The utilization of *Lagenaria breviflorus* seed oil in the synthesis of alkydresins

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Abstract

Lagenariabreviflorus seed oil was used in the formulation of two grades of alkyd resins using a two-stage alcoholysis-polyesterification method. The oil was extracted with n-hexane and oil content of 29.50% was obtained. The physicochemical analysis of the seed oil revealed that *L.breviflorus* is semi-drying (iodine value, 110mg I_2/100 g) containing74.40% unsaturated fatty acids with linoleic acid (61.12%), stearic acid (14.98%), oleic acid (13.01%) and palmitic acid (10.34%) being the most abundant. Properties of the alkyds having oil content of 40% (I) and 50% (II) were evaluated. The acid value of prepared alkyds was observed to decrease as the reaction progressed. The infra-red spectra peaks of the alkyds compared well with that of the commercial sample indicating that *L. breviflorus* seed oil has been successfully converted to alkyd resin. Evaluation of prepared alkyds by determination of acid values, iodine values, solubility in xylene, toluene and acetone; resistance of dry film to acid, alkali and water; scratch and gouge film hardness and drying time revealed that *L. breviflorus* seed oil is a potential raw material for the coatings industry.

Keywords: *Lagenaria breviflorus*; Seed oil; Linoleic acid; Oleic acid; Alkyd resin

1. Introduction

Lagenaria breviflorus Robert (family Cucurbitaceae) is commonly referred to as wild colocynth; it is a perennial climber with scabrid leaves. The fruits (Fig. 1) are dark green with creamy blotches, and are ovoid [1, 2]. It usually lies on the ground and climbs over shrubs and herbs by means of axillary tendrils [3]. The fruit and seeds are extremely bitter, non-edible and contain good amount of alkaloids – tetracyclic triterpenoids [4]. According to Umerie *et al.*[3], an average-size *L. breviflorus* fruit hasup to 400 seeds containing fixed oil.

In the surface coating industries, oil-modified polyestersor alkyds dominate as the most widely used solvent borne binder [5]. These low molecular weight esters which are products of polycondensation reaction of polyol, diacid, and drying or semi-drying triglyceride oils or fats are very flexible and versatile and have been found to be compatible with other polymer systems [6]. Despite the advantages of alkyd such as good adhesion, film flexibility, colour, gloss, broader temperature range application, etc., there are some draw-backs, for instance, its susceptibility to alkali as a result of the splitting of the ester linkages by hydrolysis, this reaction is very typical of esters. In addition, compared to other synthetic resin, alkyd resin is relatively slow-drying. However, it can be modified by several physical (blending) and chemical (co-polymerization) processes which will lead to products that could meet up with wide range of applications. This is borne out of the fact that it is very compatible with a variety of other polymers, such as, acrylic, nitrocellulose, phenol, epoxy resin, etc, where the technical features of both polymers are combined to give products with improved properties that meet up with a wide range of end utilization [7, 8].

In most developing countries, the over dependence of the coatings industry on food-grade oils has led to a decline in the amount available for human consumption. This development is not only responsible for the rising costs of conventional vegetable oils used in coatings but has partly contributed to the increases recorded in the cost of finished coating products such as paints and varnishes. In order to reduce the demand for food grade oils that is used in industrial production, there is a need to explore new sources of oil [9].

The physicochemical properties and fatty acids composition of seed oil of *L. breviflorus* have been studied; seed oil reported to contain high percentage of polyunsaturated linoleic acid indicating a semi-drying property [10, 11]. Umerie *et al*. [3] recorded iodine value (137.2 mg KOH/100g) for *L. breviflorus* seed oil (which indicates its drying property) and suggested its exploitation by the surface coatings industry. Studies on the preparationof alkyds from oil extracted from seed of a number of crops namely *Hura crepitans*[12], valley tomato [13], rubber [14], African locust bean [15], and *Jatropha curcas* seed oils [16], *Ricinodendron heudelotii* [17], *Thevetia peruviana* [18], *Millettia pinnata* (L.) Panigrahi [19], and cotton seed [20] just to mention but a few have also been reported in the literature. It is worthy of note that the oil chosen for the production of alkyds usually has a profound effect on the properties of the finished alkyds [21, 22].

