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Preparation and Comparative Analysis of Neem and Dehydrated Castor Seed Oil Alkyd Resin Paint Coatings

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ABSTRACT

Coatings such as paint are made up of four important components; pigments, binders, solvent and additives. Gloss paint also known as oil paint used majorly alkyd resin as its binder. In this study, medium oil length alkyd resins were synthesized from Neem (*Azadirachta indica*) and dehydrated Castor (*Ricinus communis*) seed oils by alcoholysis. The alkyd resins were used to prepare gloss paints and their final properties were compared. The two seed oils were extracted with n-hexane in a soxhlet apparatus and their physico-chemical properties were determined. The acid values were 12.69 mgKOH/g and 12.64 mgKOH/g for castor and neem seed oil, respectively. The saponification and iodine values were 184.30 mgKOH/g and 97.61 gI_2/100g, respectively for the castor seed oil, while the neem seed oil has saponification and iodine values of 192.35 mgKOH/g and 84.20gI_2/100g, respectively. After dehydration, castor oil has iodine value of 131.00 gI_2/100g which made it a drying oil. The Fourier Transform Infra-Red (FTIR) spectroscopic analysis of the medium oil length alkyd resins from dehydrated castor and neem seed oils revealed the same ester functional group at 1722.00 cm^{-1}. The absorption at 2926.00 $cm^{-1}$and 2922.20 cm^{-1} for dehydrated castor and neem seed alkyd resin, respectively showed aliphatic chain of their polyesters. The dehydrated castor alkyd paint has drying-through time of about 24 hours while the neem seed oil alkyd paint has drying-through time greater than 48 hours. It was discovered that the dehydrated castor seed alkyd resin paint showed better properties than neem seed oil alkyd resin paint.

Keywords: Physico-chemical properties, Alcoholysis, Monoglycerides, Alkyd resins.

Introduction

Coatings such as paints are described as any liquid, liquefiable, or mastic composition which after application to a substrate in a thin layer is converted to an opaque solid film. It is most commonly used to protect, colour or add texture to objects.

Coatings such as paint can be dated to the prehistoric times. For instance, paint was made more than 35000 years ago by prehistoric man as they mixed clays and chalks with animal fats and used for paints to depict their hunts on cave walls. By 2500 BC the Egyptians improved on this technology considerably. Today, coatings found applicable in various areas of life.

These coatings are generally made up of four components; pigments, resins or binder, solvent and additives.[1] Gloss paint which is also referred to as oil paint used majorly alkyd resin as its binder. The binder serves as vehicle which holds the pigment and other ingredients in the coating composition.[2] Alkyd resins are by far the most important class of coatings resins and are the most common type of resins used in "oil-based" paints. The popularity of alkyd resins as vehicle for coatings is largely due to their special qualities which are film hardness, durability, gloss and gloss retention, resistance to abrasion, impacted on them through modification with drying oil.[3] Thus, alkyd resins have then become indispensable raw material used for the production of industrial household finishes.[4]Alkyd resins are product of polycondensation reaction between polybasic acid and polyhydric alcohol modified with fatty acid or drying oil. The term alkyd is a modification of the original name "alcid", reflecting the fact that they are derived from alcohol and organic acids.[5] The alkyd synthesis reaction is considered to be the most versatile resin-forming reaction known due to the way the reaction can be modified to improve the resin properties. Alkyd resin is a synthetic resin which has a dominant position among the synthetic resins with respect of production volume and the frequency of the use in paint and varnish materials. In spite of the growing popularity of acrylic, polyurethane and epoxy resins, alkyd resins is highly preferred among paint producers for its variability of compositions and excellent qualities. No other resin lends itself to greater internal variation or to more useful modification by physical or chemical blending with other polymers as alkyd resins.[6] It is estimated that alkyd resins contribute about 70% of the binders used in surface coatings today and the properties of the finished alkyd resin coating depends so much on the nature of the drying oil especially the drying property of the oil.[7] In view of the demand for oils that are gradually increasing with growing world population there is the need for more research to be carried out towards exploring new sources for industrial oil and expanding the present oil supplies that are being used in the synthesis of alkyd resins.[4]

