

(RESEARCH ARTICLE)



Preparation and physicochemical characterization of emulsion alkyd resins from cottonseed, *Hura crepitans* L. seed and palm kernel oils

Ibanga O. Isaac ^{1,2,*}, Itoro E. Willie ¹ and Ndifreke S. Idio ¹

¹ Department of Chemistry, Faculty of Physical Sciences, Akwa Ibom State University, Ikot Akpaden, Akwa Ibom State-Nigeria.

² Department of Chemical Sciences, Ritman University, Ikot Ekpene, Akwa Ibom State, Nigeria.

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Abstract

The growing environmental concerns have led to the formulation of new coating strategies by employing inherently waterborne binders as a key component in order to eliminate the toxic volatile organic solvents from protective coatings. *Hura crepitans* L. seed oil (HCSO) was extracted from the seed pulp via solvent extraction and characterized using standard methods. Cottonseed oil (COSO), palm kernel oil (PKO), and HCSO were used in the formulation of emulsion alkyd resins (EMAR) via a two-stage alcoholysis-polyesterification process followed by the introduction of maleic anhydride in the polymer backbone and neutralization of the free acid functionality with triethylamine (TEA). Three grades EMAR (50%) were prepared using these oils, phthalic anhydride, glycerol, and maleic anhydride. The percentage yield of HCSO was 87.93%. While the iodine and acid values of HCSO were 145.90 g I₂/100 g and 1.26 mg KOH/g respectively. COSO emulsion alkyd (CWAR), HCSO emulsion alkyd (HWAR), and PKO emulsion alkyd (PWAR) were highly soluble in water, while the iodine value of 81.38 gI₂/100g, 85.02 gI₂/100g, and 10.45 gI₂/100g was recorded for CWAR, HWAR, and PWAR respectively. The solid content of the binders varied between 84.88 – 85.48%. While the viscosity varied between 1756.00 – 1768.00 mpa.s. HCSO and COSO can serve as good starting raw materials for the synthesis of environmentally friendly waterborne alkyd resin.

Keywords: Waterborne binders; Coatings; Triglyceride oils; Viscosity; Iodine value

1. Introduction

Environmental concerns, limited fossil resources, economic competitiveness, and the emission of toxic volatile organic compounds (VOCs) during the application and curing process from solvent-based alkyd coatings have motivated academic and industrial researchers towards the enhanced use of bio-renewable feedstock for the production of environmentally friendly materials [1,2]. Agricultural resources such as vegetable oil (VO), starch, and protein are bio-renewable resources and are eco-friendly. The use of these materials as monomers for polymer synthesis will not only enhance sustainable development but maintain and sustain the integrity of the environment.

Various vegetable oils have been used in the alkyd resins synthesis as reported in the literature, for example, cottonseed and melon seed oils [3-5], rubber seed and linseed oils [6-8], *Riciodendron heudelotii* oil [9], *Thevetia peruviana* seed oil [10], palm oil, soy oil, and sunflower oil [11-12], *Jatropha curcas* Linneaus seed and castor oils [13-16] and *Hura crepitans* seed oil [17] have been used in the synthesis of modified alkyd resins for solvent borne coatings. Alkyd resin is one of the oldest biopolymer or polyester resins prepared from triglyceride oils. They are only soluble in volatile organic solvents or compounds that are toxic and possess serious threats to the environment and human health [1]. Alkyd resin has acquired a prominent position in the paints and coating industries because of its economy, ease of

