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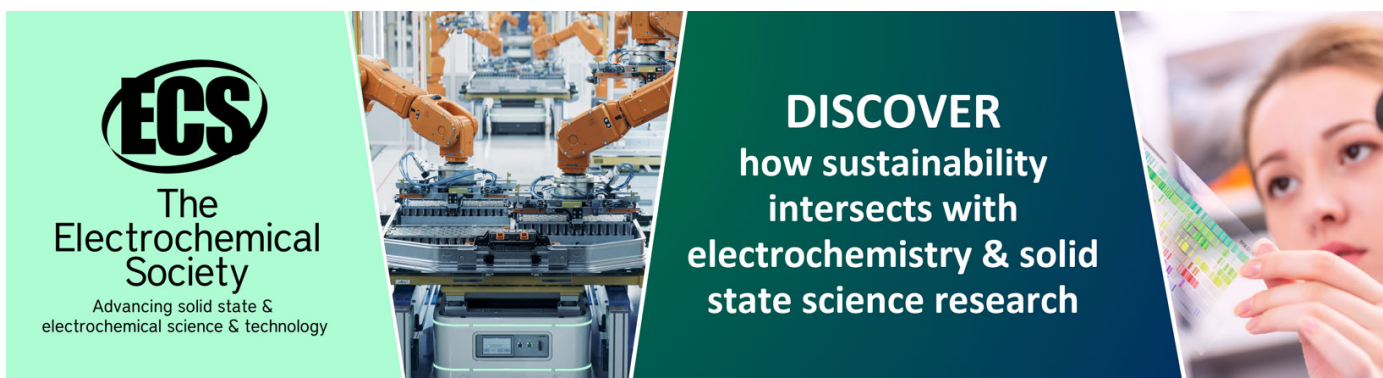
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Production and characterization of electrical transformer insulating fluid from blending Castor and Neem seed oils

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Abstract. Castor and neem seed oils were first purified separately through the process of acid degumming, neutralization, bleaching, filtering, washing and drying. The refined castor and neem seed oils were mixed thoroughly at a ratio of 40:60, 50:50 and 60:40 to make up weight of 100g each. The blended ratios were synthesized separately through epoxidation and transesterification reactions to satisfactorily remove glycerol and unsaturation and form an epoxy in the triglycerides of the mixture. The epoxidation process of the triglyceride significantly increased the viscosity whereby making the blended fluids more physically, chemically and thermally balanced. The blended oil esters produced were washed properly with distilled water and dried to purify the esters. The blended epoxy castor-neem oil methyl ester in ratio of 40:60, 50:50 and 60:40 (BEME46, BEME55 and BEME64) were characterized and the following results were obtained: the viscosities of BEME46, BEME55 and BEME64 at a lower temperature of 30°C were 9.30 cSt, 9.45 cSt and 9.48 cSt respectively, while the viscosities at a higher temperature of 70°C were 3.30 cSt, 3.32 cSt and 3.48 cSt respectively, using a glass capillary kinematic viscometer and hot water bath setup. The dielectric strength of BEME46, BEME55 and BEME64 were 30.55 kV, 30.75 kV and 32.02 kV respectively using a megger test set. The acid value or neutralization number of BEME46, BEME55 and BEME64 were 0.1683 mgKOHg⁻¹oil, 0.1405 mgKOHg⁻¹oil and 0.1405 mgKOHg⁻¹oil respectively. The densities were 0.8260 gcm⁻³, 0.8251 gcm⁻³ and 0.8219 gcm⁻³ respectively, while the flash points were 165°C, 168°C and 160°C respectively. BEME46, BEME55 and BEME64 were compared with Transformer Mineral oil obtained from APO Transmission station. It was revealed that the blended oil esters met the transformer standard specifications.

