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SYNTHESIS, CHARACTERISATION AND ANTIMICROBIAL EVALUATION OF THE ALKYD RESINS DERIVED FROM CASTOR SEED OIL

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ABSTRACT
Quantity of castor seed oil (CSO) obtained from local market was determined based on acid value, percentage free fatty acid, iodine value, refractive index, saponification number, specific gravity and moisture content. The CSO was employed in the preparation of alkyd resin (oil modified ester) using monoglyceride method. This involved the reaction of the oil with glycerol (alcoholysis) and subsequent reaction of the monoglycride obtained with phthalic anhydride to form the alkyd resin. The CSO and its corresponding alkyd resin were characterized using FTIR. The antimicrobial activity of the alkyd resin was evaluated using paper disc diffusion method. The result showed that prepared alkyd resin is active is active against some selected bacterial srains at concentrations 50mg/ml and 100mg/ml on E. coli, Sallmonella and Staphylococcus
Keywords: Alkyd resin, Castor Seed Oil, Phthalic anhydride

INTRODUCTION

Castor Seed Oil (CSO)

Castor bean is cultivated for the seeds which yield viscous, pale-yellow, non volatile and non-drying oil. It has been used for industrial and medicinal purposes (Stubiger *et al.*, 2003). Its botanical name is *Recinus communis L* of the family Eurphobiace as a plant indigenous to many part of the world (Salimon *et al.*, 2010). It has been reported that its modification by severe dehydration resulted in an oil with excellent drying properties for use in the production of alkyd resin (Onukwli *et al.*, 2008).

Alkyds are oil modified polyesters consisting of a polyol (usually glycerol, trimethalol propane or pentaerythritol), a multi functional acid (phthalic acid or trimellitic acid) and an unsaturated fatty acid formed by poly-condensation reaction. They are classified as short (30 – 42%), medium (43 – 54%), long (55 – 68%) and very long (≥68%), based on the percent weight fraction of vegetable oil in the resin (Manawwer *et al.*, 2014).

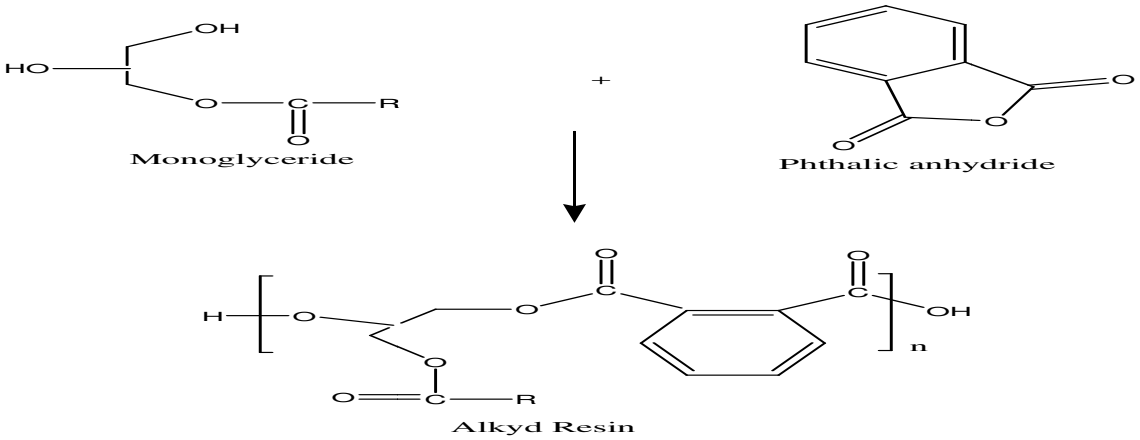

Fig 1:Chemical transformation of vegetable oil to alkyd

MATERIALS AND METHODS

Castor seed oil was purchased from Kasuwar Kurmi market in Kano city, Kano Nigeria. All chemicals and solvents used were of analytical grade and were used without further purification. The glass wares used were washed with detergent, rinsed with distilled water and dried before used. All weighings were carried out on an electric Mettler balance model H30AR, iodine value, saponification number, acid value, percentage free fatty acid, specific gravity were carried out using burrette,

standard laboratory apparatus. FTIR spectral analysis of the castor seed oils and alkyd resin samples were recorded in the range of 4000 – 650 cm^{-1} using CAREY630 machine, Antimicrobial activity studies were carried using petri dishes in an incubator.