* Corresponding author: Ibanga O Isaac

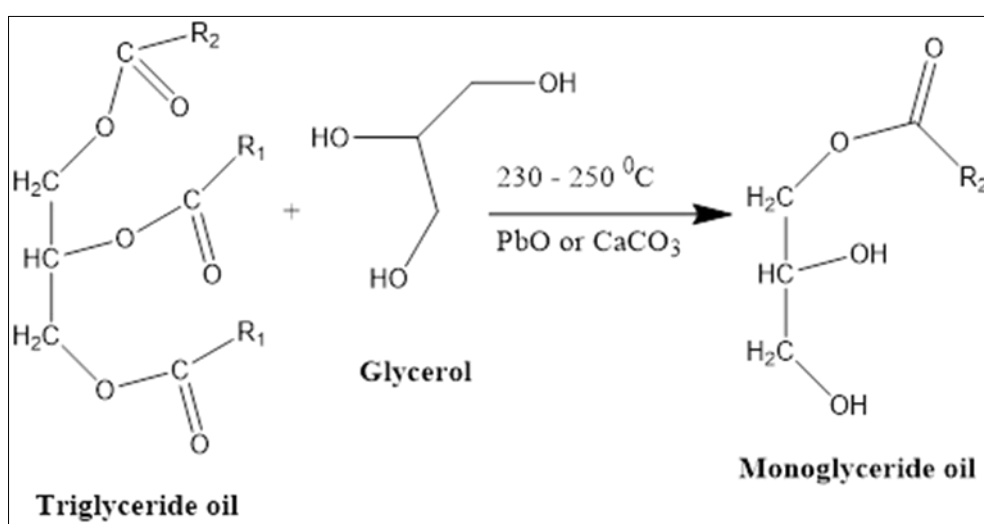
application, and good protection of materials from environmental attack [18], but suffers from its solubility in only VOCs.

Using water instead of toxic VOC is the rationale behind waterborne coatings which is aimed at the total reduction of hazards without compromising the integrity of the environment. The term waterborne is attributed to those coatings systems that primarily use water as the solvent or sometimes up to 80% water with small amounts of other solvents such as glycol ether, propan-2-ol, etc. [19].

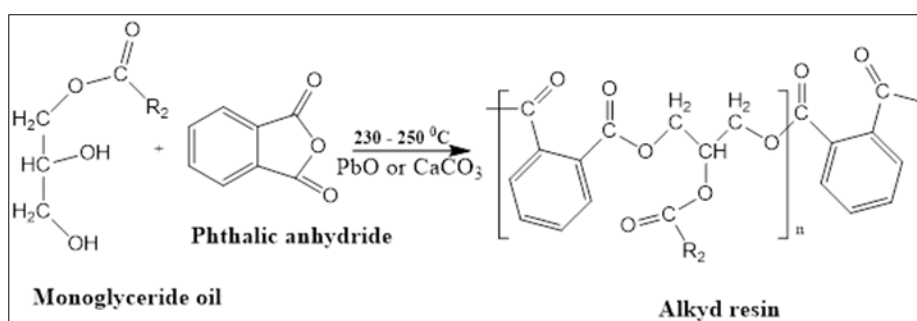
Varieties of approaches have been adopted in an effort to lower VOC levels [20] in coatings. Among these include alkyd emulsions [21], polyurethane and acrylic dispersions which are the most widely available and popular systems that fulfill the various end-user demands. Utilization of maleinized rubber seed oil and its alkyd resin as binders in waterborne coatings has been reported [22].

Conventional alkyds can be converted into water-soluble alkyd resins by introducing hydrophilic carboxylic acid groups into the resin structure or backbone. The chemistry is illustrated as follows (figure 1):

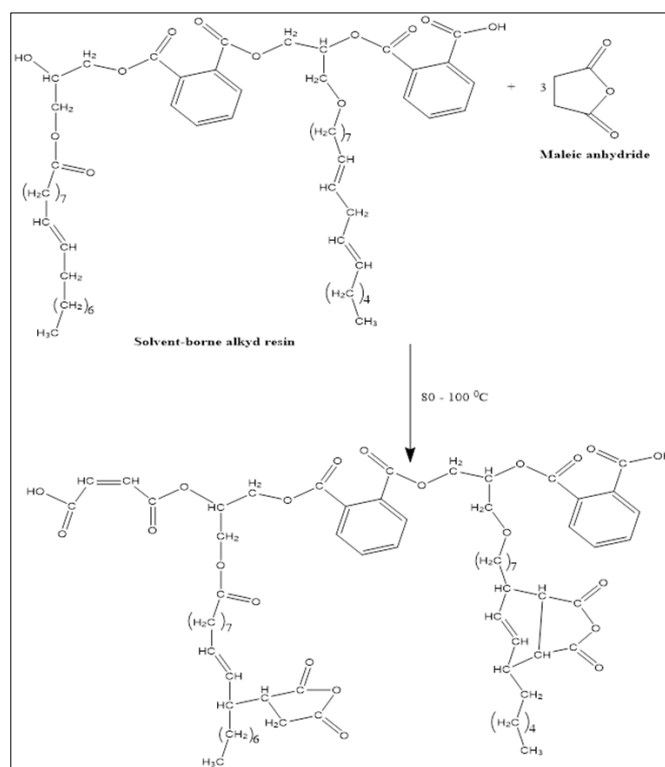
Step 1 Conversion of triglyceride oil to monoglyceride oil (alcoholysis)