As part of our contribution to the growing interest of exploring new sources of oil for application in the surface coatings industry, this paper reports the extraction, characterization and preparation of alkyd resins from the seed oil of *L. breviflorus*in a view to diversify its usage.

Figure 1: Fruits of *Lagenaria*

2. Materials and methods

2.1 Materials

Analytical grade phthalic anhydride (BDH Chemical, Poole, England), calcium (II) oxide (BDH Chemical, Poole, England) and glycerol (May & Baker, England) were used as obtained without further purification. The reference alkyd was a commercial medium oil alkyd purchased from Aba Industrial Layout, Nigeria.

The mature fruits of *L. breviflorus* were harvested from the wild in Abak Local Government Area of Akwa Ibom State, Nigeria. The samples were identified and authenticated by Dr. M. E. Bassey of the Department of Botany and Ecological Studies, University Uyo, Akwa Ibom State. The seeds were obtained from the matured dried fruits. The seeds were deshelled, sun-dried and pulverized in the laboratory. The ground seed of *L. breviflorus* (577.9 g) was soaked in hexane for 72 hours and fixed oils obtained by distillation. The oil samples were placed in the oven at 50 ºC for 48 hours to ensure that all traces of the solvent evaporated.

2.2.Fatty Acids Composition Analysis

The fatty acids composition of the oil was evaluated by Gas Liquid Chromatography of fatty acid methyl esters (FAMEs) prepared by boron trifluoride-catalyzed trans-esterification [23, 24]. FAMEs were analyzed on a HP 6890 Powered with HP ChemStation Rev. A09.01 (1206) software and equipped with a hydrogen flame ionization detector (FID). Separation was performed using a fused capillary column (HPINNOWax, 30 m × 0.25 mm × 0.25 μm) as stationary phase. The oven temperature was programmed as follows: initial temperature at 60ºC, first ramping at 12ºC/min for 20 min; second ramping at15ºC/min for 3 min, and maintained for 8 min. The injector and detector temperatures were 250ºC and 320ºC respectively. The carrier gas was nitrogen and a split ratio of 20:1was used. The FAMEs were identified by comparing their retention times to those of a standard mixture of fatty acids and the peak areas were integrated.

2.3. Physico-chemical properties and infrared spectrum of seed oil

The physicochemical properties of the seed oil: refractive index, iodine value, saponification value, free fatty acids/acid value and peroxide value were determined according to the methods described in ref [24].

2.4. Preparation of alkyd resins

The oil sample was measured into a three-necked reaction flask and heated to a temperature between 220 and 240ºC with constant stirring. A calculated amount of glyceroland calcium (II) oxide (0.5%) based on oil content was added (Table 1) while maintaining the temperature at 240ºC until alcoholysis reaction was completed [25]. The completion of alcoholysis was monitored by removing aliquots (two drops) of the reaction mixture from the reaction flask after every 5 minutes and testing for its solubility in four drops of methanol. A complete dissolution of the aliquot in methanol to give a clear solution indicated the completion of alcoholysis reaction. The reaction mixture was allowed to cool to 180ºC, when a measured quantity of polybasic acid followed by xylene (5% by weight of the total ingredients charged) were added (Table 1), and the temperature raised quickly to about 230ºC.

Table 1: Formulations for the different alkyd resins

Sample Composition (g)	LB-SOA	LB-MOA
Oil	30.00	36.50
Phthalic anhydride	29.70	25.54
Glycerol	15.30	10.96
CaO	0.04	0.02
Total	75.04	73.02
Oil content (%)	40	50

LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd

The temperature was maintained between 230–250ºC throughout the duration of the reaction. The extent of polycondensation reaction was monitored by removing the aliquots (2.0 g) of the reaction mixture at 30 minutes intervals and dissolving it in a neutralized mixture of toluene/ethanol (1:1 ratio). The resulting mixture was also titrated with standard KOH (0.1 M) solution using phenolphthalein as indicator. The reaction was quenched by allowing it to cool when the acid value was about 10 mg KOH/g.

