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# RESEARCH ARTICLE

## INFLUENCE OF POLYBASIC ACID TYPE ON PHYSICOCHEMICAL PROPERTIES OF JATROPHA SEED OIL ALKYD RESIN

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**ABSTRACT**

Jatropha seed oil was employed in the preparation of different alkyd resin samples using alcoholysis and polycondensation process. The alkyds formation was monitored by determining the acid number of the reaction mixture at time intervals and the extents of the polyesterification reactions were evaluated. Both the Jatropha seed oil and its alkyd resins were characterized by Fourier Transform Infra red (FTIR) spectroscopy. Performance evaluation shows that properties of alkyd resin derived from Malic anhydride are comparable with pthalic anhydride alkyd resins. However the Maleic alkyd resin shows better color, and lower poly condensation time. Better results were observed from the blending of pthalic and small portions of maleic anhydride.

## INTRODUCTION

The chemistry and application of alkyd resins have attracted the interest of many chemists from both academia and industry as can be seen from the huge number of papers and patents covering this field. The term alkyd is a variant of "acid" meaning the product of an alcohol and acid. (Mukhtar *et al*, 2007). The fatty acid esters derived from the triglyceride vegetable oils are an attractive source of raw materials for polymer synthesis because of their environmentally benign properties. One of the most widely used types of organic coatings is alkyd resin, which is produced by the reaction of polybasic acid with polyhydric alcohol modified with triglyceride vegetable oils. Alkyd resin has acquired a good reputation because of their economy, ease of application and availability of raw materials. In addition, they are largely biologically degradable polymers because of the oil and glycerol parts, and they are eco – friendly compared to petroleum – based polymers that constitute environmental pollution and degradation. Other unique properties of alkyd resins that make it an indispensable raw material in surface coating industry include gloss and gloss retention good adhesion and hardness properties, film flexibility and durability. (Ibanga *et al*., 2015) Although changing in formulation of long oil alkyd resin through replacing maleic anhydride with phthalic anhydride not only made the stereo structure of the alkyd molecule bigger but also reduced the condensation time of esterification reaction. Therefore the viscosity of resin increased within the same time interval (Vaso *et al*, 2010).

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## MATERIALS AND METHODS

**MATERIALS:** Jatropha seed oil were purchased from National research institute for chemical technology Zaria., Phthalic anhydride, Maleic anhydride, Glycerol, Calcium carbonate, Xylene, were of analytical grade and use without further purification. All other reagents and solvents used throughout this research were chemically pure grade.

**METHODS:** Two stages are involved in preparation of alkyds from Jathropha oil; the first stage of the reaction consist of forming a monoglyceride structure by alcoholysis of the glyceride oil with added polyol. It converts the insoluble polyol and glyceride phases into a single homogeneous monoglyceride phase and which in turn provides a solvent for the polybasic acid added for the next step, esterification of the monoglyceride with diacid to complete the alkyd reaction.

**Physicochemical properties of oil:** The physicochemical properties of the seed oil; such as viscosity, acid value, saponification value, and iodine values were determined using American oil chemists society method (AOCS, 1996), the results obtained are presented in table 1.

**Preparation of Alkyds:** Three different alkyds from each oil, having oil contents of 48.8%, (w/w), and 47.7% (w/w) were prepared using a two stage alcoholysis- polycondensation method. According to the procedure described by ( shaker *et al*,2012).The alkyds were labeled as follows: ALJ1 48.8 % oil (Jathropha oil and Pthalic anhydride),ALJ2 48.8%oil (Jathhropha oil and Maliec anhydride ),ALJ3 48.8% (Jathropha oil with Pthaliec and Maliec ), In preparing each of the alkyds, a known weight of Jathropha seed oils were heated to a temperature between $220^0$C and $240^0$C. Glycerol (10.0 g) and 0.3g Calcium carbonate were added while maintaining the temperature at about $240^0$C.

Alcoholysis was monitored by taking samples of the reaction mixed with anhydrous methanol in a 1:1 volume ratio until a clear solution was obtained. Xylene, Pthalic anhydride, Maliec and their mixture were added while the reaction continued at about 240°C. The reaction was monitored by acid value determination every one hour until a value of 10 or less was reached, then the reaction was quenched by immersing the reaction vessel in cold water. The drop in acid value with time for the different alkyd samples are shown in Figure 1.

