

Development of Eco-Friendly Transformer Oil from Neem Seed Oil using Periwinkle Waste Shell as Catalyst

Olatunde Ajani OYELARAN^{1*}, Sunday Gbenga BORISADE², James Oyelowo ABIOYE³,
Bukola Olalekan BOLAJI⁴

¹*Department of Mechanical Engineering, Faculty of Engineering, Ikole-Ekiti Campus, Federal University Oye-Ekiti, PMB. 373, Ekiti State, Nigeria*

²⁻⁴*Department of Materials and Metallurgical Engineering, Faculty of Engineering, Ikole-Ekiti Campus, Federal University Oye-Ekiti, PMB. 373, Ekiti State, Nigeria*

Abstract – The urge for a sustainable and non-polluting industry has raised the need for a search for an alternative insulating fluid that could be used in transformers, because of the negative environmental effect of mineral-based insulating fluids. Natural esters from non-edible oil seeds showed to be feasible alternative, unfortunately they possess poor thermo-oxidative properties. Neem trees grow nearly on all types of soils and can be as old as 45 years before they start to produce less fruits. Neem tree can produce up to 50 kg of its fruits yearly from the tenth year and the seed oil content is 45 % making it a sustainable tree. The neem seed transformer oil (NSTO) was produced via catalysed transesterification process using neem seed oil and methanol with periwinkle shell as catalyst. Prior to the transesterification reaction the seed oil was degummed. The produced ester also underwent water treatment process. The produced NSTO density, water content, acidity flash point, pour point, viscosity, breakdown voltage, dielectric, relative dielectric constant, dissipation factor and resistivity properties were tested, presented and discussed. Results show that NSTO meet most of the standard requirements of new natural ester fluids as insulating oil. The dielectric breakdown voltage of the oil is 32 kV, which is more than the required for new natural ester fluids. The dissipation factor limit value for new natural ester of 0.20 % at 25 °C limit value was met by NSTO from 100 Hz to 400 Hz, and the relative dielectric constants of NSTO are 3.01 k, which value also meets the specification of between 2.9 k and 3.2 k. The pre-treatment of degumming of the seed oil prior to transesterification and water treatment of the produced ester appears to be a promising route for the production of electrical insulating oil for transformer.

Keywords – Catalyst; degumming; dielectric; neem seed oil; periwinkle waste shell

Nomenclature

BET	Brunauer-Emmett-Teller
$\tan\delta$	Dielectric dissipation factor
ϵ_r	Relative dielectric constant
ρ	Resistivity
AC	Ash content

* Corresponding author.

E-mail address: ajanioyelaran@gmail.com

C_s	Capacity oil-filled test cell
C_a	Capacity of empty test cell
ω	Angular frequency
ε	Dielectric constant of oil

1. INTRODUCTION

The continuous exploration on sustainable development requires vivid use of energy and resources, which involves venture on greener processes and renewable materials [1]. The remarkable increase in number of transformers in the last few decades has led to greater demand of transformer oil. Transformer oils are insulating dielectric liquids that are capable of insulating power equipment against electrical stresses, for instance capacitors, tap changers, circuit breakers, transformers, bushings and cables. Insulation oils are specifically refined with a goal of getting the appropriate characteristics of insulation, they are used as heat transfers, electrical insulators and health monitoring agents [2]. Balat wrote that in 2012, all over the world about 89 million barrels of petroleum was consumed every day [3]. If urgent action is not taken, the world oil reserves are projected to exhaust in the next 50 years at this consumption ratio [4]. Since the exhaustion of crude oil resources are estimated for some time, the rising cost of petroleum products and ecosystem apprehension necessitates scientist and engineers to strive for alternative and eco-friendly sources, hence bio-based transformer oil has become an area of high interest as alternatives to mineral transformer oil. Kirtay wrote that several countries are searching for renewable sources as substitute that can be able to balance GHG emissions [5]. For over a century, mineral oils have been utilized in ensuring cooling in power transformers and electrical insulation. Transformer oils are largely derived from petroleum products but are not environmentally friendly and non-renewable. However, in the last few decades, ester fluids and synthetic hydrocarbon fluids were introduced but with limited advantages and use [6]. Also, mineral oils are non-biodegradable, highly flammable and hazardous to the environment, its spillage contaminates both soil and water [7].