2.5. Evaluation of alkyd resins

The alkyd resins were tested for various performance properties such as solubility, hardness and chemical resistance using the standard methods ASTM D1639-90 and ASTM D1647-89 [26 – 28]. The resins were also tested for drying performance using the touch method specified in ASTM standard [27] (ASTM D1640-69). Each resin (3.0 g) was dissolved in acetone, toluene and xylene (5 ml each) respectively with intermittent stirring. The drying time of the alkyd resins were studied at indoor and outdoor temperatures using various substrates - wood and mild steel panels with cobalt drier. Each alkyd resin (6.5 g) was mixed with cobalt drier (0.32 g) and xylene (4 g) in a 100 ml beaker. The sample mixtures were applied on the surfaces of wood and mild steel panels and monitored periodically for set-to-touch and dry through time. The sharpened edges of a set of different grades of pencils were used to press slowly along the surface with force. The hardness was expressed in terms of the designation of the hardest pencil that failed to scratch the film to give a continuous mark.Infrared spectroscopic analysis was also performed on the prepared and commercial alkyds. The FTIR spectra were recorded with a Shimadzu FTIR spectrophotometer (100 series).

3. Results and discussion

3.1. Physicochemical properties of L. breviflorus seed oil

The physicochemical properties of the seed oil are presented in Table2. The seed oil was liquid at room temperature with percentage yield of 29.5%. The oil yield is within the range obtained for other cucurbits: musk melon (29.4%), bitter gourd (34.8%), red pepper (26.3%) and snake gourd (31.8%) reported by Vibhute [29]. Analysis of *L. siceraria, L. breviflorus* and *L. cylindrica* seed oils by Essien *et al*. [11], gave percentage oil yield within the range 22 –29%. Umerie *et al*. [3], in their work on *L. breviflorus* equally obtained 22.9% of oil from the plant seed. This variation may be due to specie, season of harvest or method of processing.

Table 2: Physico-chemical properties of *L. breviflorus* seed oil

Property	Values
% yield	29.50
Colour	Brown
Refractive index (25 ºC)	1.57
State (28 ºC)	Liquid
Acid value (mgKOH/g)	5.57
Iodine value (g I_2/100 g)	110.0
Saponification value (mgKOH/g)	213.18
Peroxide value (meq. O_2/kg)	7.50
% Free fatty acids	2.8

The iodine value (110 gI_2/100g) of *L. breviflorus* oil obtained in this study is in congruence with iodine value of other cucurbits: musk melon (111.3 gI_2/100g), bitter gourd (128.8 gI_2/100g), rough cocklebur (133.4 gI_2/100g), red pepper (136.4 gI_2/100g) and snake gourd (124.7 gI_2/100g) [29]. Similar iodine values of some seed oils: rubber (136.2 gI_2/100g), melon (112.9 gI_2/100g), soybean (128.4 gI_2/100g) and jojoba (95 gI_2/100g), used in the synthesis of alkyd resins have been reported in literature [30 – 34]. Umerie *et al*. [3] reported iodine value (137.2 gI_2/100g) for *L. breviflorus* seed oil, which is slightly higher than the value obtained in this study, and suggested its use for alkyd synthesis.

The saponification values of the investigated seed oils (213mgKOH/g) were comparable with some seed oils: bitter gourd (186.1 mgKOH/g), rough cocklebur (197.2 mgKOH/g), snake gourd (185.4 mgKOH/g), bottle gourd (221.0 mg KOH/g) and lump-in-neck gourd (225.1 mgKOH/g) [29, 35].

The acid values (5.57 mgKOH/g), the percentage free fatty acids (2.8%) and peroxide values (7.5 meq.O_2/kg) for seed oils (Table 2) are low in line with some studied cucurbits: musk melon, bitter gourd, red pepper and *L. siceraria* [11, 29]. The acid value of the investigated seed oil is slightly higher than results obtained for oils of *C. maxima* (2.04 mgKOH/g), *C. lanatus* (2.33 mg KOH/g) and *M. charantia* (2.73 mgKOH/g) [36 – 38]. According to Chawla [39] and Umerie *et al*. [3], low acid value and peroxide value are indications of reduced early onset of rancidity of oils, hence, long shelf-life. The level of acidity of oil used in alkyd resins synthesis also determines the quantity of excess polyol required to neutralize the acid for the polycondensation reaction [25]. Thus, the relatively low values of acid indexof *L. breviflorus* seed oil further suggest the preference of the seed oil for alkyd resin synthesis and other industrial applications.