Neem seed oil is obtained from Neem plant (*Azadirachta indica*). The tree is a native of South East Asia and is a member of the Mahogany family *Meliaceae*.[8] Neem tree is popularly known in Nigeria as *Dongoyaro.* The neem tree is noted for its drought resistance. Normally, the plant thrives in an area with annual rainfall between 400-1,200 millimetres (16-47 in). It can also grow in regions with annual rainfall below 400 mm. Neem grows in different types of soil and can tolerate high to very high temperatures but cannot survive in temperature below 4^0C (39ºF). Nigeria climatic conditions favour the growth of neem and are grown in several parts of the country including Zaria in Kaduna State. All parts of this tree are very useful due to their biological activities and the most economic part of neem tree is the oil obtained

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from the kernel of its seed. Neem seed oil has been used in various parts of the globe for medicinal and agricultural purposes.[9] This plant oil has been employed in the production of various items such as soap, commercial pesticides and cosmetics, lamp oil, candle production and lubricating oil but with little or no report on its use in organic coatings. [10]

The castor oil plant, *Ricinus communis,* is a species of flowering plant in the spurge family, *Euphorbiace.* It belongs to the genus *Ricinus*, and subtribe, *Riciniae*. Castor (*Ricinus communis)* like neem plant is also an important drought-resistant shrub which is believed to be native of Ethiopian region of tropical Africa and has become naturalized in tropical and temperate regions throughout the world. Castor seed which is often refers to as castor bean (*Ricinus communis L.)* is the source of castor oil. The demand for Castor oil has grown in international market because of its over 700 applications which include medicines, cosmetics, production of biodiesel, plastic, and lubricants.[11]

Neem seed oil has been widely reported to have oil content varying between 43.48 – 46.38% and castor seeds even though they are of different varieties, but on the average, they contain about 46–55% oil by weight depending on the variety of the seeds and the methods of extraction.[12-14] Since plant oil is one of the vital reagents in the synthesis of alkyd resin, the high oil content of neem and castor seeds justify their choice as the modifying oil in the synthesis of alkyd resins which will ultimately be used in preparation of paint coating. Also, several plant oils have been used in the synthesis of alkyd resins with little or no publications on the performance of their resins in final coating. In this research work, neem and dehydrated castor seed oil were used to synthesize medium oil length alkyd resins which were later used to prepare gloss paints and comparative analysis of their coatings were carried out.

Materials and Methods

Chemicals and plant materials

All chemicals reagents used for this work were of analytical grade and were used without further purification except otherwise stated. The castor and neem seeds were obtained from Basawa town in Zaria, Sabon Gari, Local government of kaduna State in Nigeria. The seeds were identified in the herbarium at the Department of Botany, Faculty of Life Sciences, Ahmadu Bello University, Zaria, Kaduna State, Nigeria.

Extraction of Castor and Neem Seed Oils

The castor and neem seed oil were extracted using solvent extraction method using soxhlet apparatus and n-haxane (extraction solvent). The washed, oven dried seeds were ground and filled into the thimble of the soxhlet apparatus and the solvent (n-hexane) of the flask of the apparatus was heated to temperature of 60^{o}C for five hours and thereafter allowed to cool. The collected extract was heated and evaporated using rotary evaporator to obtain solvent-free oil.