Step 2 Polycondensation to form conventional alkyd resin



Step 3 Introduction of hydrophilic carboxylic acid backbone into conventional alkyd resin



Step 4 Neutralization of the free carboxylic acid functionality to form waterborne alkyd resin

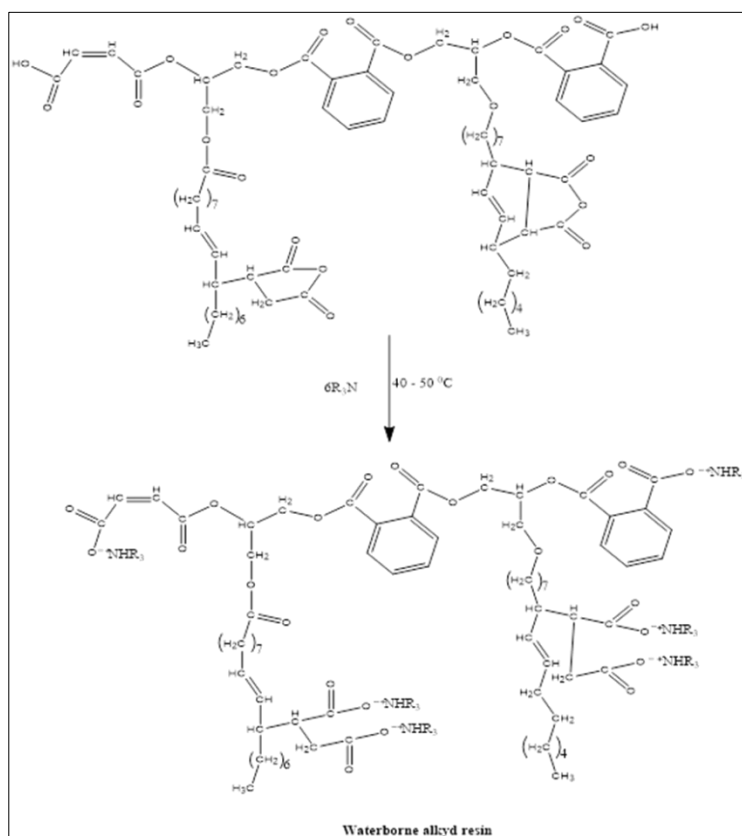


Figure 1 Proposed reaction scheme for the preparation of waterborne alkyd resin

Hura crepitans L. plant, also known as sandbox tree, possum wood, or Jabillo is an evergreen tree of the spurge family (Euphorbiaceae) [23]. It has the characteristics of short densely crowded and pointed spines on the trunk and branches. The leaves, stem bark, roots, and seeds of this plant have been reported to exhibit several therapeutic applications, which include the treatment of skin diseases, rheumatism, and intestinal worms in leprosy [24]. *Hura crepitans* L. seed oil (HCSO) contains a high percentage of polyunsaturated linoleic acid [25], indicating a semi-drying property. Besides being a source of seed oil, it may possibly have edible uses, industrial applications, and pharmaceutical importance [26]. The utilization of this oil as an oil phase in the production of emollient cream has been reported [23]. The seeds have high oil content which makes them a suitable naturally occurring renewable resource non-edible oil. In this research, an effort had been made to introduce the hydrophilic carboxylic acid backbone into the conventional alkyd resins from *Hura crepitans* L. seed oil (HCSO), cottonseed oil (COSO), and palm kernel oil (PKO) alkyd resins. The physicochemical properties and water solubility test was performed on the emulsion alkyd samples.