3.2 Fatty acids composition of L. breviflorus seed oil

The fatty acids profile of *L. breviflorus* seed oil is tabulated in Table 3. The result reveals the presence of four major fatty acids: palmitic (10.3%), stearic (14.98%), oleic (13.0%) and linoleic (61.12%) acids. The studied seed oil has higher percentage of unsaturated fatty acids (74.40 %) than saturated(25.6%). The result follows the trend (linoleic acid > oleic acid > palmitic acid) of fatty acids composition of some legume seeds as described by Olaofe *et al.* [35]. The high linoleic acid content makes it particularly suitable for the manufacture of a range of protective coatings.

3.3 Changes in acid value during synthesis

The fall in acid values during the polyesterification stage of alkyd resin synthesis was determined at suitable time intervals. The decrease in acid value is distinct for the short and medium oils alkyds. Thus the rate of decrease in acid value can be said to depend on the amount of oil used in the formulation. The change in acid value from the plots (Fig. 2) was more rapid at the early stage than in the later. Similar observation has been reported in the literature [15, 40].

The rapid initial changes are attributed to the different reactivities of primary and secondary hydroxyl groups of the glycerol; primary hydroxyl group reacts faster with the carboxyl group of the phthalic anhydride than the

secondary. Also, high amount of water was absorbed by the calcium oxide used at the early stages of the reaction and then decreased as the reaction progressed. This can be attributed to the high initial esterification rate. Generally, the rate of polyesterification decreases towards the end of the reaction as the concentration of the free acids decreases [32].

Table 3: Fatty acids composition of *L. breviflorus* oil

| Fatty acids	% Composition
Myristic acid (C14:0)	0.19
Palmitic acid (C16:0)	10.34
Palmitoleic acid (C16:1)	0.12
Stearic acid (C18:0)	14.98
Oleic acid (C18:1)	13.01
Linoleic acid (C18: 2)	61.12
Linolenic acid (C18:3)	0.15
Arachidonic acid (C 20:0)	–
Behenic acid (C22:0)	0.06
Lignoceric acid (C24:0)	0.03
Total saturated acids	25.60
Total unsaturated acids	74.40
Monounsaturated acids	13. 13
Polyunsaturated acids	61.27
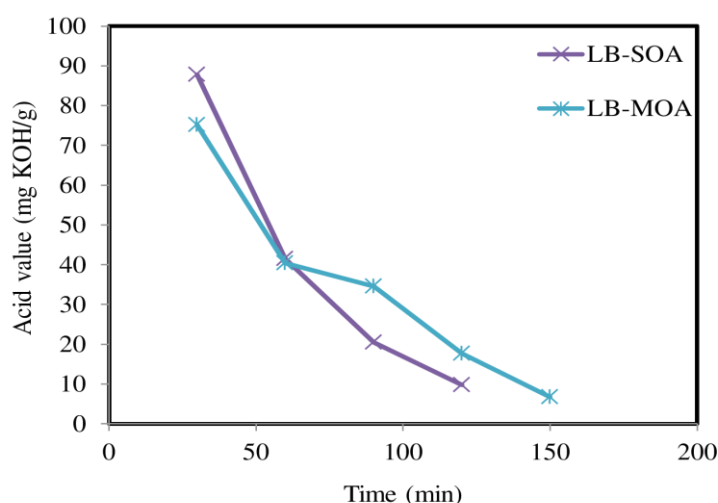
Figure 2: Changes in acid value with reaction time

3.4 *FTIR spectra of the alkyd resins*

The infrared spectra of the short oil alkyd, medium oil alkyd and the commercial sample are shown in Figs. 3–5, respectively.

All the samples had absorption bands at 1735.99–1726.35 cm^{-1} suggesting a phthalate type of ester group is present. It also suggests a carbonyl stretch of an ester. Similar (C=O) bands have also been observed elsewhere for carbonyl esters of alkyd resins [12, 28, 40, 41]. A broad band peak between 3493.20 cm^{-1} and 3450.77 cm^{-1} in the spectra, results from free OH group; also reported by Chawla [39], Ezeh *et al.* [12] and Dhoke *et al.* [42]. Carbon–oxygen stretch at 1276.92–1269.20 cm^{-1} in all the samples suggests an ester group [43]; exclusive absorption above 3000 cm^{-1} reveals an aromatic ring substituent. Also, all the samples exhibited absorption bands at 987.59–704.04 cm^{-1} and this could indicate a monosubstituted aromatic ring. The spectra also show C-