1. Introduction

Transformer oil plays an important role to keep distribution and power transformers active and operate efficiently. It keeps the transformer cool and works as an insulator between the transformer windings and body [11]. In power holding networks electrical insulation material like the transformer oil play a very essential role, they resist and prevent the conduction of electricity between two or more conducting materials [13]. It is suggested that synthesized ester is as good as mineral oil in many ways and a viable alternative fluid for cooling and insulating power transformers [1]. Mineral oil has shown over the century to be unreliable and not sustainable because of spill ups, high flammability which eventually leads to high transformer fire, high toxicity and less economic benefits [5][13][21]. In order to perform its function, electrical power transformers need an insulating liquid to function as a cooler and insulator [11]. There are a few characteristics that an insulating medium is required to have in order to function effectively in transformers. These characteristics include long term stability due to possible problems brought about by oxidation, contamination and excessive temperature which can affect the reliability of the equipment and the suitability of the oil in rheological aspect [11][21].



2. Materials and Methods

2.1. Materials

The materials and reagents were purchased from FIN Laboratory and Chemicals, Abuja. The materials used in the course of this study include the following: Citric acid ($C_6H_8O_7$, purity: 99.7%), Sodium sulphate anhydrous (Na_2SO_4), Hydrogen Peroxide (about 30% H_2O_2 GPR, assay 30-31%, BDH chemical ltd), Sodium hydroxide (NaOH, purity: 99.9%), Sulphuric acid (H_2SO_4 , purity: 99.8%) Silica gel, Glacial acetic acid (CH_3COOH , purity: 99.8%), Toluene ($C_6H_5CH_3$, purity: 99.7%), Methanol (CH_3OH , purity: 99.8%), Filter paper (24cm, medium crystalline; Whatman, England), Phosphoric acid (H_3PO_4 , purity: >85%), Oxolane (THF), Distilled water (purified in SHESTCO), Crude castor seed oil (purchased from NARICT, Zaria), Crude Neem seed oil (purchased from NARICT, Zaria), Coconut shell activated carbon (SHESTCO).

2.2. Crude Seed Oil Refining

The process steps for separately refining crude castor seed oil and crude neem seed oil includes degumming, neutralization, bleaching, and deodorization [15]. The following procedures were carried separately for castor seed and neem seed oil.

250g crude sample of oil was heated in a 500 ml conical flask to 70°C. (6% volume) of 64% aqueous citric acid solution was added gently and mixed thoroughly with a magnetic stirrer for 15 minutes at 800rpm. The oil sample was decanted gradually. 4 ml of 8% Sodium hydroxide (NaOH) solution was gently added and the mixture magnetically stirred at 400 rpm for 15 minutes. The sample was washed in a separating funnel with warm distilled water, and then dried in vacuum oven at 85°C for 50 minutes to reduce the water content in the oil. 2 g silica gel was added to the mixture at 70°C and it was agitated rigorously for 30 minutes at 300 rpm to prevent it from settling out. Coconut shell activated carbon was then added and magnetically stirred for 30 minutes at 95°C. The sample was then filtered and then washed with distilled water 3 times. The sample was dried over anhydrous sodium sulphate Na_2SO_4 . Then the sample was dried in vacuum oven at 85°C for 50 minutes to reduce the water content in the oil [15].

2.3. Epoxidation process of blending castor and neem oils

100g of oil was used for the epoxidation process.

2.3.1 (PAA) preparation

Glacial acetic acid CH_3COOH (13 g) was placed in a 250 ml flat bottom flask followed by the addition of toluene C_7H_8 (13.5 g). The mixture was continuously stirred with a magnetic stirrer and after 30 minutes, followed by the addition of 30% aqueous hydrogen peroxide H_2O_2 (38 g) and continuously stirred (1200rpm) for 1 hour to produce peracetic acid (PAA).

2.3.2 Epoxidation at ratios of 40:60, 50:50 and 60:40

Refined castor seed oil and refined neem oil in ratios of 40:60, 50:50 and 60:40 were mixed separately to make 100g and then was poured into a two-necked flask equipped with a reagent injector and a condenser and stirred continuously and then was heated to 70°C. 1 % Sulphuric acid H_2SO_4 (10 g) was poured into the sample and continuously stirred for 15minutes at a constant temperature of 55°C. Followed by the addition of the PAA mixture into the two-necked reactor flask using the reagent injector and added in drops. The mixtures were continuously stirred (1200 rpm) at a regulated temperature between 40°C and 65°C for 7 hours. Aliquot was taken out for FTIR analysis to determine the effect of reaction time on the product. The reaction mixture was transferred from the flat bottom flask into a 1 litre separating funnel and then allowed for an interface to form. Then the bottom layer was discarded and the upper layer was collected. The upper layer was washed with distilled cold water 3 times and following by washing with distilled warm water 5 times and then distilled cold water again 2 times. The sample was dried over anhydrous sodium sulphate Na_2SO_4 . The sample was put in the vacuum oven at 105°C to remove toluene C_7H_8 from the sample. Aliquot was taken for FTIR analysis to determine the formation of epoxide in the product [1][16].