Bio-based transformer oil is now gaining recognition as an alternative mineral based transformer oil since it possesses quite a few advantages over conventional transformer oil which includes, being more environmentally friendly, renewable and non-toxic. The easy access of producing natural esters from vegetable oils has made them favourable substitute to replace petroleum-based insulation oils for transformers. A lot of researches are ongoing to determining the viability of natural ester insulation fluid for both distribution and power transformers [8]–[10]. This is expected since natural ester insulation oils are biodegradable. Furthermore, natural ester insulation oils possess good dielectric properties and higher fire point. They are believed to aid in increasing the lifecycle of transformers since they have better hydrophobic properties in comparison to petroleum-based insulation fluids [11], [12]. For natural esters to be used in transformer, characteristic like as pour point, flash point, viscosity, specific gravity and dielectric strength must be meet as par IEC and ASTM standards.

A large number of feedstocks have been tested and acknowledged as suitable for biobased transformer oil production, such as rapeseed and nut oils [13], *jatropha* oil [13], [14]. There exist many non-edible oil seed such as neem (*Azadirachta Indica*), karanja (*Pongomia Pinnate*) and thevetia (*Thevetia peruviana*), that can be experimented for use as transformer oil. However, among them, neem seed, which contain between 25 % and 45 % oil in its dry

state [15] is a potential feedstock for biobased transformer oil production. Neem (*Azadirachta Indica*) is of the mahogany tree family Meliaceae that grows in large quantities in different parts of the world. They grow on nearly every kind of soils and can be as old as 45 years before they produce little or no fruit, depending on the soil conditions. They are deciduous and evergreen tree with straight trunk and long spreading branches growing to a height of about 12–18 m [16]. Neem trees start bearing fruits at about 3 to 5 years of planting and can produce fruits of 50 kg yearly from the tenth year [17]. The extracted seeds produce about 45 % oil, making it highly recommended for oil production, and the oil is hydrophobic in nature [18].

Apart from alcohol and oil in esters production, catalysts are also key resources [9]. There are different types of catalyst such as enzymes, heterogeneous and homogeneous catalysts. Complications of catalyst residues and post washing treatments, are some of the drawback suffered using catalytic homogenous process [10]. However, the use of heterogeneous catalyst is still the best option [11]. Heterogeneous catalysts have little disposal setbacks, environmentally and are non-corrosive. Their separation from liquid products is easy and they possess longer catalytic lifetimes [12]. Currently, CaO is the most commonly used catalyst for transesterification with high yield among the alkaline earth metal oxides [18]. The objective of this research is to produce biobased transformer oil from neem oil using waste periwinkle shell as catalyst. The obtained oil was analysed and the results compared standards for bio-based transformer oil.

2. MATERIALS AND METHODS

2.1. Neem Seeds Collection and Characterization

The neem seeds used for this research were collected from Kano, periwinkle waste shell was collected from De Pillars Apartment in Ogbomoso, Nigeria. All reagents were purchased from Lagos, the reagents are of high grade and used as received.

2.2. Oil Extraction

Soxhlet extraction process was adopted in extracting the oil from powdered seeds (60 °C, 8 h), n-hexane was used as the solvent. The extraction was conducted in a conducive environment suitable for soxhlet extraction process. The oil was recovered from the resulting solution by distillation. After the process, the solvent was recovered utilizing a rotary evaporator. The percentage yield of crude lipid was evaluated in Eq. (1). The oil was kept at 20 °C till it was needed for experiment.

$$\text{Crude oil (\%)} = \frac{\text{Weight of the extracted oil}}{\text{Weight of the sample}} \cdot 100 \quad (1)$$

2.3. Degumming of Neem Oil

Neem seed oil was heated to 80 °C, mixed with 5 % volume of water and stirred for 15 minutes using magnetic stirrer. The mixture result was then centrifuged for 20 minutes to degum the neem seed oil.

