extraction of the vegetable oil used for short oil resin formation were bought from Ekeukwu market, Douglas Road Owerri, Imo State of Nigeria. A large quantity of the soya beans was carefully examined to remove stones, sand and the hulls.

The seed coats were removed and the seeds sun–dried and ground into fine powder to increase the surface area prior to solvent extraction. The powder was placed in a polyethylene bag which was then tied, and kept for the extraction of the soya bean oil.

2.2 Extraction of Soya Bean Oil Using Solvent Extraction

Soya bean oil was extracted from the soya bean powder by means of solvent extraction using a soxhlet extractor. 50.00 g of the soya bean powder were weighed and placed inside a thimble, loaded into the main chamber of the soxhlet extractor.

The soxhlet extractor was placed onto a flask containing 200 cm^3 of petroleum ether. The soxhlet was equipped with a condenser. The solvent was heated to reflux and the distillate as it dropped from the condenser, collected in the chamber.

By coming in contact with the ground soya bean seed in the thimble, the liquid effected the extraction. As the chamber filled to the level of the upper reach of the siphon arm, the yellow solution emptied from the chamber into the boiling flask by a refluxing action.

The cycle of vaporization, condensation, extraction and reflux actions continued until there was no evidence of oil extract from the sample in which the resulting solution became clear. Thereafter, the petroleum ether was recovered and the oil obtained. The obtained oil in the round bottom flask was allowed to cool and re-weighed.

The oil yield was calculated and expressed as percentage oil yield in terms of oil length with the relationship. Table 1 show the formation of the short oil resin (stage process etherification or alcoholysis).

Table 1: Recipes for the Preparation of the Short Alkyd Resin

Raw Materials	Weight / g	Equivalent % Weight
Soya bean oil	88	44
Lithium hydroxide	0.1	0.05
Glycerol	40	20
Phthalic	66	33
Fumaric acid	4	2
Cobalt	1.8	0.9
Total	199.9	99.95

About 88.00 g of the soya bean oil were transferred into a preparation vessel and 30.00 g of glycerol were mixed into the vessel containing the oil. The mixture was heated to form the corresponding mixed partial esters. The mixture was heated to a temperature of 220°C – 230°C under an inert gas blanket with agitation.

At the temperature of alcoholysis 230°C, then 0.10 g of lithium hydroxide was added to the mixture. The reaction (heating) was continued until a sample of the reaction mixture became soluble in 4cm^3 of methanol, hence the alcoholysis stage stopped.

The remaining ingredients; 10.00 g of glycerol, 66.00 g of phthalic anhydride, 4.00 g of fumaric, and 1.80 g of cobalt crystal were added and etherification was carried out by heating until a high viscous liquid was formed.

Then the reaction was stopped by placing the reaction vessel on ice. Finally, the mixture was filtered and transferred to a clean container, stoppered and subjected to further analysis and characterization.

2.3.1 Determination of Acid Value

About 10.00 g of the short oil resin sample were weighed into a 250cm^3 flask. 10 cm^3 of 95% (v/v) ethanol and 3 drops of phenolphthalein indicator were added and titrated against 0.10 $moldm^{-3}$ aqueous solution of KOH, until a pink colour appeared which persisted for 15 seconds.

The titre value was noted and the titration repeated for two more times. The average titre value was used for the calculation of the acid value of the oil as presented thus:

$$\text{Acid value (A.V)} = \frac{\text{TV xCx5.61}}{\text{Weight of sample}}$$

Where,

Weight of sample = 10.00 g

Titre value (T.V) = average volume of KOH used for titration

C = Molar concentration of KOH solution = 0.10 $moldm^{-3}$

2.4 Determination of the oil length yield

After the extraction of the oil, the round bottom flask containing the oil obtained was re-weighed. The quantity of oil obtained was calculated using the expression:

$$\text{Quantity of oil x 100} = \frac{W_2\text{-}W_1\text{ x100}}{\text{Weight of sample}}$$

Where,

W_1 = Weight of empty round bottom flask.

W_2 = Weight of flask + oil.

2.5 Determination of Hydroxyl Number

The hydroxyl number was determined by addition of the oil length yield and the acid value. Oil length Yield + Acid Value = Hydroxyl Number

2.6 Determination pH

The pH of the short oil resin was determined by the use of a pH meter. The short oil resin sample was placed in a cleaned beaker and the electrode was inserted into the beaker after standardization with distilled water. The reading at 25 °C was taken and then recorded in one hour's time.

2.7 Drying Test

The short oil resin was applied on a treated clean surface and allowed to dry. The time taken to completely stick onto the surface of a substrate was recorded per day. It was repeated for five days and the average time evaluated. Temperatures were noted.