H vibrations 2926.11 cm^{-1} (LB-SOA), 3009.05 cm^{-1}, 2924.18 cm^{-1} (LB-MOA), and 3064.99 cm^{-1}, 2924.18 cm^{-1} (COA), indicative of C=C-H bond in the alkyd structures; similar interpretation of spectra is also reported by Hlaing and Oo [41]. The similarity in stretching frequencies of implicated functional groups of the prepared alkyd resins and the commercial alkyd suggest successful conversion of the seed oil to alkyd resins.
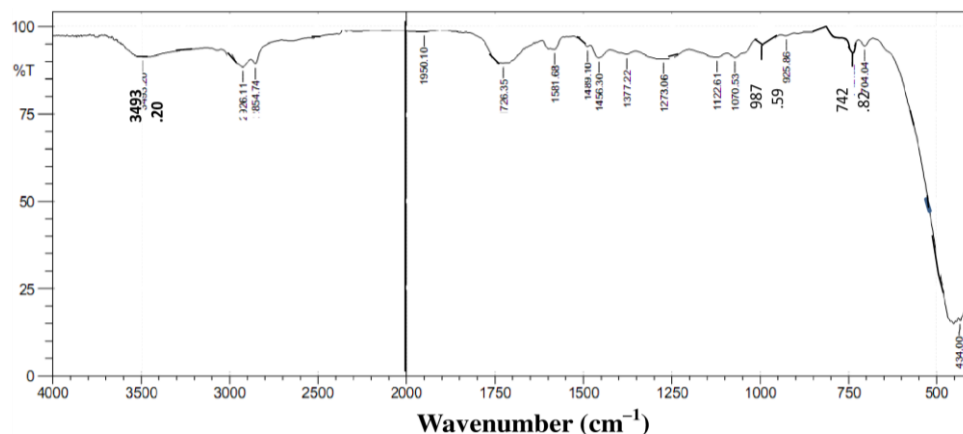

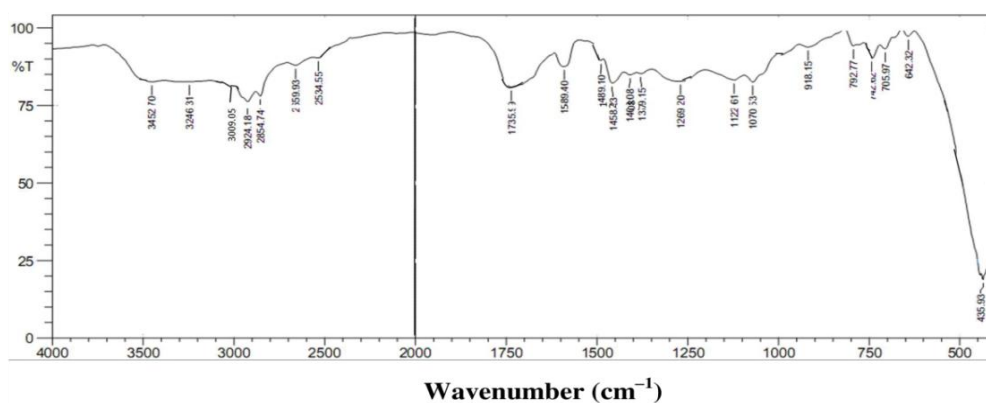**Figure 3:** Infrared spectrum of Lagenaria breviflorus short oil alkyd

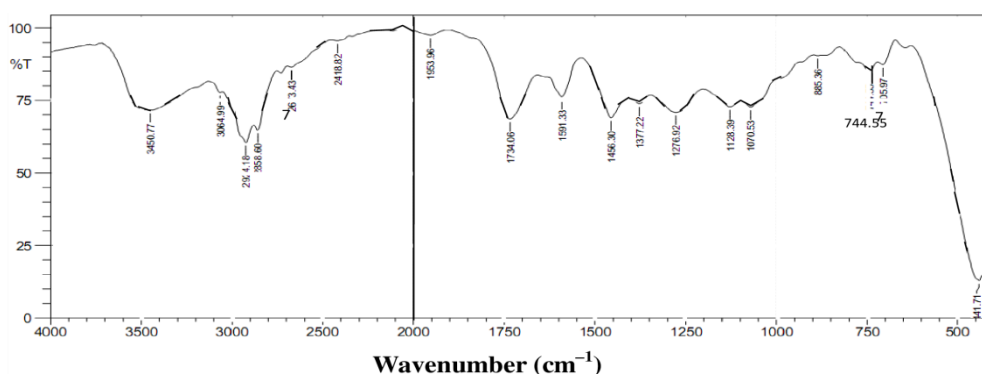**Figure 4**: Infrared spectrum of Lagenaria breviflorus medium oil alkyd