Physico-chemical Properties Neem and Castor Seed Oils

The physico-chemical properties which are the specific gravity, colour, acid value, saponification value and iodine value were determined using AOAC standards method.[15]

Preparation of Dehydrated Castor Seed Oils

The extracted castor seed oil was dehydrated in a round bottom flask with 2 % (wt %) of $NaHSO_4$ as catalyst. [3] The flask containing the oil was connected to a condenser and it was heated to temperature of about 180^{0}C under vacuum and the dehydration time was 75 minutes. The physico-chemical properties especially the iodine value of the dehydrated castor oil was carried out using ASTM standard methods. [16]

Preparation of the Neem and Dehydrated Castor Seed Oils Alkyd Resins

The alkyd resin was prepared using alcoholysis method using the above-mentioned oils with glycerol and phthalic anhydride while Calcium (II) oxide was used as catalyst. The synthesis was carried out in two stages. Medium oil length alkyd resins were prepared with the dehydrated castor and neem seed oils in three-necked flask fixed with mechanical stirrer and nitrogen inlet (to create an inert environment). The recipe for the synthesis of the medium oil length dehydrated castor and neem seed oil alkyd resins was as shown in Table 1. The duration of the reactions and the water of esterification liberated are also shown in Table 1.

Table 1: Recipes for the Preparation of Medium Oil Length Dehydrated Castor and Medium Neem Seed Oil Alkyd Resins

S/N	Reagents (g)	MDCOA	MNSOA
1.	Dehydrated castor/Neem Seed Oil	214.62	210.12
2.	Phthalic Anhydride	143.41	142.95
3.	Glycerol	91.97	96.93
	TOTAL	450.00	450.00
4.	Duration of Monoglyceride	3 h 55 mins	4 h 35 mins
5.	Duration of Polyesterification	6 h 10 mins	6 h 45 mins
6.	Liberated Water of Esterification (g)	17.00	16.00

MDCOA: Medium Oil Length Dehydrated Castor Resin
MNSOA: Medium Neem Seed Oil Alkyd Resin

Stage 1 (Alcoholysis): This involved the preparation of monoglyceride by reacting the glycerol with Dehydrated castor and neem seed oils at a temperature of about 230-250^{o}C. The reaction was catalyzed by CaO. Alcoholysis was considered completed when the solubility test in methanol was positive; when one part of the reaction mass was soluble in three parts of methanol. The reaction mixture was cooled to about 140^{o}C.

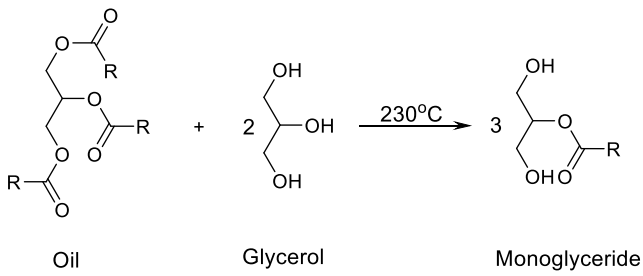

Figure 1: Alcoholysis Reaction Scheme.

Stage 2 (Polyesterification): In this stage, phthalic anhydride was added to the monoglyceride mixture. The temperature was maintained at 230-250^{o}C. Xylene was also added at this stage as azeotropic solvent. The xylene is usually about 10% of the reaction mass. The Dean and Stark apparatus which has an inverted U-shape helps in removing the water of esterification. The progress of the reaction was monitored by periodic determination of the acid value of the mixture until the acid value dropped to about 10.[6] The water of esterification of the in-process alkyd resins was collected and the amount recorded for the two alkyd samples.

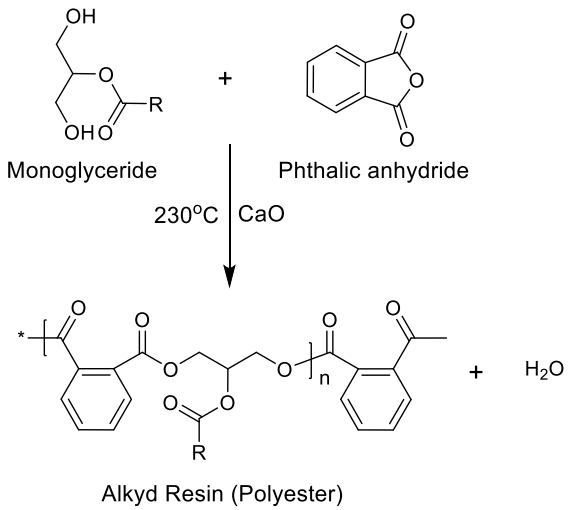

Figure 2: Polyesterification Reaction Scheme.