2. Material and methods

2.1 Sample collection and preparation

Palm kernel oil (PKO) was purchased at Akpan Andem market, Uyo, while cottonseed oil (COSO) was purchased at Sabongari market, Kano. Mature and dry *Hura crepitans* (sandbox) tree seeds were collected between April and May 2022 from shelter Afrique in Uyo, Ete community in Ikot Abasi L.G.A., State College Secondary School in Ikot Ekpene and Ikono Local Government Headquarter, all in Akwa Ibom state, Nigeria. The pods containing the seed were deshelled and the kernels were obtained as shown in figure 2. The kernels were dried in a laboratory oven at a temperature of 105 °C for about 72 hours to remove the moisture. The dried sample was milled into uniform powder using a Corona traditional grain mill REF121 (100 µm mesh size).

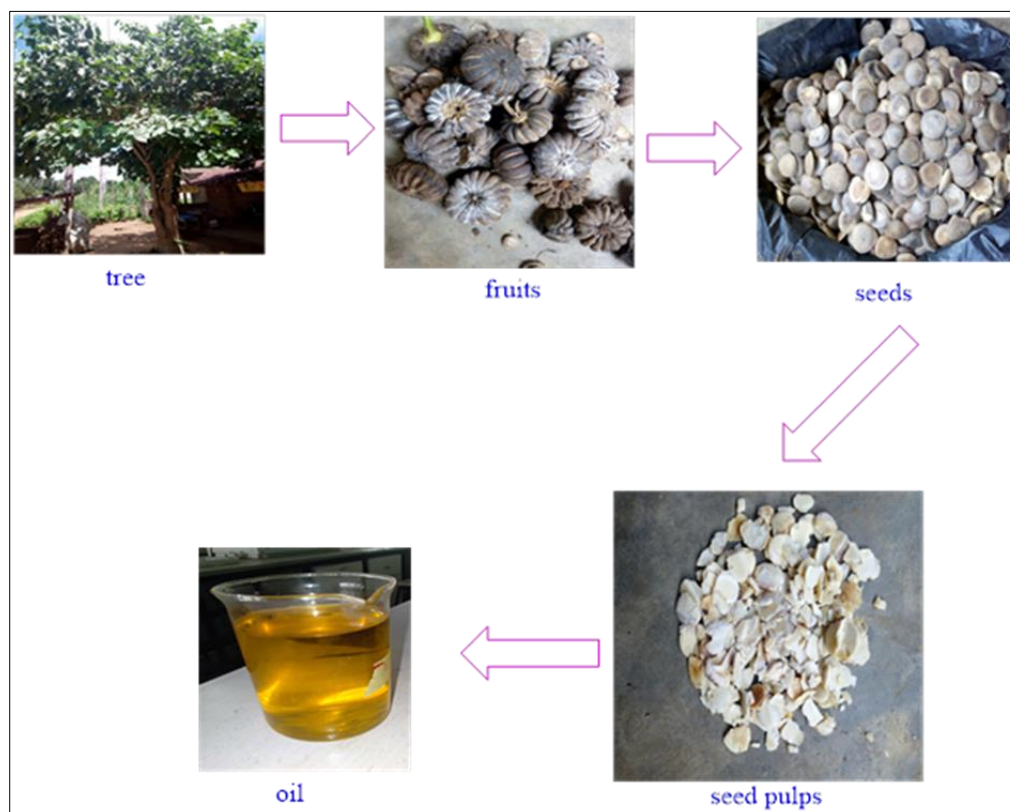


Figure 2 Tree, fruits, seeds and oil of *Hura crepitans* L

The oil was extracted from the milled sample through solvent extraction with analytical grade petroleum ether (60 – 80 °C) following the method described by Isaac and Ekpa [3] with slight modifications. 500 g of milled sample was soaked, and macerated with 2000 ml of petroleum ether and filter to separate the oil which is soluble in petroleum ether from the cake. The filtrate was kept overnight and the debris that settled at the bottom of the container was removed via decantation. The mixture was concentrated under reduced pressure at 60 °C to separate the oil and the solvent. A golden yellow colour oil was obtained and labeled as HCSO and kept for further use.