Figure 5: Infrared spectrum of commercial alkyd resin

3.5. Physicochemical properties of the alkyd resins
Some physicochemical properties of the synthesized light yellow alkyd resins and commercial standard are presented in Table4. The acid value ranged from 5.1 to 10 mg KOH/g; iodine value (20.81 to 28.67 gI$_2$/100g); and non-volatile matter (98 to 98.3 %). The acid values of all the prepared alkyds fall below 15 mg KOH/g. The

low acid value of the alkyds is necessary to reduce corrosion on substrates coated with substances formulated with the finished alkyds [44]. The non-volatile organic matter of the prepared alkyd resins was high compared to the commercial sample. The iodine values of the prepared alkyd resins increase with increase in oil content. The trend is attributed to the level of unsaturation in the fatty acid chain as the oil content increases [25].

Table 4: Physiochemical properties of the prepared alkyd resins

Alkyds	Colour	Acid value (mg KOH/g)	Iodine value (g I_2/100 g)	Non-volatile organic matter (%)
LB-SOA	Light yellow	10.00	20.81	98.30
LB-MOA	Light yellow	5.10	28.67	98.00
COA	Light yellow	–	47.71	52.90

LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd; COA = Commercial alkyd

3.6. Performance properties of the alkyd resins

The results of the solubility and chemical resistance tests for the alkyd resins and the commercial standard are shown in Table5.All the alkyd resins were soluble in the three solvent media: acetone, toluene and xylene within 5 minutes. It was also observed that the short oil alkyds took a longer time (5 minutes) to dissolve than the medium oil alkyd (4 minutes). The rate of dissolution of the alkyd resins was observed to decrease with increased viscosity. Short oil alkyds are known to have higher viscosity due to higher extent of polymerization, which necessitates swelling before dissolution [45]. The alkyd films showed resistance to distilled water, aqueous sodium chloride and dilute H_2SO_4 solutions. However, the prepared alkyd samples showed poor resistance to sodium hydroxides solution (alkali medium). This tendency is likely due to the basic hydrolysis of the ester linkages in the alkyd resins [46].

Table 5: Performance properties of alkyd resins

	Solubility			Chemical resistance			
Alkyd	Toluene	Acetone	Xylene	Distilled water	0.1 M NaOH	0.1 M H_2SO_4	5% NaCl
LB- SOA	s	s	s	n	b	n	n
LB-MOA	s	s	s	n	b	n	n
COA	s	s	s	n	n	n	n

s= soluble, b= blistering of film, n = no effect on film; LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd; COA = Commercial alkyd

The drying properties of the prepared alkyd resins on wooden and metallic substrates are presented in Tables 6 and 7 respectively. The drying time is comparable with other oil-modified alkyd resins [25]. Generally, the set-to-touch, surface dry and dry-through time for the alkyds increased with oil content. This could be linked to the degree of cross-linkage, which is highest for the short oil alkyd and reduces towards the long oil when subjected to same manufacturing conditions [15]. The differences observed on the drying performances of the alkyds indoor and outdoor are evidences to the fact that sunlight, temperature and volume of oxygen, influence the drying properties of alkyd resins [25]. The nature of substrate also affects the drying performance of alkyd resins. According to Yadav *et al.* [47], the presence of some polar functional groups (OH, COOH and COO⁻) on wood surface similar to those on the alkyd resin, helps to enhance adhesion and by implication improve the dry-through property of the alkyd resins.