Determination of Physico-Chemical Properties of the Alkyd Resins
The physico-chemical properties such as colour, acid value, saponification value, iodine value and equivalent molecular weight of the alkyd resins were determined using the American Society for Testing and Material (ASTM) standard methods.[16]

FT- IR Analysis of the Alkyd Resins Functional Groups
Fourier Transform Infrared Spectrophotometer (FT-IR) was used to analyze the functional groups present in the dehydrated castor seed oil (DCO) and neem seed oil (NSO) alkyd resins.

Performance Characteristics of the Finished Alkyd Resins in Different Media
The resistance of the alkyd resins in different media (water, acid, alkali and brine) was carried in order to ascertain the durability of the alkyd resin films in these media.[17]

Preparation of Dehydrated Castor and Neem Seed Oil Alkyd Resins Paints
Alkyd resins gloss paints were prepared using dehydrated castor and neem seed oil alkyd resins and other reagents. The detailed recipe and the functions of the reagent used were given in Table 2. The alkyd resin paints were prepared using standard method in a reaction flask with electric motor stirrer with revolution of about 750 per minute. The reagents were added in the order given in Table 3.2 and the flask content was thoroughly stirred until homogeneous mixture was obtained before subsequent addition. The stirring continue for about 20 minutes after all the reagents were added to obtain the finished paint. The paint samples were allowed to settle for about 24 hours and thereafter it was analyzed.

Determination of the Physico-Chemical Properties of the DCOA AND NSOA Paints

Specific Gravity Test: The specific gravity of the paint sample was carried out using standard method. The empty specific gravity (SG) bottle was weighed and its weight recorded. The SG bottle was later filled with water and the weight taken. The same SG bottle was filled with equal volume of DCOA and NSOA paints and their weights were recorded. The specific gravity was calculated as the ratio of the weight of the paints to an equal volume of water.

Opacity Test: The opacity test was carried out as reported by Uthman in 2011[18] and this test was done by coating 20 cm^2 surfaces with the paint samples. The covering and the hiding power of the paints were observed visually.

Drying Time: The drying time was determined using ASTM D1640 standard method of Drying, Curing, or Film Formation of Organic Coatings at room temperature. [19] The following tests were carried out.

Set-to-touch time: In this test, microscopic slides were coated with the DCOA and NSOA paints and in each case the film was lightly touched with the tip of a clean finger, and the finger-tip was placed against a piece of clean, clear glass to determine when the film does not adhere to the finger or transfer to the clean glass.

Dust-free time: This was done by dropping absorbent cotton fibres from a height of 25 mm on the coated slides. The coated paint films were considered to be dust free when the cotton fibres can be lightly blown off from the coated slides.

Dry-through (dry-to-handle) time: The test coated slides were placed in a horizontal position at such a height that when a thumb was placed on the paint film, the right thumb was used to press down the paint film exerting moderate pressure and at the same time twisting the thumb through an angle of 90°. The test film was considered dry through when the film was not distorted by bearing down with moderate thumb pressure and twisting through angle 90°.

Determination of flash point
Flash point is the lowest temperature at which a heated substance ignites on application of flame. The flash point was determined using Cleverland open cup method according to ASTM D9266. The test cup was filled with 20g of the sample. The temperature of the sample was increased rapidly at first and then at a slow constant rate of about 5ºC using a hot plate as the flash point was approached. A fixed thermometers' bulb was immersed in the sample but not touching the base of the cup. At specified intervals a small test flame was applied to the cup with the aid of an igniter. The lowest temperature at which application of the test flame causes the vapors above the surface of the sample to ignite was taken as the flash point.