2.4. *Transesterification process of blended epoxy alkyl esters carried out for ratio of 40:60, 50:50 and 60:40*

100g of blended epoxidized sample of 40:60, 50:50 and 60:40 were introduced separately into the flat bottom glass equipped with a magnetic stir bar. The epoxidized sample was heated to 55°C on a hot plate and magnetically stirred. Followed by addition of Oxolane (Tetrahydrofuran) (5g) (CH₂)₄O into the flask of epoxidized sample and stirred continuously. A mass of 1 g of sodium hydroxide NaOH (1.0 wt %) as catalyst was weighed. And the catalyst was completely dissolved in 66 ml of pure methanol CH₃OH to form sodium meth-oxide. The resulting mixture (Sodium meth-oxide) was then added to the sample (oil + THF) in a flat bottom flask and heated to 60°C on a regulated hotplate and magnetically stirred at a rotational speed of 800 rpm. The set up was equipped with a reflux condenser. The mixture was refluxed and stirred for 2 hours at 60 - 65°C. The mixture was transferred into a 500 ml separating funnel and kept in an oven for 50 minutes at a temperature of 85°C. The bottom layer containing the glycerol was discarded while the top layer containing epoxidized alkyl ester was collected. The collected layer containing epoxidized alkyl ester was washed with 0.01 M phosphoric acid H₃PO₄ 4 times and then washed with distilled warm water 3 times; the emulsion was kept in oven for one hour for separation. The bottom layer which contains washed out of residual glycerol and sodium hydroxide NaOH was discarded, while the top layer containing purified epoxidized alkyl ester was collected. The purified epoxidized alkyl ester + THF + methanol were dried over anhydrous sodium sulphate Na₂SO₄. The purified epoxidized alkyl ester + THF + methanol was then placed in a rotary evaporator under reduced pressure of about 10 mbar and temperature of 60°C to remove the excess methanol. The sample was then put in the vacuum oven at 105°C to remove to Oxolane (THF) from the sample [1][2][5][18].



Figure 1. Overview of the experimental process sequence

2.5. *Characterization of the blended epoxy castor-neem oil esters*

2.5.1. Fourier Transform Infrared (FT-IR) Spectrometry: The FT-IR spectra were measured using a Thermo Scientific Nicolet iS5 FT-IR spectrometer. A drop of the sample was placed on the spectrometer measurement cell and a spectrum was obtained.

2.5.2. Dielectric Strength: The dielectric strength of the blended castor-neem samples were measured using the megger (OTS 60SX). The operation of the test set is simple and the results are displayed on a bright LED screen. The instrument is an automatic machine that can assess the quality of oil based on American Society for Testing and Materials (ASTM). The oil sample was placed between two electrodes with a 2.5 mm gap. A constant increasing voltage was applied until the oil discharges at a certain kV; the kV was recorded as the breakdown voltage [17].

2.5.3. Kinematic Viscosity: Viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress. For liquids, it corresponds to the informal concept of “thickness”. It was determined following ASTM D 445 standard specification. A suspended-level capillary viscometer was used to determine the kinematic viscosity. The viscometer is a U-shaped piece of glassware that was immersed into the hot water bath to regulate the varied temperature difference. This was multiplied by a constant specific for every viscometer (S3 viscometer used), to give a measure of the sample viscosity at 40°C, 50°C, 60°C and 70°C. The initial temperature was set at 30°C [1][7].

2.5.4. Flash Point: The flash-points of the samples were determined using an improvised method. Apparatus used were 50 ml conical flask, thermometer, heating mantle and a gas blower torch. A conical flask (50 ml) was filled with 5 ml of oil and equipped with a thermometer and then heated at

as very low constant rate on the hot plate. A gas blower torch was brought to the mouth of the conical flask and gradually passed across the mouth of the conical flask after every degree increase in temperature as heating continued until a pop sound that ignited the fire was seen. The flash point was obtained when application of a test flame causes the vapour above the oil to ignite. The temperature was then recorded as the flash point of the sample. The procedure was repeated for the various samples [8][9].