Table 6: Drying properties on wooden substrate

Alkyd	In-door (26 – 28 ℃± 2 ℃)			Out-door (28 – 31 ℃ ±2 ℃)		
	Set-to-touch (min)	Surface dry (h)	Dry through (h)	Set-to- touch (min)	Surface dry (h)	Dry through (h)
LB-SOA	35	3.0	8.5	25	2.5	5.0
LB-MOA	45	4.0	9.0	35	3.0	5.5
COA	40	3.5	8.5	30	3.0	5.0

LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd; COA = Commercial alkyd

Table 7: Drying properties on metallic substrate

Alkyd	In-door (26–28 ℃± 2 ℃)			Out-door (28–31 ℃ ±2 ℃)		
	Set- to – touch (min)	Surface dry (h)	Dry through (h)	Set- to- touch (min)	Surface dry (h)	Dry through (h)
LB-SOA	50	5.0	9.5	35	3.0	8.5
LB-MOA	60	5.5	10.5	45	3.5	9.0
COA	50	5.5	10.0	40	3.5	9.5

LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd; COA = Commercial alkyd

Table 8: Pencil hardness of alkyds

Alkyd	Hardness	
	Scratch	Gouge
LB-SOA	4H	5H
LB-MOA	3H	4H
COA	4H	6H

LB-SOA = L. breviflorus short oil alkyd; LB-MOA = L. breviflorus medium oil alkyd;

Table 8 shows both scratch and gouge hardness of the prepared alkyd samples. Both scratch and gouge hardness were observed to increase with decrease in oil content. This shows that hardness is influenced by the drying time of the resins; the faster the drying, the harder the film [25].

Conclusions

Oil was extracted from*L. breviflorus* seed and characterized using standard procedures. The oil obtained was used in the synthesis of short and medium oil alkyd resins.The results of the study reveal that the underutilized and non-conventional vegetable oil from *L.breviflorus*could be a potential raw material for the coatings industry. Some properties of the synthesized alkyd resins compared well withthe commercial standard. Hence, *L. breviflorus*seed oil is suitable for the formulation of alkyd resins.It could be blended with fast drying resins to impart gloss, flexibility and to reduce cost. Chemical modification by co-polymerization with styrene, for example, may also be necessary to enhance performance; since it is known that polystyrene is very resistant to the action of alkalis both in alcoholic and non-aqueous media. This may lead to products that could meet up with a wide range of applications.