Results and Discussion

Physico-chemical Properties of Neem and Castor Oil
The physico-chemical properties of the extracted neem and castor seed oils were given in Table 3. The colour of the neem oil was light brown while that of the castor oil was colourless. The acid and saponification values of the two oils were also as given in Table 3. The acid value of 12.64 mg KOH/g for neem and 12.69 mg KOH/g for castor oil was a little lower than that of rubber seed oil used for similar alkyd resin preparation.[7] The saponification value of 192.35 mg KOH/g and 184.30 mg KOH/g for neem and castor oil respectively revealed that the two plant oil were made up of normal triglyceride which makes them suitable for the preparation of alkyd resins.[20] However, the iodine values of 84.20 gI_2/100g and 97.61 gI_2/100g showed that both oils are non-drying.

Table 2: Formulation for Preparing Dehydrated Castor and Neem Oil Alkyd Resin Paints

S/N	Chemical Reagents	Functions of Reagents	DCOAP (g)	NSOAP (g)
1.	DCO/NSO Alkyd resins	Binder	80.00	80.00
2.	Kerosene	Solvent	50.00	50.00
3.	Titanium dioxide (TiO_2)	Opacifier/white pigment	8.00	8.00
4.	Pigments	Yellow colouration	6.00	6.00
	Yellow pigment	Red colouration	3.00	3.00
	Red pigment			
5.	Texanol (3-Hydroxyl-2,2,4-trimethylpentyl, 2-methyl propanoate)	Leveling and surface flow additive	2.00	2.00
6.	Anti-skin (Methyl Ethyl Ketone oxime); $CH_3C(NOH)CH_2CH_3$	Prevention of surface skin formation/peeling off of paint on the substrate	2.50	2.50
7.	Drier (Multivalent organometallic)	Acceleration of drying through auto oxidation reaction	6.00	6.00
8.	Preservative (Vinkocide); 5-chloro-2-methyl-2H-isothiazol-3-on	Prevention of algae and fungi growth on the paint; in-can and after application	2.50	2.50

DCO = Dehydrated Castor Oil; NSO = Neem Seed Oil; DCOAP = Dehydrated Castor Oil Alkyd Paint; NSOAP = Neem Seed Oil Alkyd Paint.

Physico-chemical Properties of Dehydrated castor seed oil (DCO)
Table 4 showed that the physico-chemical properties of the dehydrated castor were similar to the extracted castor oil except for the iodine value that has increased to 131.00 gI_2/100g which made the dehydrated castor oil a drying oil suitable for the preparation of paint coatings. It has been reported that castor seed oil is the only commercial source of hydroxylated fatty acid specifically Ricinoleic acid[21] and during dehydration the hydroxyl group is with hydrogen atom as water molecule creating double bond which increase the iodine value of the dehydrated castor seed oil. The dehydrated castor oil is considered a drying oil at this point because it has iodine value greater than 130 gI_2/100g.

Physico-Chemical Properties of the Neem and Dehydrated Castor Seed Oil Alkyd Resins
The physico-chemical properties of the neem and dehydrated castor seed oil are very similar as shown in Table 5. The acid value of both resins was less than 10 mg KOH/g which revealed a very high percentage extent of reaction during synthesis.[22] The higher Saponification value of the two oil alkyd resins in comparison with their respective oil is an indication that the free fatty acids of the oils took part in the formation of the alkyd resins. The iodine value of 80.37 gI_2/100g for dehydrated castor seed oil alkyd resin and 67.98 gI_2/100g for neem seed oil alkyd resin showed that the dehydrated castor seed oil alkyd will exhibit better drying properties than the neem seed oil alkyd resin.

Table 3: Physico-chemical Properties of Neem and Castor Oil.

Physico-chemical Properties	Neem Seed Oil	Castor Seed Oil
Colour	Very light Brown	Colourless
Specific gravity	0.935	0.960
Acid value (mg KOH/g)	12.64	12.69
Saponification value (mg KOH/g)	192.35	184.30
Iodine value (gI_2/100g)	84.20	97.61
Average Molecular Weight (Mw)	291.66	304.40

Table 4: Physico-chemical Properties of Dehydrated Castor Seed Oil (DCO).