2.2 Preparation of water-soluble alkyd resins

The method described by Ling *et al.* [11] with slight modifications was used in the preparation water-dispersible alkyd resins. The conventional solvent-borne alkyd resin was first synthesized using the monoglyceride method which involves two steps. The first step is the trans-esterification of triglyceride oils by glycerol (glycerolysis) in the presence of PbO or CaCO₃ as the catalyst at an elevated temperature. The second step involves the reaction of the monoglyceride oil with excess phthalic anhydride (polyesterification) to form solvent-borne alkyd resins. The conventional alkyd resin was made to react with maleic anhydride to introduce the hydrophilic carboxylic acid groups into the resin structure or backbone, resulting in the formation of water-soluble alkyd resins. The free acid functionality was neutralized with TEA to afford an environmentally friendly surface coating binder.

2.3 Preparation of monoglycerides

50% oil-length monoglycerides were prepared from HCSO, PKO, and COSO respectively. A three-necked, 1000 ml round-bottomed flask equipped with a mechanical stirrer, dean and stark apparatus, reflux condenser, and thermometer was charged with the calculated amount of HCSO (Table 1) and heated to a temperature between 220 °C and 240 °C with constant stirring, followed by addition of 28.80 g of glycerol and 0.15% by weight of PbO (see the recipe in Table 1) as the catalyst. The glycerolysis product, however, may be expected to be consisting majority of α -monoglyceride (>60%) while a minor presence of β -monoglycerides, $\alpha\alpha'$ -monoglycerides, $\alpha\beta$ -diglyceride, triglyceride, and glycerol was unavoidable [11, 27-29]. The reaction was continued until almost all the triglycerides were converted into α -monoglycerides. The extent of conversion or completion of alcoholysis or glycerolysis was monitored by removing aliquots of the products from the reaction flask using a glass rod to test its solubility in methanol. At 10 min intervals, two drops of the sample were added to four drops of methanol in a clear glass slide until a clear (non-colloidal) liquid was observed [30-31], indicating the end of glycerolysis.

2.4 Polycondensation of the monoglycerides to form conventional alkyd resin

This is the second step in the preparation of emulsion alkyd from triglyceride oil. It involves polycondensation of monoglyceride moiety obtained from transesterification of the triglyceride oils with glycerol with a calculated amount of phthalic anhydride. The reaction continues in a three-necked, 1000 ml round-bottomed flask equipped with a motorized stirrer, a dean and stark trap attached to a reflux condenser, and a thermometer.

The temperature of the reaction mixture was first reduced to 180 °C, followed by the addition of 51.2 g of PA and 34.00 g of xylene as azeotropic solvent. The temperature was raised gradually to 230 °C and kept at 230–250 °C. The reactions were followed with acid value determination. The acid value was determined by titration as reported by Huang *et al.* [32]. The poly-condensation reaction was continued until the acid value came to 12-14 mg KOH/g.

2.5 Introduction of hydrophilic carboxylic acid backbone into conventional alkyd resin

Table 1 Recipe for the formulation of waterborne alkyd resins from COSO, HCSO, and PKO

Ingredient	HVAR	PVAR	CVAR
<i>Hura crepitans</i> L. seed oil (HCSO) (g)	80.00	-	-
Palm kernel oil (g)	-	80.00	-
Cottonseed oil (g)	-	-	80.00
Glycerol (GL) (g)	28.80	28.80	28.80
Phthalic anhydride (PA) (g)	51.20	51.20	51.20
Maleic anhydride (MA) (g)	12.00	12.00	12.00
Triethylamine (TEA) (g)	34.00	34.00	34.00