2.8 Solid Content (Non- Volatile Component) Determination

About 200.00 g of the short oil resin were weighed and placed in a pre-weighed crucible and heated until the water content and any other volatile components were completely vaporized leaving behind the solid. The crucible with its contents was allowed to cool and re-weighed. Percentage solid content (Non- volatile component) = $\frac{W_1 - W_2}{W} \times 100$

Where,

W_1 = Weight of crucible + resin, before heating

W_2 = Weight of crucible + resin, after heating and cooling.

W = Weight of resin before heating.

2.9 Physical Properties Analyses

Colour test: the colour of the short oil resin was examined visually. odour test: About 2.00 g of the sample was placed on the tip of a smelling blotter and the odour perceived.

Dispersion test: a sample of the short oil resin was brushed on opacity paper and the degree of dispersion was assessed visually.

3. Results

The results of the various tests carried out are given in Table 2.

Table 2: Analytical Characterization

Parameter	**Results**
Appearance	Viscous
Colour	Dark brown
Odour	Choking pungent smell
Dispersion	Smooth surface with spots
Drying time	Fast drying time, this (W) 50°C
P^H	3.8, turns blue litmus red
Solid content	49.5%
Acid value	9.95 mgKOH/g
Hydroxyl number	50.60
Percentage oil length yield	40.67

3.1 Discussion

The chemical reactions involved in the formation of the short alkyd resins were alcoholysis and polymerization.

Alcoholysis stage: Conversion of triglyceride to monoglyceride.

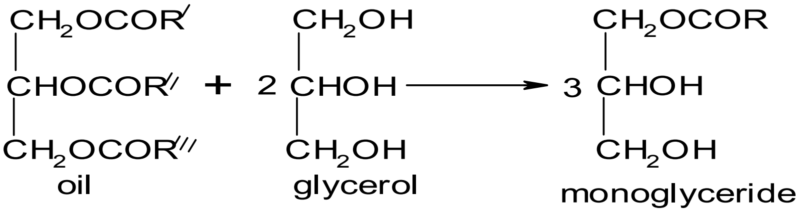

Fig. 1: Polymerization stage

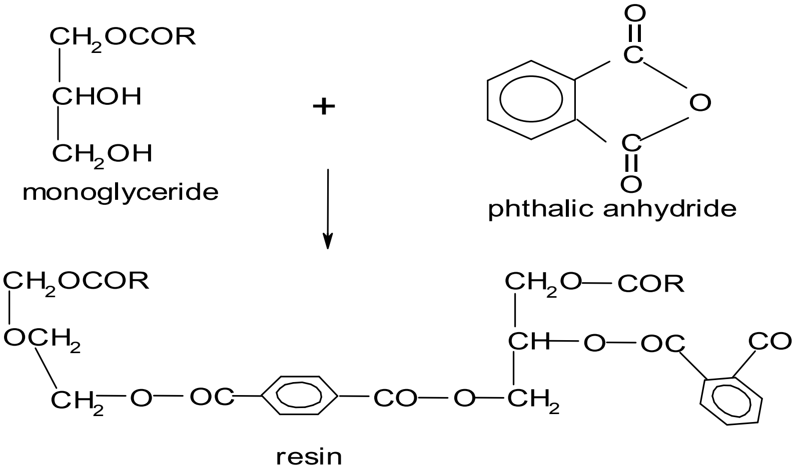

Fig. 2: Structural formula of resin

On physical observation of the resin product, it was viscous with dark brown colouration and a choking pungent small. The resin drays on brush on application unto a surface below 200 °C and turned blue litmus paper red, which confirms with literature.

The experimental result obtained of the acid value, 9.93, indicates that the resin is acidic and contains a good hydroxyl groups for acetylation indicated by the hydroxyl number, 50.60.

The solid content 49.5% conforms to the set standard of 50% for alkyd resins. The resin has a good consistency in fluidity and showed a smooth surface with spots on dispersion.

The oil alkyd resin is one of the most versatile groups of synthetic resin, having good adhesive properties, such as fast drying time, glossy and good adhesion to surface applied.

4. Conclusion

Short oil alkyd resin can be produced using Soya bean oil and other organic materials e.g glycerol, polyols etc. The resin obtained is a good adhesive, having fast drying time adhesion to surface, glossy and has acceptance percentage solid content and acid value.

4.1 Recommendations

The result indicated therefore, that, it can find applications in lacquers, paints, amino acid curing. Alkyd paints are also suitable for storing enamels and wood finishes. Thus, soya bean oil can be utilized in the alkyd resin formation.

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