Physico-chemical Properties	Castor Seed Oil	Dehydrated Castor Oil
Colour	Colourless	Dark Brown
Specific gravity	0.960	0.934
Acid value (mg KOH/g)	12.69	33.75
Saponification value (mg KOH/g)	184.30	189.40
Iodine value (gI_2/100g)	97.61	131.00
Average Molecular Weight (Mw)	304.89	296.20

Table 5: Physico-Chemical Properties of the Neem and Dehydrated Castor Seed Oil Alkyd Resins DCO Alkyd Resins.

Physico-chemical Properties	MDCOA	MNSOA
Colour	Dark Brown	Dark Brown
Specific gravity	0.94	0.93
Acid value (mg KOH/g)	5.61	4.53
Saponification value (mg KOH/g)	277.80	254.60
Iodine value (gI_2/100g)	80.37	67.98
Average Molecular Weight (Mw)	201.94	220.35

FTIR Spectra of Dehydrated Castor Oil (DCO) and Neem Seed Oil (NSO) Alkyd Resins
The FTIR spectra for the DCO and NSO alkyd resins are shown in Figure 3 and 4 respectively. Both alkyds have similar absorption bands of polyester. The detailed analysis of the functional groups present in the resins and their sources are as given in Table 6.

Table 6: Functional Group Analysis of the FTIR Spectra of DCO and NSO Alkyd resins

S/N	ALKYD RESIN SAMPLE	WAVE NUMBER (Cm^{-1})	LITERATURE VALUES	ASSIGNED GROUP	SOURCE
		3444.1	3570-3200[3]	-OH	Hydroxyl from Hydroxyl excess of □, ß-Hydroxyl groups of the glycerol.
1.	MDCOA	2926.0, 2855.1	2926-2850[3]	$-CH_2$, CH_3	Aliphatic chain of the alkyd resin
		1722.0	1730-1717[3]	-COO-	Ester group of the alkyd resin
		1453.7	1485-14402[23,24]	CH_2	Aliphatic chain of the alkyd resin
		741.7	1000-600[24]	-CH	Out-of-plane bending vibration of the C-H bonds of unsaturated groups of the alkyd aliphatic chain.
2.	MNSOA	3444.1	3570-3200[3]	-OH	Hydroxyl from Hydroxyl excess of □, ß-Hydroxyl groups of the glycerol.
		2922.2, 2855.1	2926-2850[3]	$-CH_2$, CH_3	Aliphatic chain of the alkyd resin
		1722.0	1730-1717[3]	-COO-	Ester group of the alkyd resin
		1457.4	1485-1440[3]	CH_2	Aliphatic chain of the alkyd resin
		741.7	1000-600[23,24]	-CH	Out-of-plane bending vibration of the C-H bonds of unsaturated groups of the alkyd aliphatic chain.