2.5.5. Acid value and free fatty acid (FFA): The acid value of an acid or fat is the number of mg potassium hydroxide required to neutralize the free acid in (1 g) of the sample. The FFA is an aliphatic carboxylic acid which contain a long hydrocarbon; It can be hydrophilic or hydrophobic [14].

Procedure:- Mix 25ml diethyl ether with 25ml ethanol. Add 1ml of 1% phenolphthalein indicator solution. Neutralize with 0.1M potassium hydroxide solution. Dissolve 1 to 10g of the oil in the neutralized solvent mixture. Titrate with 0.1M potassium hydroxide solution. Shake constantly until a pink colour which persists for 115seconds is obtained [7][8][14][19].

Calculation:

$$\text{Acid value} = \frac{\text{titration (ml)} \times 5.61}{\text{Wt of sample used}} (\text{mg KOH/g}) \quad (1)$$

$$\text{Also acid value} = 2 \times \text{Free fatty acid (FFA)}. \quad (2)$$

FFA% is approximately $\frac{1}{2}$ AV [8][14].

2.5.6. Peroxide value: The peroxide value is a measure of the peroxides contained in the oil [14].

Procedure:- The test should be carried out in subdued daylight. Weigh out 1g of oil into a clean dry boiling tube. Add 1g powdered potassium iodide and 10ml of the solvent mixture. Place the boiling water bath so that the liquid boils within 30seconds and allow to boil vigorously for more than 30seconds. Pour the content into a titration flask containing 20ml freshly prepared 5% potassium iodide solution. Wash the tube twice with 25ml portions of water and add the washings to the titration flask. Titrate with 0.002M sodium thiosulphate solution using starch as indicator (aml). Carry out a blank determination. Blank titre (bml) [8][9][19].

Calculation:

$$\text{Peroxide value} = \frac{2(a-b)}{\text{Wt of oil}} (\text{mEq/kg}) \quad (3)$$

2.5.7 Saponification value: The saponification value of oil is the number of mg of potassium hydroxide to neutralize the fatty acids resulting from the complete hydrolysis of the sample [14].

Procedure:- Weigh 2g of oil (using a dropping pipette) into a flask. Add exactly 25ml of the alcoholic potassium hydroxide solution into the flask. Attach a reflux condenser and heat the flask on a water bath for 1 hour with occasional shaking. At the end of 1 hour remove the flask from the water bath and add 1ml of the 1% phenolphthalein indicator. Titrate while still hot with the standard 0.5M hydrochloric acid (aml). Carry out blank determination omitting oil (bml) [8][9][10][14].

Calculation:

$$\text{Saponification value} = \frac{(b-a) \times 28.05}{\text{Weight of oil}} (\text{mg KOH/g}) \quad (4)$$

2.5.8 Iodine value: The iodine value of oil is the weight of iodine absorbed by 100 part by weight of the sample. The iodine value is a measure of the degree of unsaturation of the fatty acids in the oil. [14].

Procedure:- Weigh accurately about 0.2 – 0.5g of the oil (with the acid of a dropping pipette) into a glass stopper flat bottom flask of about 25ml. Add 10ml carbon tetrachloride to the oil and dissolve. Add exactly 20ml Wiji's solution and insert the stopper which has been moistened with potassium

iodide solution. Mix and allow to stand in a dark cupboard for 30 minutes. Add 15ml 10% potassium iodide solution (freshly prepared), 100ml water and mix. Titrate the mixture with 0.1M standard sodium thiosulphate solution using starch as an indicator just before the end point (aml). Carry out a blank simultaneously omitting the oil (bml) [8][9][14][6].

Calculation:

$$\text{Iodine value} = \frac{(b-a) \times 1.269}{\text{Weight of oil/fat}} \quad (5)$$

Note: if $b - a$ is greater than $b/2$, repeat the test using smaller amount of the sample. Record result in Wiji's.