Physico-Chemical Characterization of Castor Seed Oil

The physico-chemical properties of the castor seed oil sample including: refractive index, iodine value, saponfication number, acid value, percentage free fatty acid and specific gravity analysis were determined in accordance with the American Oil Chemists Society (AOCS),

Synthesis of the Castor Seed Oil Alkyd Resin

The measured oil sample (castor seed oil) was poured into the three necked flask and heated to about 120^{o}C to expel moisture. A glycerol was added and the temperature was to 230^{o}C. After 30 minutes an aliquot of the reaction mixture was checked for solubility in methanol, a test for the formation of monoglyceride. The temperature was then lowered to about 180^{o}C, followed by addition of phthalic anhydride was added, and subsequent addition of about 120ml xylene (120ml) into the reaction mixture to aid the removal of water of esterification by forming an azeotrope. The temperature was gradually increased between 240 – 250^{o}C and maintained for about 4 hours. Aliquots were taken from the reaction mixture at intervals of one hour to check for drop in acid value. The reaction was quenched by immersing the reaction vessel in cold water. Nitrogen was used to blanket the surface of the reaction mixture during the reaction (Onukwli *et al*., 2008).

Physico-chemical characterisation of the alkyd resin

The physicochemical properties of the alkyd resins prepared from the castor seed oils; such as colour, viscosity, acid value and FTIR spectral analysis were determined using American oil chemists society methods (AOCS, 1996).

Antimicrobial Activity of Alkyd Resin

The antimicrobial activity was carried out by employing agar disc diffusion method (Afroditi, 1996), the suspension of the test bacteria which were spread in the nutrient agar plates were incubated with test organisms for 24 hours at 37 ^{0}C (*E. coli, Salmonella, Staphlococcus*). One disc from each sample was placed in the petri-dishes with sterile forceps (Karigar *et al*., 2010). Clear inhibition zones around the disc indicating the presence of antimicrobial activity was recorded.

RESULTS AND DISCUSSION

The Physico-Chemical Characterization of the Castor Seed Oils

Table 2 showed the results for the physico chemical characterizations of the castor seed

The results of the physical and chemical properties of the oils are shown in Table 2. The iodine value is 87.613 for CSO, which is less than 100, hence, the CSO is of lower degree of unsaturation (Ayo *et al.,* 2007). The result shows that the castor seed oil had saponification value of 169.34 which is found to be slightly lower than the range reported by Ogunniyi (2006) i.e 177 – 182 mg/g. The acid value of the castor oil was found to be 12.04. Acid value is the measure of the extent to which the constituents' glycerides have been decomposed by lipase action, and has shown to be a general indication of the edibility of the oils (AOAC, 1980; Pearson, 1981). However, the refractive index of the oil is found to be 1.4604, the values is within the range of edible oils (Rossell, 1991). The specific gravity for the oil are: 0.9080, which implies that castor seed oil is less dense than water (Momodu *et al*, 2011). The percentage free fatty acid of the castor seed oil as shown in Table 2 is 7.023%. Also, the percentage moisture content of the oil is found to be high up to 9%. This is probably due to impurities contained that could cause hydrolysis of the ester linkages, thereby increasing the free fatty acid level (Nkafamiya *et al;* 2010).

Table 1: Formulations of the different alkyd resins

Raw materials	Oil (g)	Glycerol (g)	Phthalic acid	Oil length (%)
Compositions	94.04	62.00	148.12	31

Table 2: The Physico-Chemical Characterization of the CSO

Acid value	FFA (%)	Iodine value	Refractive index(25^{0}C)	Saponification no. (mg/g)	Specific gravity	%moisture
12.034	7.023	87.613	1.4604	169.34	0.9080	9.00

The Physico-Chemical Characterisation of the Alkyd Resins

Table 3 shows the physico-chemical properties of the alkyd resin prepared. The colour of the alkyd is dark brown compared to its corresponding precursor oil.

The darkening in the colour of the alkyds could be attributed to the high temperature of the reaction, oxidation and the catalyst (MacDonald, *et al.*, 1994). The acid value and viscosity of the alkyd is lower compared to the commercial standards C-AKD and L-AKD (Shaker *et al.*, 2012).

Table 3: The Physico-Chemical Properties of Alkyd Resins

Properties	CSO-resin	C-AKD	L-AKD
Colour	Dark Brown	Brown	Brown
Acid value(mg/g)	11.00	25.80	35.60
Viscosity (cp)	3.84	10.00	12.80

Antimicrobial Evaluation of the CSO Alkyd Resin

From Figure 1 the antimicrobial activity test for the castor seed oil modified alkyd resin (CSO Resin) determined showed that the resin was active against all the tested organisms at all concentration.

