

# PREPARATION AND EVALUATION OF ALKYD RESIN FROM CASTOR OIL

D S Ogunniyi\* and G N Njikang

Department of Chemistry, University of Ilorin, Ilorin, Nigeria

(Received 18 June 1998; accepted 3 February 2000)

Alkyd resin is known for its good drying performances and good resistance to weathering and has almost replaced drying oils as binders for gloss finishes, varnishes, wood primers, enamels and lacquers.

Castor oil is used in many chemical and polymer industries (Ehrlich *et al* 1959; Yeadon 1959; Finar 1973), Varying the amount of castor oil could produce changes in some physical properties of the polymers (Qipeng *et al* 1990; Ogunniyi *et al* 1996). Here the production of castor oil-based alkyd resins and their drying properties are discussed.

**Material.** Castor seeds were separated from impurities, sun-dried for one week and subsequently oven-dried at 100°C for 24h. Thereafter, castor oil was obtained by extracting the crushed nuts with light petroleum either at a temperature of 60°-80°C in a Soxhlet extractor followed by distillation.

Castor oil was dehydrated by heating the oil at 260°C for one hour under partial vacuum and in the presence of few drops of concentrated sulphuric acid (Shreve 1967) as a catalyst in an inert atmosphere of nitrogen. The extracted and dehydrated oils were tested for iodine value, acid value and water content by standard methods (AOAC 1980; Weiss 1983; ASTM 1985b).

**Preparation of alkyd resins.** Alkyd resin was prepared in a two-necked reacting vessel under nitrogen atmosphere. Four different classes of alkyd resins were prepared with different amounts of dehydrated oil (Table 1). A known weight of castor oil was heated to 240°C after which, glycerol, part of the xylol and calcium carbonate (catalyst) were added. Samples of the reaction mixture were then taken every five minutes and mixed in a 1:1 volume ratio with anhydrous methanol. Alcoholysis was said to be completed when the mixture gave a clear solution. At this juncture, phthalic anhydride and more xylol were added. This phase of polyesterification was monitored by periodic determination of the acid value of the mixture. The reaction was quenched when the acid value had fallen to 10.

**Analysis of prepared alkyd resins.** The prepared alkyds and commercial sample used as standard were analysed

\*Author for correspondence

for acid value, solubility, solidification time and thickness. Solubility was determined by dissolving 3g of each of the resin in 5ml of turpentine, benzene and toluene and allowing the solution to stand for 8h with intermittent stirring. A micrometer screw gauge was used to measure the thickness of the films dried on chromatographic plate. The hot-plate gel time method (D'Alelio 1946) was used to determine solidification time.

**Drying performance of alkyd resins.** The touch method (ASTM 1985a) was used to determine the drying performance of the alkyd resins and the formulated paints (Turner 1988). The paint formulation used, on a percentage basis, is as follows: Titanium dioxide (12.93%); Talc powder (21.94%); Turpentine (15.03%); Cobalt naphthanate (0.5%); Alkyd resin MAKD-2 (49.6%).

**Extraction and dehydration of castor oil.** After extraction, a percentage oil yield of 41.67% was obtained. 87.5% of the extracted oil was recovered after dehydration. The untreated and dehydrated castor oil had acid values of 3.27 and 6.14 and iodine values of 85.65 to 132.93 respectively.

**Preparation of alkyd resins.** The results of production of alkyd resins of the monitoring of alcoholysis and polyesterification for all the oils produced are shown in Fig 1-2. It is worth mentioning that only monoglycerides are soluble in anhydrous methanol.

The approximate time required for alcoholysis for any class of alkyd using dehydrated castor oil can be calculated from the equation below (Fig 1).

$$T = 40.9P + 1080$$

where T is the time for alcoholysis in seconds and P is the percentage oil length.

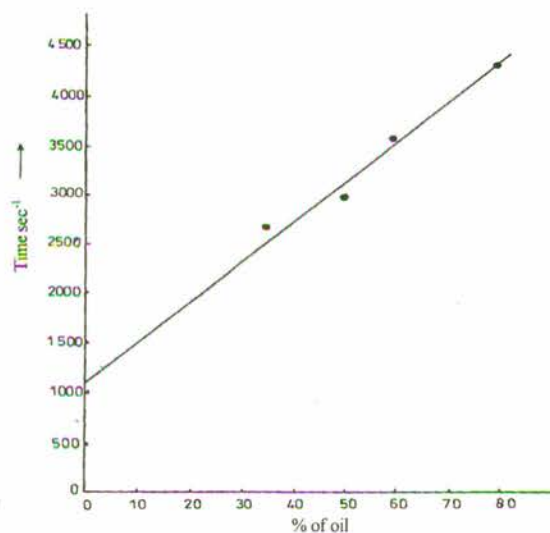


Fig 1. Graph of oil length versus time.