The temperature of the conventional alkyd obtained was reduced to 80 °C and the calculated amount of maleic anhydride (MA) 12.00 g (Table 1) was added into the reaction mixture to introduce hydrophilic carboxylic acid groups into the resin structure followed by the addition of 34.00 g of xylene to help wash the sublimate of MA into the reaction mixture in order to have a uniform mixture. The reaction was stirred at 80 - 100 °C until an acid value of 40 - 60 mg KOH/g was obtained. The free fatty acid was neutralized by addition of 34.00 g of triethyl amine (TEA) at 40 - 45 °C. Water solubility test was carried out, by adding propan-2-ol to form a 70 % (wt.) solution. Deionized water was added

to obtain a 50 % solution. The crude waterborne alkyd of COSO, HCSO, and PKO respectively was labelled as CWAR, HWAR, and PWAR respectively.

3. Results and discussion

3.1 Extraction of *Hura crepitans* L. seed oil

The percentage yield of HCSO is given in Table 2, the high percentage yield (87.93 %) of HCSO obtained in this study compared to those reported in the literature of 38.20 % [33], and 47.80 % [25] respectively (Table 2) may be attributed to the maturity of the seed, season of collection of the seed, geographical location, and method of extraction. Chen *et al.* [34] stated that the solvent extraction method is the most popular method of the extraction of seed oil mainly because of the method's high extraction efficiency (over 99 %) as well as its capability to handle large quantities. *Hura crepitans* L. seed is a potential candidate for locally sourced triglyceride oil for industrial applications due to its high percentage of the oil yield.

The iodine value of *Hura crepitans* L. seed oil of 145.33 gI₂/100 g shows that it is a drying oil, this means that it can dry to form a thin film on a surface via oxidation on exposure to air while that of COSO shows that it is a semi-drying oil, but other reports such as Umoren *et al.* [33] and Isaac and Ekpa [3] respectively gave the iodine value of HCSO and COSO respectively as 122.08 I₂/100 g and 100.25 I₂/100 g classifying them as semi-drying oils. The iodine value of PKO shows that PKO is a non-drying oil. The iodine value of HCSO and COSO indicates a high degree of unsaturation and their suitability for the preparation of alkyds via the monoglyceride process. The result obtained in this research corroborates that reported by Oyekunle and Omode [25] in which the iodine value of HCSO was determined as 149.10 gI₂/100 g. The iodine value of HCSO determined by Umoren *et al.* [33] is far smaller than the value obtained in this research. The difference could be attributed to the biodiversity of plant products, variation in season, or maturity of the seed. The iodine value of HCSO compares favourably with other seed oils like rubber seed oil 146.55 gI₂/100 g [22], and *Ricinodendron heudelotii* seed oil 156.82 gI₂/100 g [9] that have been used in the preparation of air drying binders for surface coating products.

Table 2 Physicochemical properties of HCSO, COSO and PKO

Quality parameters	HCSO			COSO	PKO
Percentage yield (%)	38.20 ^a	47.80 ^b	87.93 ^c	-	-
Specific gravity (at ±30 °C)	-	-	0.91	0.90	0.92
Acid value (mgKOH/g)	1.64	4.10	3.25	1.45	3.12
Saponification value (mgKOH/g)	210.30	202.00	183.34	188.23	242.63
Iodine value (gI ₂ /100 g)	122.08	149.10	145.33	125.13	12.69
Viscosity (mpa.s)	-	-	457.00	-	-
Ester value	208.74	197.70	180.09	186.78	239.51

^a values gotten from Umoren *et al.* (2001); ^b values gotten from Oyekunle and Omode (2008); ^c values gotten from this study

The low acid value of *Hura crepitans* L. seed oil and palm kernel oil compares favourably with those reported for other seed oils like *Jatropha* seed oil of 2.1 mg KOH/g [9], African bean seed oil of 3.25 mg KOH/g [35] and yellow oleander oil of 0.66 mg KOH/g [10]. This suggest its application as good edible oil. The high viscosity of *Hura crepitans* L. seed oil describes its resistance to flow. The specific gravity of HCSO 0.91, COSO 0.90, and palm kernel oil 0.92 is similar to the 0.912 specific gravity reported for Yellow oleander seed oil [10].