2.4. Preparation and Characterization of Catalyst

Periwinkle shell waste was cleaned by removing all the left over remaining shell after which it was thoroughly washed with warm water severally. It was then dried at 80 °C overnight in an oven. The shell was then crushed, grinded and then sieved (38–75 µm). It was then heat treated in a furnace [19]. After which it was calcined at 1000 °C for 4 h at heating rate of 10 °C /min in air atmosphere. The pore volume, mean pore diameter and surface area were evaluated using adsorption-desorption of nitrogen (N₂) at 77 K using Autosorb-1 Model No. *ASIMP.VP4*, USA Quantachrome Instrument. The pore volume and mean pore diameter were obtained by utilizing desorption branch by applying Barret-Joyner-Halenda (BJH) method [20]. The surface area was however, calculated using the Brunauer-Emmett-Teller (BET) equation.

2.5. Production of Biodiesel Using Base Catalyst

An experimental production of biodiesel was performed on laboratory scale with a conical flask of 250 mL enclosing 40 g oil of neem seed. The experiment was carried out in an oven chamber at a temperature of 60 °C. The oil pre-treated was transferred into a flask and heated. The calcined periwinkle shell powder was dissolved in methanol (1 %), as catalyst and degummed neem seed oil was added. The transesterification process was left to continue for 2 hours [21]. The mixture produced was left for 24 hours under gravity to settle before separating the neem ester. The glycerol was first tapped, being the lower layer, then the ester layer was washed between three and four times, using warm water. To dry the layer, it was passed over anhydrous calcium chloride.

2.6. Characterization of Produced Neem Seed Transformer Oil

2.6.1. Determination of Density

The density of oil was measured using a simple method, where an electronic weighing balance was used. The weight of a beaker was first determined after which 2 mL of the oil was poured into the measured beaker and the new weight was also noted. Density was calculated using Eq. (2).

$$\text{Density (\%)} = \frac{\text{Mass of oil}}{\text{Volume of oil weight}} \cdot 100 \quad (2)$$

2.6.2. Determination of Water Content

The water content of oils measured in accordance with ASTM D 6304 Test Method using *Metrohm 831 Karl Fisher (KF)* Coulometer. For the measurement, 1 mL of the oil was used [22].

2.6.3. Determination of Acidity

A *Metrohm 877 oil Titrano plus* was utilized to measure the oil acidity in accordance to ASTM D 974 Test Method. 10 g of oil was used during the experiment [23].

2.6.4. Determination of Flash Point

ASTM D93 Test Method was adopted for this work where the ester samples were separately introduced into an aluminium container with thermometer and heated at the rate of 10 °C per minute using a Bunsen burner. At every 5 °C increase a small flame is passed over the oil surface until a flash appeared. The temperature at which the flash occurred is their flash points. On the other hand, fire point of a sample is the temperature at which for over five seconds after ignition by open flame the flash in the sample is sustains [24].

2.6.5. Cloud and Pour Point Determination

The cloud point of the produced ester was determined using the ASTM D2500–02 Test Method. The sample was cooled at given rate and was frequently examined. The temperature which a cloud is observed first at the bottom of the jar to the nearest 1 °C is the cloud point. The samples were further cooled and at intervals flow of oil are monitored until no flow of samples was observed. The temperatures of which the samples no longer flow is termed the pour point. Bertrand and Hoang, [25] wrote that the highest temperature at which the oil begins to solidify is the pour point.

2.6.6. Determination of Viscosity

Brookfield Viscometer with the proper spindle was used for the measurement of the ester viscosity. The container with the ester and temperature sensor that monitored the temperature of ester was placed in water bath, heated on a hot plate controlled thermostatically by varying within accuracy of ± 0.1 °C sample temperature. For accuracy, the average of the three measurements performed for each was calculated and noted as the viscosity.