**Ftir analysis:** The FTIR spectra of the Jathropha seed oil and the prepared alkyd resin samples were acquired using FTIR CARY 630 agilent at 4000-600$cm^{-1}$. The key absorbtion peaks obtained are presented in Table 2.

**Solvent resistance test:** The dried panels were immersed in a beaker containing distilled water for 18 h. The panels were taken out, air-dried and inspected for any defects, like whitening, shrinkage, film softened, loss of gloss and lift off. Acid resistance test was carried out by preparing Aqueous solution of acid hydrochloric 0.1 M (HCl, in a beaker and warmed up to 80˚C.

The coated panels were immersed in the solution for 1h at 80˚C,and then removed from the solution. They were then dried at room temperature for 24 h before checking for any deterioration.. The water and Alkali resistance were determined using ASTM (D 1647-89, 2004). The resistance of the films to different solvent media (Ethyl methyl ketone, Acetone and toluene) were determined according to ASTM (D1647-59, 1996). Table 5 and 6.

**Drying time test:** In order to determine set-to-touch time, the coated films are lightly touched with the tip of a clean finger and immediately placed against a clean piece of clear glass .The film is considered dry-to-touch when film no longer sticks to finger and does not rub when the finger is lightly moved across the surface.

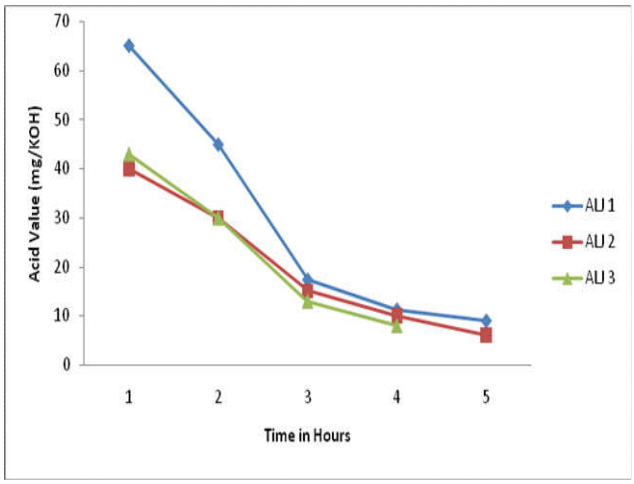
**Flexibility test:** 2cm by 5cm size cardboard paper was used as the test panels. A brush was applied on one side of the panel and allowed to dry. The test was carried out by bending the film on paper to angle 180 in order to check any evidence of crack on the film.

**Adhesion measurement**

Adhesion test was designed to assess the adherent strength of the film according to Shahla 2011. A criss-cross lattice pattern with six cuts in each direction was made after making the required cuts; the film was brushed lightly with a soft brush to remove any detached chips of coatings. A piece, about 75 mm long, of pressure-sensitive tape was separated and placed over the grid and smoothed into place by finger. The tape is rubbed firmly with eraser to ensure sufficient contact with the film. The tape was removed after 90s in an angle close to 180°. The grid area was inspected by visual inspection for removal of any coating from the substrate. The adhesion is rated according to ASTM D3359-94.

## RESULTS AND DISCUSSION

The golden yellow jathropha seed oil used is of higher purity, with lower degree of unsaturation and of higher molecular weight fatty acid as shown in Table 1.




**Graph of drop in acid value with time for jathropha alkyd resin sample**

**Table 1. Physico chemical properties of Jathropha**

| Property | Value |
|---|---|
| Iodine value (mg/g) | 82.4000 |
| Saponipication value (mgKOH/g) | 96.5000 |
| Acid value (mgKOH/g) | 9.2400 |
| Peroxide value (meq/kg) | 36.100 |
| Free Fatty Acid % | 4.6200 |
| Refractive Index | 1.4630 |
| Viscosity (MPa) 28˚C | 91.530 |
| Specific gravity | 0.9131 |

**Table 2. FTIR absorption frequencies of Jatropha seed oil**

| Band number | Literature frequecies | Experimental frequencies | Remark |
|---|---|---|---|
| 1 | 1100-1300 | 1162 | C─O Streching |
| 2 | 1500 ─ 1400 | 1460 | C=Cstretching |
| 3 | 3000 ─ 2850 | 2855, 2924 | C─H Streching.$SP^3$ |
| 4 | 1760 ─ 1735 | 1745 | C=O Streching |