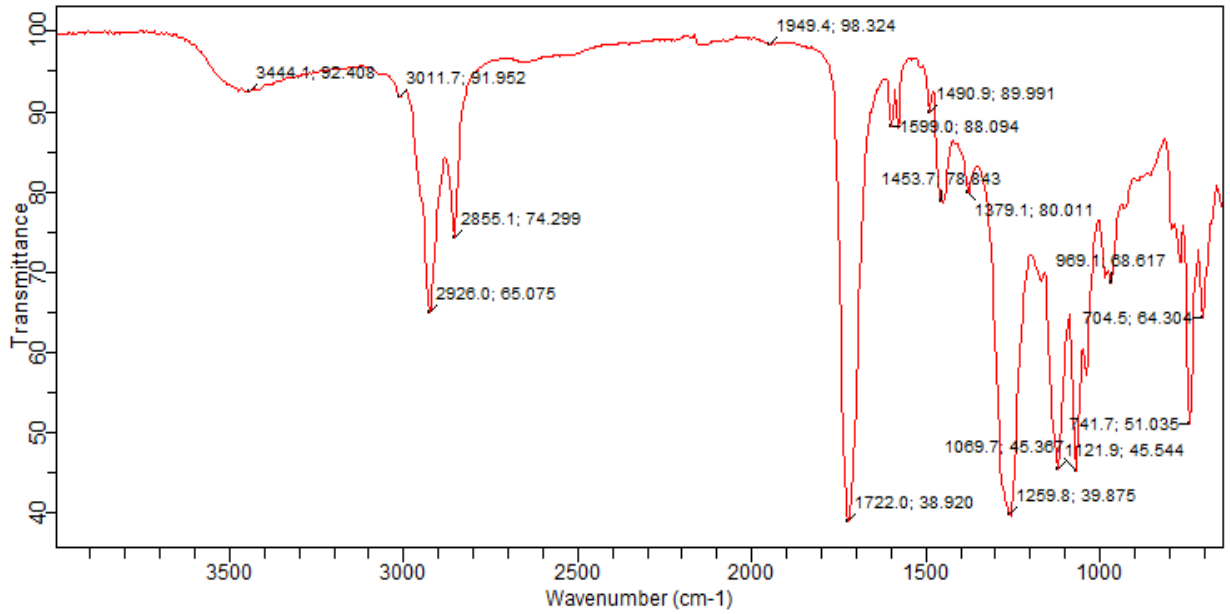

Figure 3: FTIR Spectra of Medium Dehydrated Castor Seed Oil Alkyd Resin (MDCOA).

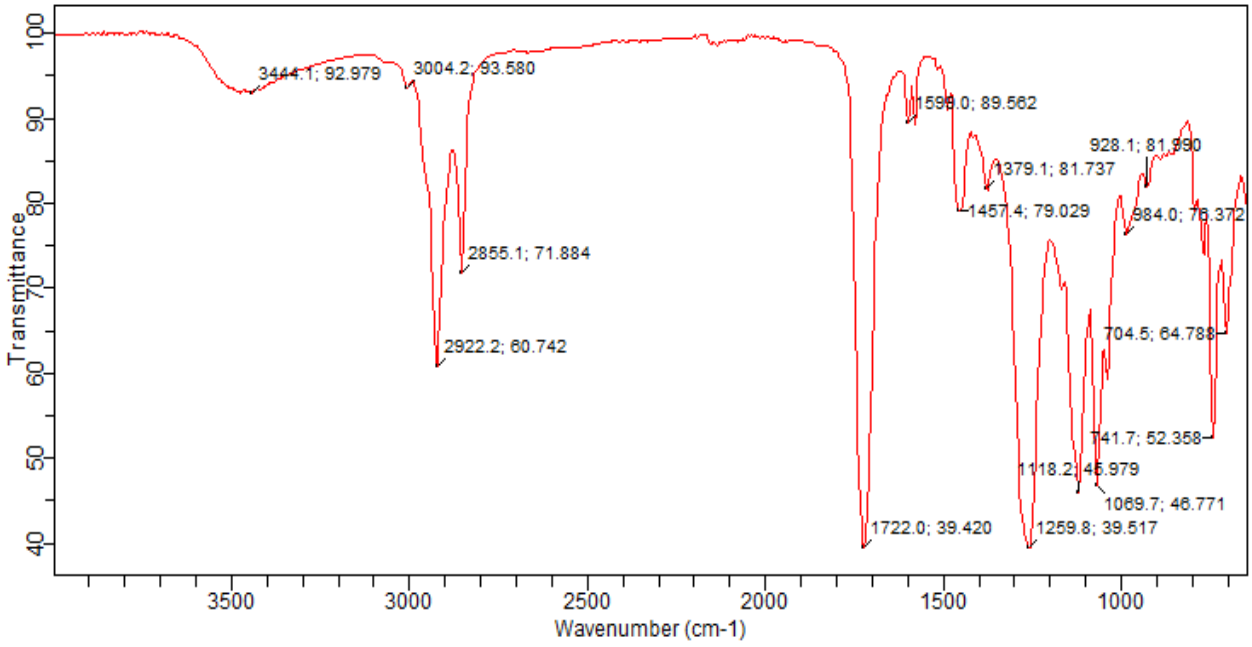

Figure 4: FTIR Spectra of Medium Neem seed oil Alkyd Resin (MNSOA)