2.5.9 Water content: This is a measure of the % moisture lost due to drying at a temperature of 105 degrees Celsius [14]. 2g of the sample was weighed (W_1) into preweighed crucible (W_0) and placed into a hot drying oven at 105 degrees Celsius for 3 hours. The crucible were removed, cooled in a desiccator and weighed. The process of drying, cooling and weighing were repeated until a constant weight (W_2) was obtained [9][19][14]. The weight loss due to moisture was obtained by the equation 6:

$$\text{Moisture (\%)} = \frac{W_1 - W_2}{W_1 - W_0} \times 100 \quad (6)$$

Where: W_0 = Weight of the empty crucible (g). W_1 = Weight of the sample + empty crucible (g). W_2 = Weight of dried sample + empty crucible (g).

2.5.10 Density: The weight of a small beaker was determined using an electronic weighing balance. 2 ml of the oil was poured into it and the weight was noted [8][12].

Calculation:

$$\text{Density} = \frac{(\text{Mass of oil})}{(\text{Volume of oil weight})} (\text{gcm}^{-1}) \quad (7)$$

2.5.11 Boiling point: The boiling point of a liquid is the temperature at which the vapour pressure of the liquid equals the pressure surrounding the liquid and the liquid changes into a vapour. The oil was placed in a container with an inserted thermometer and heated to the time the first bubble was observed. The temperature was noted, the experiment was repeated twice and the average temperature calculated [8].

2.5.12. Specific gravity: An empty 10 ml pycnometer bottle was weighed. The 10ml pycnometer bottle was filled with water and weighed. The 10 ml pycnometer bottle was then filled with the same volume of oil as that of the water and weighed [19][19]. The specific gravity is calculated with equation 8 shown below.

Calculation:

$$\text{Specific Gravity} = \frac{W_3 - W_1}{W_2 - W_1} \quad (8)$$

where, W_1 = weight of empty bottle. W_2 = weight of bottle + water. W_3 = weight of bottle + sample oil.

2.5.13. Appearance or oil colour

The procedure was visual.

3. Results and Discussion

The FT-IR study of the blended epoxy castor-neem oil ester at ratio of 40:60, 50:50 and 60:40

The methylene vibration of CH_2 in which all groups of straight chain alkanes in the blended samples vibrate in Phase, this peaks at 722.29 cm^{-1} and 721.80 cm^{-1} respectively in Figure 3, 4 and 5. The samples of observed broad peak in BEME46, BEME55 and BEME64 were observed to peak between the region of the spectrum of 750 cm^{-1} and 1000 cm^{-1} . The carbon-carbon spectra peak wasn't present in the BEME46, BEME55 and BEME64 spectra [20].

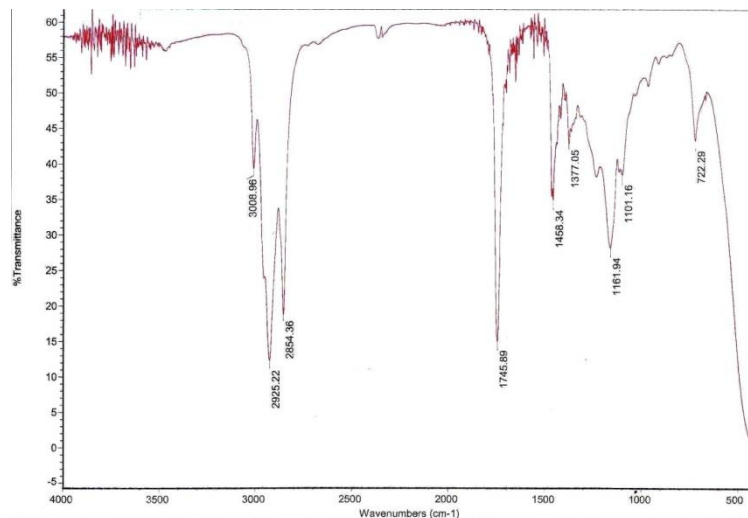


Figure 2: Fourier Transform Infrared FTIR Spectrum of Blended castor-neem oil methyl ester in ratio of 40:60 respectively (BEME46)