**Table 3. Recipe of jathropha seed oil Alkyd resin**

| INGREDIANTS | ALJ1(g) | ALJ2(g) | ALJ3(g) |
|---|---|---|---|
| Jathropha seed oil | 48.80 | 48.80 | 48.80 |
| Phthalic anhydride | 24.10 | 0.00 | 23.68 |
| Maliec anhydride | 0.00 | 15.9 | 1.59 |
| Glycerol | 10.00 | 10.00 | 10.00 |
| Calcium carbonate | 0.3 | 0.3 | 0.3 |
| xylene | 0.03 | 0,03 | 0.03 |
| Mole ratio (oil:glycerol) | 1:2 | 1:2 | 1:2 |

Key; ALJ1 Alkyd resin sample of Jatropha seed oil and pthalic anhydride
ALJ2 Alkyd resin sample of Jatropha seed oil and Maleic anhydride
ALJ3 A lkyd resin sample of Jatropha seed oil, phallic and Maleic anhydride.

**Table 4 Physico chemical properties of Jatropha Alkyd sample**

| Alkyd sample | Colour | Acid value (mg/KOH) | Iodine value $gI_2/100g$ | Saponification value. | Viscosity 30rpm ($27^0$c) | Refractive index | Relative density |
|---|---|---|---|---|---|---|---|
| ALJ1 | brown | 8.056 | 31.20 | 120.60 | 892.3 | 0.79 | 1.38607 |
| ALJ2 | Light brown | 9.180 | 27.20 | 125.70 | 995.9 | 0.79 | 1.49240 |
| ALJ3 | brown | 7.06 | 30.00 | 122.00 | 983.5 | 0.80 | 1.36678 |

**Table 5. Resistance of Alkyd resins in various solvent**

| Alkyd resins | Water resistance | Alkali resistance 10% NaOH | Acid resistance | | Solvents resistance | | |
|---|---|---|---|---|---|---|---|
| | | | $H_2SO_4$ 20% | HCl 10% | Acetone | Ethyl methyl ketone | Toluene |
| ALJ1 | Excellent | Fair | Excellent | Excellent | I | I | I |
| ALJ2 | Excellent | poor | Excellent | good | I | I | I |
| ALJ3 | Excellent | poor | Excellent | good | I | I | I |

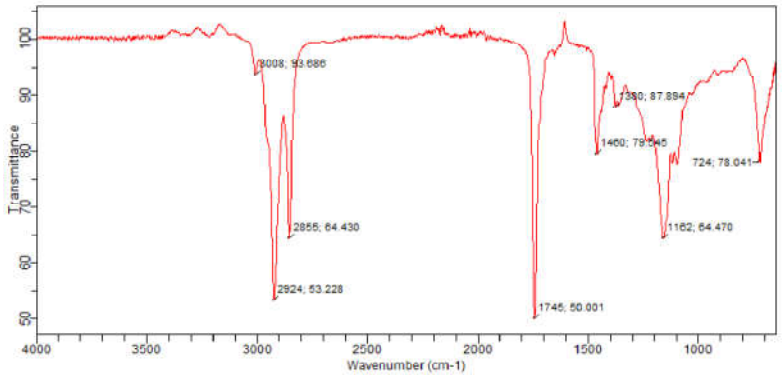
**Table 6. Dryness schedule of Jatropha alkyd resins**

| SAMPLES | CHARACTERISTICS | | |
|---|---|---|---|
| | Set to touch (min) indoor | Set to touch (min)outdoor | Dry through (hour) |
| ALJ1 | 30 | 14 | Overnight |
| ALJ2 | 27 | 17 | Overnight |
| ALJ3 | 28 | 15 | Overnight |