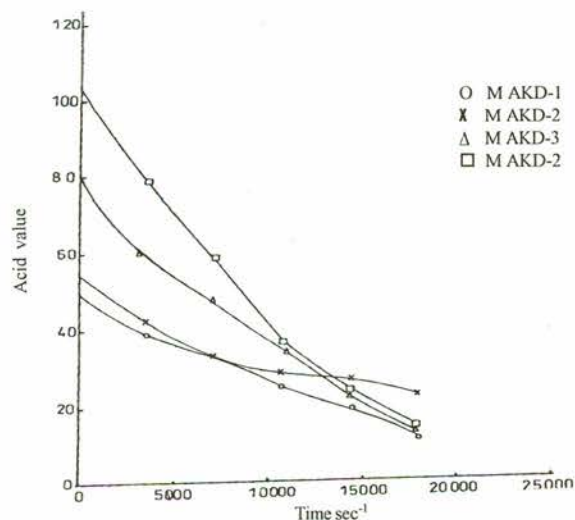


Fig 2. Acid value change with time during polyesterification.

Figure 2 shows that the greater the oil length of the reaction mixture, the smaller the initial acid value. This might be due to the high amounts of di- and triglycerides present in the reaction mixture of long oil alkyds, thereby diluting the mixture, and reducing the concentration of the stronger acid, phthalic anhydride.

For RM-AKD (short oil alkyd resin with untreated castor oil), polyesterification was stopped 21 minutes after the addition of phthalic anhydride because of the formation of a foam-like substance that filled up the reaction vessel, which may be due to the extra hydroxyl functionality brought about by the -OH of the ricinoleic acid present in castor oil; the insolubility of the unknown product in common solvents suggests crosslinking.

**Analysis of alkyd resins.** All the alkyd resins produced had good coloration, with the colour intensity decreasing with increasing length of the resins (Table 2). The thickness of the films formed by these alkyds varied from 0.02mm to 0.06mm, with the short oil alkyd (35% oil) having the greatest thickness.

**Table 1**  
Recipes used for the alkyd synthesis

Ingredient	%Weight Based on Total Content				
	M-AKD-1 80% Oil	M-AKD-2 60% Oil	M-AKD-3 50% Oil	M-AKD-4 35% Oil	MR-AKD 35% Oil
Dehydrated Castor Oil	77.6	59.1	47.1	35.8	35.8*
Glycerol	7.4	13.5	17.5	21.2	21.2
CaCO <sub>3</sub>	0.1	0.2	0.2	0.3	0.3
Phthalic anhydride	12.5	22.8	29.5	35.8	38.8
Xylol	2.4	4.4	5.7	6.9	6.9

\*In this sample, untreated castor oil was used.

Shorter oil alkyds have higher concentrations of acid and monoglyceride and so higher degrees of polymerization and increase in viscosity.

All the four classes of resins were soluble in both benzene and toluene while only the long oil alkyds (M-AKD-1 and M-AKD-2) could easily dissolve in turpentine. The short oil alkyd (RM-AKD) produced from undehydrated castor oil, was not affected by turpentine and was only swollen up by benzene and toluene. This is likely due to the cross-linking of the polymer.

**Drying performances of alkyd resins.** The short oil alkyds (M-AKD-3 and M-AKD-4) have shorter curing periods than the long oil alkyds (Table 3).

Drying through sunlight or by incorporation of cobalt naphthanate enhanced the drying performances of the resins (Table 3). Longer oil alkyds respond better to sunlight and short oil alkyds, to cobalt naphthanate.

Both, the paints formulated using 60% oil alkyd (M-AKD-2) and the commercial standard (A2986) were able to dry hard within four hours (Table 4). However, the former had comparatively better gloss retention and spreading properties.