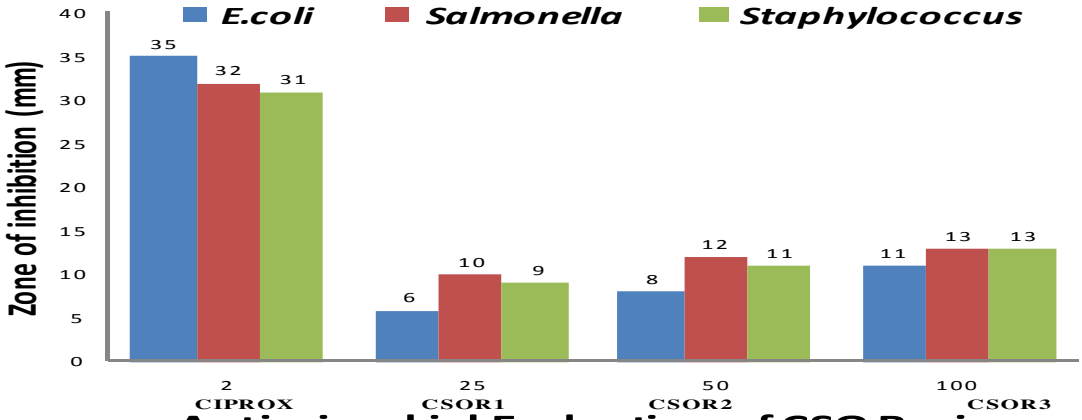

Fig. 3.1: **Antimicrobial Evaluation of CSO Resin**

Ciprox = ciproflaxin with conc. 2mg/ml, CSOR1 = CSO resin with conc. 25mg/ml CSOR2 = CSO resin with conc.50mg/ml, CSOR3 = CSO resin with conc.100mg/ml

FTIR Absorption analysis

The FTIR analysis for alkyd resins derived from castor seed oil is shown on Table 4.

Table 4 and Fig. 2 show FTIR spectra and peaks of interest for castor seed oil alkyd resin (CSO-Resin). From the table, it can be observed that the OH group from the glycerol (Fig.3.1) disappears in the spectrum of the CSO-Resin (Fig. 2). While, the appearance of the absorption band at 1601cm^{-1} indicates the presence of C = C stretching for an aromatics from the phthalic anhydride. Other band of interest that was found in the spectrum is the 1728 cm^{1} which corresponds to C=O stretching for an ester.

Table 4: FTIR Absorption Frequencies of Castor Seed Oil Alkyd Resin (CSO Resin)

Bands (cm^{-1})	FTIR Peak origin
1723	C = O Stretching due to conjugation with aromatics
1728	C = O Stretching for an ester
1601	C = C Stretching for aromatics

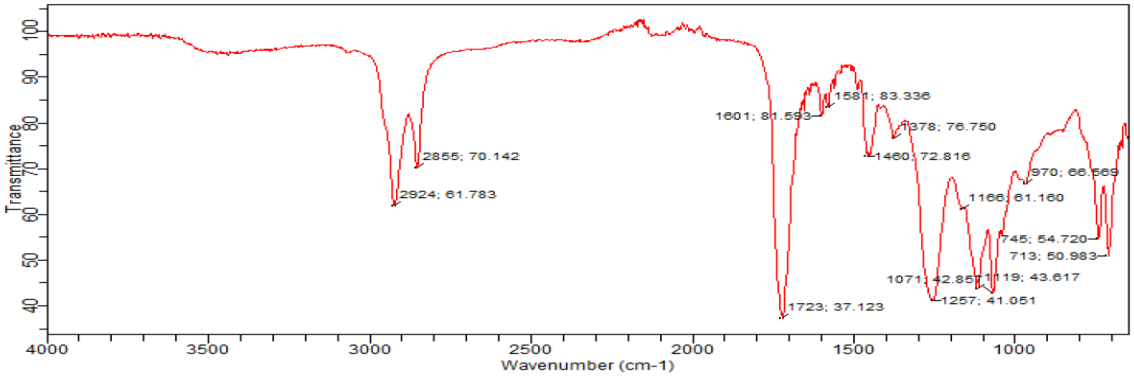

Fig 2: FTIR of castor seed oil alkyd resin (CSO resin)

CONCLUSION

The results obtained from this study show that the castor seed oil has been successfully converted to alkyd resin. The ability of this alkyd resin to show antimicrobial property, suggests that it has some potential for health care applications.

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