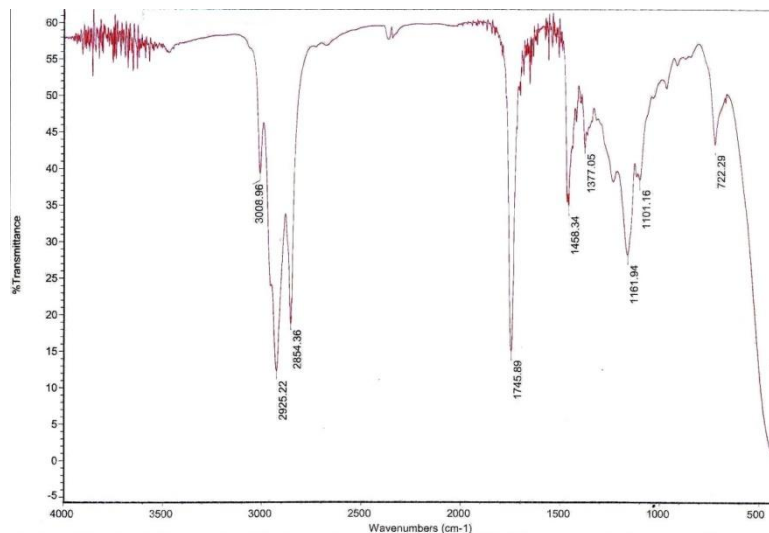


Figure 3: Fourier Transform Infrared FTIR Spectrum of Blended castor-neem oil methyl ester in ratio of 50:50 respectively (BEME55)

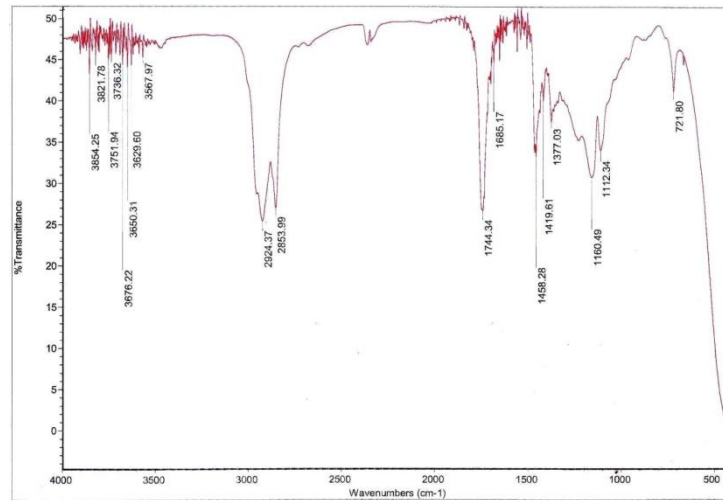


Figure 4: Fourier Transform Infrared FTIR Spectrum of Blended castor-neem oil methyl ester in ratio of 60:40 respectively (BEME64)

Table 1: Description of experimental samples

Sample	Description
BEME46	Blended epoxy castor-neem methyl ester {in ratio of 40:60}
BEME55	Blended epoxy castor-neem methyl ester {in ratio of 50:50}
BEME64	Blended epoxy castor-neem methyl ester {in ratio of 60:40}
MinOil	Transformer Mineral Oil obtained from AT3 Transmission Power Station

Table 2: Overall properties of BEME46, BEME55 and BEME64 and comparing with transformer oil specification of ASTM standard

Property	Values				
	BEME46	BEME55	BEME64	MINOIL	Transformer oil ASTM Standard
Dielectric Strength, kV, 27°C	30.55	30.75	32.03	40.53	25 - 40
Viscosity, cSt, @ 30°C	9.30	9.45	9.48	16.03	9.3 – 27
Viscosity, cSt, @ 50°C	6.02	6.91	6.91	10.93	-
Viscosity, cSt, @ 70°C	3.30	3.32	3.48	5.46	-
Density, gcm⁻³ @ 27°C	0.8260	0.8251	0.8219	0.8888	0.55 – 0.89
Flash point, °C	165	168	160	138	Above 140
Acid value, mg KOH g⁻¹ oil	0.1683	0.1405	0.1405	0.0846	0.01 – 0.3
Boiling point, °C	150	149	151	134	Above 120
Specific gravity	0.8491	0.8483	0.8502	0.8909	0.89 – 0.91
Saponification value, mg KOH g⁻¹ oil	153.573	156.379	155.678	151.470	150 – 244
Peroxide value, mEq g⁻¹ oil	8.240	8.000	9.115	4.440	5 – 10
Iodine value, g100g⁻¹ oil	55.902	56.376	56.488	28.178	55 – 120
Free fatty acid, mg KOH g⁻¹ oil	0.0842	0.0703	0.0703	0.0421	0.01 – 0.08
Moisture content, %	0.045	0.064	0.062	0.013	-
Appearance	Yellow tint	Yellow tint	Yellow tint	Yellow tint	White / yellow tint

*Transformer oil specification for ASTM standard was sourced from [8].