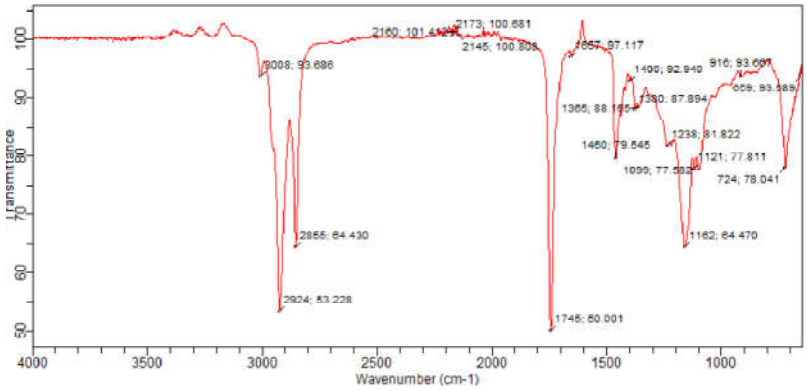
**Table 7. Adhesion, flexibility test of alkyd resin samples**

| Alkydresin samples | Adhesion test | Flexibility |
|---|---|---|
| ALJ1 | 4B | pass |
| ALJ2 | 3B | pass |
| ALJ3 | 3B | pass |

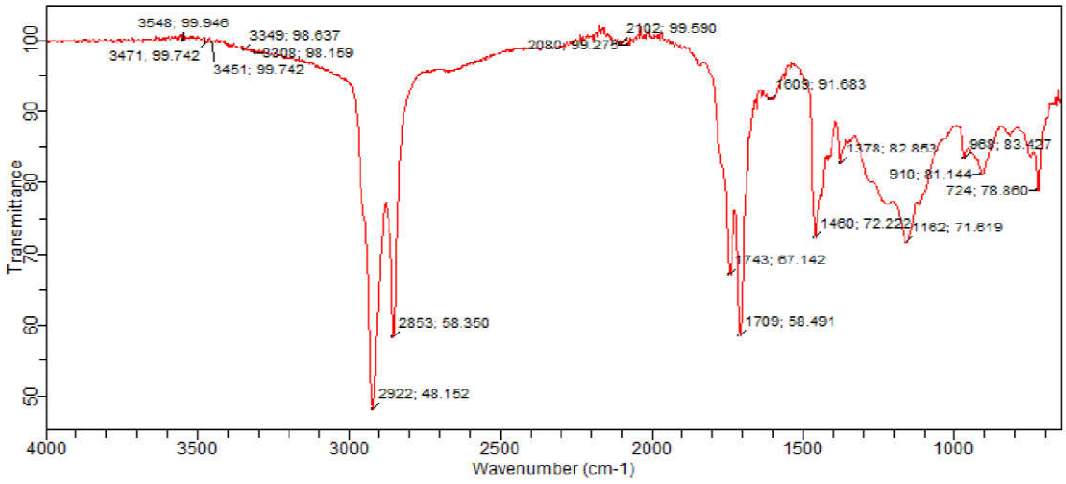
## FTIR SPECTRA OF JATHROPHA SEED OIL



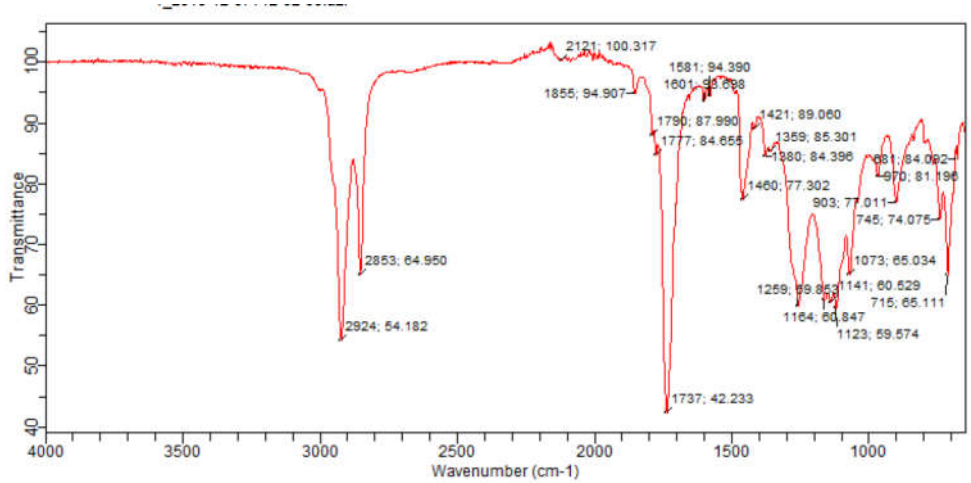

## INFRA RED SPECTRA OF ALJ1



**INFRA RED SPECTRA OF ALJ2**




**INFRARED SPECTRA OF ALJ3**

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