References

1. Oridupa O.A., Saba A.B., Suleiman L.K., *Trop. Veterinary* 29 (2011) 22.
2. Dutta A.C., Botany for Degree Students. 6th ed., Calcutta India: Oxford University Press, pp. 89-99, 1995.
3. Umerie S.C., Onuagha M.C., Nwobi S.C., *Nat. Appl. Sci. J.* 34 (2009) 104.
4. Kar A.,Pharmacognosy and Pharmacobiotechnology. 2nded., New Delhi, India: Age International (P) Limited Publishers, pp. 60-90, 2007.
5. Bognar J.R., Kovacs L., Szabo L.,Hungarian Patent 55(1991) 421, Chem. Abstract, 115: 210377X.
6. Athawale V.D., Chamankar A.V., Athawale M., Alkyd Ketonic Blends for Coating Applications, Paint India 2000, pp. 39-44.
7. Aigbodion A.I., Okieimen F.E.,*Eur. Polym. J.*32 (1996) 1105.
8. Wang C., Lin G., Pae J., Jones F.N., Ye H., Shen W., *J. Coat. Technol.* 72 (2000) 55.
9. Talaqani T.E., Shafik J., Mustafa .F.K., *Ind. J. Agric. Chem.* 19 (1986) 147.
10. Akintayo E.T., Bayer E., *Bioresour. Technol.* 85 (2002) 95.
11. Essien E.E., Udo I.I., Ogunwande I.A., *Int. J. Bio.Pharm. Allied Sci.* 10 (2013)1845.
12. Ezeh I.E., Umoren S.A., Essien E.E., Udoh A.P., *Ind. Crops Prod.* 36 (2012) 94.
13. Ibrahim K.A., Abu-sbeih K.A., Al-Trawneh I., Bourghli L., *J. Polym. Environ.* 22 (2014) 553.
14. Aigbodion A. I., Okieimen F. E. (2001). *Ind. Crops Prod.* 13 (2001) 29.
15. Okieimen F.E., Aigbodion A.I., *Ind. Crops Prod.* 6 (1997) 155.
16. Odetoye T.E., Ogunniyi D.S., Olatunji G.A., *Ind. Crops Prod.* 32 (2010) 225.
17. Assanvo E.F., Gogoi P., Dolui S.K., Baruah S.D., *Ind. Crops Prod.* 65 (2015) 293.
18. Bora M.M., Gogoi P., Deka, D.C., Kakati D.K., *Ind. Crops Prod.* 52 (2014) 721.
19. Bora M.M., Deka R., Ahmed N., Kakati D.K., *Ind. Crops Prod.* 61 (2014) 106.
20. Meshram P.D., Puri R.G., Patil A.L., Gite V.V. *Prog. Organ. Coat.* 76 (2013) 1114.
21. Weiss K.D., *Prog. Polym. Sci.* 22 (1997) 203.
22. Gunstone F.D., Fatty Acid and Lipid Chemistry. Chapman and Hall, London, pp. 61–81. 1996.
23. Morrisson W.R., Smith L.M., *J. Lip. Res.* 5 (1964) 600.
24. AOAC, Official Methods of Analysis of the Association of Official Analytical Chemists. 17th ed., Washington DC, pp. 500-540, 2000.
25. Isaac I.O., Ekpa O.D., *Am. J. Polym. Sci.* 3 (2013) 13.
26. ASTM D1639-90, Annual Book of American Society for Testing and Material Standards. Philadelphia, PA; 0601, pp. 260–261, 1994.
27. ASTM D1647-89, Annual Book of American Society for Testing and Material Standards. Philadelphia, PA; 06, pp. 268–269, 1994
28. Patel V.C., Varughes J., Krishnamoorthy P.A., Jain R.C., Singh A.K., Ramamoorty M., *J. Appl. Polym. Sci.* 107 (2008) 1724.
29. Vibhute P. B. (2010) Novel utilization of non-exploited plant seed oils in coating applications. A Paper Presented at the 15th International Coating Science and Technology Symposium held at Nagpur, India, pp. 45-53.
30. Igwe I.O., Ogbode O., *J. Appl. Polym. Sci.* 78 (1999) 1826.
31. Ikhuoria E.U., Aigbodion A.I., Okieimen F.E., *Trop. J. Pharm. Res.* 3 (2004) 311.
32. Ogunniyi D.S., Odetoye T.E., *Bioresour. Technol.* 99 (2008)1300.
33. Kyenge B.A., Anhwange B.A., Ageh J.T., Igbum G.O., *Int. J. Mod. Org. Chem.* 2 (2012) 66.
34. Shaker N.O., Alian N.A., El-sawy M.M., *Der Chemica Sinica* 3 (2012) 1157.
35. Olaofe O., Ogungbenle H.N., Akhadelor B.E., Idris A.O., Omojola O.V., Omotehinse O. T., Ogunbodede O.A., *Int. J. Bio. Pharm. Allied Sci.* 1 (2012) 355.
36. Alkhalifa A.S., *J. Agri. Food Chem.* 44 (1996) 964.
37. Aswari B., Khemnani S., Arora A., Sinda R.S., *Int. J. Basic Appl. Chem. Sci.* 1 (2011) 2.

38. Borhade S., *Life Sci Leaflets* 7 (2012) 45.
39. Chawla S. A., Textbook of Engineering Chemistry. 3rd ed., North Delhi: Dhanpat Rai and Co., pp. 657-680, 2010.
40. Irshad M., Ahmad I., Goel H.C., Rizvi M.M.A., *Res. J. Phytochem.* 4 (2010) 242.
41. Hlaing N.N., Oo M.M., *World Acad. Sci. Eng. Technol*. 48 (2008) 155.
42. Dhoke S.K., Sinha T.J., Dutta P., Khann A.S., *Prog. Org. Coat.* 62 (2007) 183.
43. Silverstein M. R., Webster F. X., Kiemle D. J., Spectrometric Identification of Organic Compounds. Hoboken: John Wiley Sons Inc., pp. 78-108, 2005.
44. Menkiti M.C., Onukwuli O.D., *New York Sci. J.* 4 (2011) 51.
45. Bikales N. M., Characterization of Polymers. New York: Wiley Interscience, pp. 91-147, 1971.
46. Mahapatra S.S., Karak N., *Prog. Org.Coat.* 51 (2004) 103.
47. Yadav S., Zafar F., Hasnat A., AhmadS., *Prog. Org. Coat.* 64 (2009) 27.

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