3.2 Emulsion alkyd resin

Waterborne alkyd resin (50% oil length) was prepared with COSO, HCSO, and PKO by first preparing the conventional alkyd resins of these oils using the monoglyceride process, followed by the introduction of maleic anhydride (MA) into the polymer backbone to enhance water solubility and neutralization of the free acid functionality by triethylamine (TEA). The physicochemical properties of the waterborne alkyd samples are presented in Table 3. The results show that the iodine value of the resins is less than the iodine value of the triglyceride oils. This probably indicates the addition of maleic anhydride to the polymer backbone which is believed to undergo a kind of Diels-Alder type I and II cyclo-addition

across the double bonds of the polymer chains as shown in the proposed reaction scheme in Figure 1 decreases the degree of unsaturation of the resultant product. A previous study by Isaac and Akpanudo [36] on maleinization of cottonseed oil shows that the iodine value of the maleinized oil decreases as the percentage of MA increases and was generally lower than the iodine value of cottonseed oil. Similarly, the viscosity of the resins was high, indicating that they are highly viscous polymers. Other properties such as specific gravity, and volatile and non-volatile matter were within the recommended limit for such products.

Table 3 Physicochemical properties of water soluble alkyd samples

Parameters	CWAR	HWAR	PWAR
Colour	Reddish brown	Brown	Dark brown
Specific gravity (at ± 30 °C)	0.93	0.95	0.90
Iodine value ($\text{gI}_2/100\text{g}$)	81.38	85.02	10.45
Viscosity (mpa.s)	1760.00	1768.00	1756.00
Non-volatile matter (%)	84.88	85.48	85.23
Volatile matter (%)	15.12	14.52	14.77

3.3 Water solubility test of emulsion alkyd samples

Water solubility testing was carried out by diluting the resin with 250 ml deionized water for CWAR, HWAR and PWAR respectively and there was no precipitate formed indicating that they are highly soluble in water. The colours of the emulsion products varied from milky for CWAR, light brown for HWAR and dark brown for PWAR. The dark brown colour of PWAR is due to the influence of the colour of the binder which was dark brown. The sharp colour difference from other emulsion binders was due to the sudden rise in temperature of the heating mantle during the preparation of the emulsion alkyd resin of PKO that resulted in the charring of part of the product.

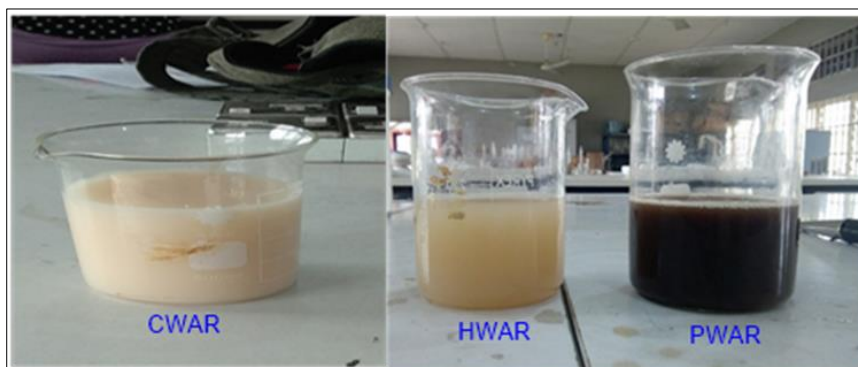


Figure 2 Water solubility capacity of CWAR, HWAR and PWAR

4. Conclusion

Hura crepitans L. seed oil can be extracted in large quantities from the mature seed of the plant. Conventional alkyd resins can be conveniently converted to emulsion alkyd resins via the introduction of maleic anhydride into the polymer backbone. The non-edible triglyceride oils such as COSO, HCSO, and PKO if properly processed can serve as an alternative for the petroleum feedstocks in the synthesis of an environmentally benign water-soluble binder for the production of emulsion paints.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that there is no conflict of interest.

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