**Table 2**  
Physico-chemical properties of alkyd resins

Alkyd resin	Acid value	Solidification time (sec)	Colour	*Viscosity	Thickness (mm)
M-AKD-1	7.62	154.8	Golden Yellow	1	0.023
M-AKD-2	10.80	142.2	Light Brown	2	0.026
M-AKD-3	13.06	100.2	Dark Brown	3	0.040
M-AKD-4	15.10	95.2	Dark Brown	4	0.053
A2986 (control)	11.64	60.0	Brownish-Yellow	1.5	0.033

\*Viscosity Unit is relative and it increases from 1 to 4.

**Table 3**  
Drying performance of alkyd resin

	M-AKD-1	M-AKD-2	M-AKD-3	M-AKD-4	A2986
<i>Indoor drying (30°C)</i>					
TSTT/SEC	15012	12528	720	288	7092
TDF/SEC	18360	14580	4500	2988	8172
TDTT/SEC	21888	16452	7308	4860	15804
TDH/SEC	24912	16740	11160	8640	18792
<i>Sunlight drying (39°C)</i>					
TSTT/SEC	6012	2412	288	216	-
TDF/SEC	6480	3888	2412	2412	-
TDTT/SEC	7200	5292	5112	4788	-
TDH/SEC	10188	6912	6588	5976	-
<i>Cobalt Naphthanate drying (30°C)</i>					
TSTT/SEC	7200	6588	324	180	4464
TDF/SEC	9000	8100	1872	900	5364
TDTT/SEC	10800	9000	2772	2412	5976
TDH/SEC	12024	9900	4284	3312	3064

TDTT, Dry-to-touch time; TDH, Dry hard time; TSTT, Set-to-touch time; TDF, Dust-free time.

**Table 4**  
Drying performance of formulated paints

	TSTT/SEC	TDF/SEC	TDTT/SEC	TDH/SEC
Gloss (M-AKD-2)	11016	13428	14076	14616
Gloss (A2986)	7272	10332	12312	14112

This work has confirmed that dehydrated castor oil can be transformed to a resinous material capable of being used as a potential binder for paint and varnish. As a binder for paints, it shows better spreading properties and has comparative gloss with the commercial alkyde resin when both were used to produce white paint. Also, it was found that the time for alcoholysis was proportional to the amount of oil used while an inverse proportionality was found between the initial acid value of reaction mixture and oil length. The binders from castor oil show remarkable drying properties as they were able to dry indoors without the incorporation of driers. Thus

dehydrated castor oil could be used for local commercial production of alkyd resin.

### Acknowledgement

We acknowledge the management of I.P.W.A. plc Ikeja for supplying some of the materials used in this work. G N Nijikang is grateful to the government of Republic of Cameroon for financial support.

**Key words:** Castor oil, Alkyd resin., Drying properties.

### References

- AOAC 1980 *Official Methods of Analysis*. Association of Official Analytical Chemist 12th ed Washington DC USA.
- ASTM D 1640 06.01.1985a.
- ASTM D 1980 D 1959 D 1962 06.03. 1985b.
- D'Alelio G F 1946 *Experimental Plastics and Synthetic Resins*. John Willey and Sons, New York, USA pp 451.
- Ehrlich A, Smith M K, Patton T C 1959 Castor polyols for urethane foams. *J Am Oil Chem Soc* **36** 149-154.
- Finar J L 1973 *Organic Chemistry: The Fundamental Principles*. Longman, London, England pp 183.
- Ogunniyi D S, Fakayejo W R O, Ola A 1996 Preparation and properties of polyurethane from toluene diisocyanate and mixtures of castor and polyol. *Iranian Polymer J* **5** 56-59.
- Qipeng G, Shixia F, Qingyu Z 1990 Polyurethanes from 2, 4-Toluene diisocyanate and a mixture of castor oil and hydroxyether of bisphenol A. *Eur Polym J* **26** 1177-1180.
- Shreve R N 1967 *Chemical Process Industries*. McGraw-Hill Kogakusha Limited. Tokyo, Japan 3rd ed pp 445.
- Turner G P A 1988 *Introduction to Paint Chemistry and Principles of Paint Technology*. Chapman and Hall, London 3rd ed pp 168.
- Weiss E A 1983 *Oilseed Crops*, Tropical Agriculture Series. Longman Scientific and Technical, London, England pp 530-564.