Table 3: Key properties of BEME46, BEME55 and BEME64 and comparing with transformer oil specification of ASTM standard

Key Property	Values				
	BEME46	BEME55	BEME64	MINOIL	Transformer oil ASTM Standard
Dielectric Strength, kV, 27°C	30.55	30.75	32.03	40.53	25 - 40
Viscosity, cSt, @ 30°C	9.30	9.45	9.48	16.03	9.3 – 27
Viscosity, cSt, @ 70°C	3.30	3.32	3.48	5.46	-
Density, gcm⁻¹ @ 27°C	0.8260	0.8251	0.8219	0.8888	0.55 – 0.89
Flash point, °C	165	168	160	138	Above 140
Acid value, mg KOH g⁻¹ oil	0.1683	0.1405	0.1405	0.0846	0.01 – 0.3
Boiling point, °C	150	149	151	134	Above 120
Specific gravity	0.8491	0.8483	0.8502	0.8909	0.89 – 0.91
Moisture content, %	0.045	0.064	0.062	0.013	-
Appearance	Yellow tint	Yellow tint	Yellow tint	Yellow tint	-

*Transformer oil specification for ASTM standard was sourced from [8]

The refining process and the formation of an oxirane or epoxy in the molecular structure of BEME46, BEME55 and BEME64 that changed the double bonds had a significant effect by increasing the viscosity and dielectric strength of the blended oil esters. The transesterification synthesis of the blended epoxy oil esters that separated the free fatty acid FFA from the glycerol was observed to also increase the dielectric strength. The purification stage was observed to alter the acid value of the oils. The acid value decreased tremendously after the refining process. The observed and clear reduction in the acidity is as a result of neutralizing the oil with a base (sodium hydroxide NaOH) [5][15]. Transesterification of the blended epoxy oil esters decreased the acid value and by this reduced the FFA of the esters. The acidity of insulating fluid can be increased by thermal degradation of the fluid [13]. This is because oxidation products are often acidic in nature. Acids formed during ageing of mineral oil are classified into two; high molecular weight acids (HMWA) and low molecular weight acids (LMWA) [4]. The flash point BEME46, BEME55 and BEME64 has shown that the esters can safely be used even where the temperature is expected to be very high. Oils with flash point above 66°C are considered as safe oils. Viscosity of BEME46, BEME55 and BEME64 is observed to decrease with increase in temperature.

4. Conclusion

The electrical transformer insulating fluid from blending Castor and Neem seed oils have been produced and characterized. The refined castor and neem seed oils was mixed thoroughly at a ratio of 40:60, 50:50 and 60:40 to make up weight of 100g each.. The blended ratios were synthesized separately through epoxidation and transesterification reactions to satisfactorily remove glycerol and unsaturation and form an epoxy in the triglycerides of the mixture. The characterization of the blended epoxy castor-neem oil methyl ester in ratio of 40:60, 50:50 and 60:40 (BEME46, BEME55 and BEME64) revealed that the viscosities of BEME46, BEME55 and BEME64 at a lower temperature of 30°C were 8.30 cSt, 8.45 cSt and 8.48 cSt respectively, while the viscosities at a higher temperature of 70°C were 3.30 cSt, 3.32 cSt and 3.48 cSt respectively, using a glass capillary kinematic viscometer and hot water bath setup. The dielectric strength of BEME46, BEME55 and BEME64 were 30.55 kV, 30.75 kV and 32.02 kV respectively using a megger test set. However, the comparison of the BEME46, BEME55 and BEME64 with the Transformer Mineral oil obtained from APO Transmission station showed that the blended oil esters met the transformer standard specifications.

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