Media Resistance of Alkyd Resins Samples

The coated microscopic slide with DCO and NSO alkyd resins exhibited excellent media resistance in brine, acid solution and cold water. However, in 0.1 M NaOH alkaline solution, the dehydrated castor seed oil alkyd exhibited resistance for some hours before blistering and later washed overnight while the coated NSO alkyd was blistered after few hours before being washed. The performance of the two alkyd resins in different media as shown in Table 7 was similar to that of Rubber seed oil alkyd resins reported by Ikhuoria *et al*, 2004.[18]

Properties of the Dehydrated Castor Seed and Neem Seed Oil Alkyd Resin Paints

The properties of the dehydrated castor seed and neem seed oil alkyd resins paints were given in Table 4.6. The two alkyd resins give paints with good properties similar to that of the paint from alkyd resin derivative of *ximenia americana* (wild olive) seed oil.[1]

The preparation of pink colour paint was successful with the dehydrated castor and neem seed oil alkyd resins and this shows that other colours of paint can easily be prepared with the resins with little pigment concentration. The specific gravity of the paints was 1.14 and 1.12 for DCOA and NSOA paints respectively, indicating that the paints can be applied by conventional painting techniques (roller, brush and spray painting). The hiding property and the opacity of both paints were very good when applied on the test surface. The NSOA and DCOA paints displayed hiding property similar to that of polyvinyl acetate (binder) texcoat paint in that the substrate was completely invisible to the eye. [20] The flash points of the two sample paints revealed that they are both non-flammable and can be readily stored and transported. However, the dry-through time of NSO alkyd paint was greater than 48 hours which seems to be too long but when its paint was applied as primer coatings and they were top coated after 48 hours of its application, it dried-through within 24 hours, the final coating dried normally and exhibited better gloss property. This therefore means that neem seed oil alkyd resins can be used in the preparation of primer coatings (wood varnishes and anti-rust paints). Neem seed oil alkyd resins can also find usage in products where non-drying alkyd is needed.

Table 7: Media Resistance of Alkyd Resins Samples

SOLUTIONS	MDCOA	MNSOA
Brine (5%, w/w, NaCl)	EXCELLENT	EXCELLENT
Alkali (0.1 M NaOH)	FAIR	POOR
Acid (0.1M H_2SO_4)	EXCELLENT	EXCELLENT
Water (cold)	EXCELLENT	EXCELLENT

Table 8: Properties of the Dehydrated Castor Seed and Neem Seed Oil Alkyd Resin Paints.

PROPERTIES	DCOAP	NSOAP
Colour	Flame Red	Flame Red
Specific Gravity	1.14	1.12
Opacity	Good Coverage	Good Coverage
Flash point	125.0ºC	140.0ºC
Set-to-touch Time (Mins)	30	45
Dust-free Time (Mins)	120	300
Dry-Through Time	Overnight	>48 h

DCOAP = Dehydrated Castor Seed Oil Alkyd Paint;
NSOAP = Neem Seed Oil Alkyd Paint

Conclusion

Neem and castor oils were successfully extracted from neem and castor seeds and the castor seed oil was dehydrated to increase its iodine value to 131.00 gI_2/100g which made it a drying oil. The physico-chemical properties of the neem, castor and dehydrated castor seed oils were determined. The dehydrated castor and neem seed oils were used to synthesize alkyd resins which were later used as binder in preparing gloss paints. The paints from dehydrated castor and neem seed alkyd resins exhibited good properties however, paint from dehydrated castor oil alkyd resin paint dried faster than the neem seed oil alkyd resin paint.

Conflict of interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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