2.6.7. Water Treatment Process

IEEE standard C57.147TM states that lower value of breakdown voltage signifies the presences of pollutants like water. Hence, the need to pretreat the oil so as to reduce the moisture content of oil so as to aid the oil's capability in enduring electric stress. For this reason, water treatment was carried out on the produced oil prior to the determination of the electrical properties by bubbling nitrogen gas (N₂) into the oil for 30 minutes as done by Wada *et al.* [26].

2.6.8. Determination of Dielectric Properties of Oils

The breakdown voltage (dielectric strength) of the oils were tested using an automated machine that assessed the oil quality. OTS 60PB Megger oil test set was utilized based on ASTM D1816 specifications. In this work, purified neem ester and SINOPEC transformer oil were aged in a stainless vessel for 18 hours at 90 °C. Thus, decreasing the content moisture in the oil. The oils were subsequently conserved at 25 °C. The dielectric strength of oils was then measured in accordance to IEC 60156 standard while relative dielectric constant and dissipation factor were measured in accordance to IEC 60156 and ASTM D924-08 standard methods. However, frequency and voltage were used to calculate the resistivity of oils.

2.6.9. Determination of Breakdown Voltage

A container with the oil was mixed gently a couple of times and the test cell cleaned with droplet of oil and drained. Clean oil devoid of bubbles of air was used to fill the cell. The cell

was closed in order to flush out the oil from air. Test voltage was applied after waiting for 5 minutes with 2.5 mm set as electrode gap with 2 kV/s rise rate of voltage in accordance with IEC 60156 standards and six measurements were carried out.

2.6.10. Determination of Dielectric Dissipation Factor, Relative Dielectric Constant and Resistivity

Dissipation factor gives vital insight on the quality of transformer oil which is contaminated and aged in service. The Dielectric dissipation factor ($\tan\delta$) was calculated using Eq. (3).

$$\tan\delta = (\omega \cdot R \cdot C)^{-1} \quad (3)$$

The relative dielectric constant of transformer oil (ϵ_r) is calculated as the ratio of oil-filled test cell capacity (C_s) to capacity of empty test cell (C_a) as seen in Eq. (4). It has been proved that for more uniform electric field higher dielectric constant is better [13].

$$\epsilon_r = \frac{C_s}{C_a} \quad (4)$$

While the voltage and frequency changes were utilized to calculate the relative dielectric constant and dissipation factor, the oils resistivity were calculated in 50–400 Hz frequency and 1–12 kV voltage ranges. The resistivity of the oils is expressed in Eq. (5) as:

$$\rho = (\tan\delta \cdot \omega \cdot \epsilon)^{-1}, \quad (5)$$

where ρ is resistivity, ω is angular frequency and ϵ is the dielectric constant of oil.

3. RESULTS AND DISCUSSION

The density of transformer oil as presented in Table 1 was found to be 0.893 g/cm³. The value meets the requirement for density as stated by IEEE C57.147, which states that the density has to be equal to or less than 0.96 g/cm³. The water content of the produced transformer oil before water treatment was found to be 261.1 ppm. In contrast, the water content of the produced transformer oil after water treatment was reduced to 155.2 ppm as shown in Table 1. It should be noted that the oil produced only met requirement as stipulated in IEEE C57.147 only after water treatment was carried out on the oil. The acidity of produced transformer oil is 0.1436 mgKOH/g as shown in Table 1. This value did not meet the natural ester fluids requirement for acidity according to IEEE C57.147 specification, which stipulates that the acidity should be equal to or less than 0.06 mg KOH/g. From Table 1, the flash point of produced transformer oil of 194 °C did not meet the flash point specification in accordance with IEEE C57.147 specification which stipulates that the flash point have to be equal to or more than 275 °C. However, the flash point values are above the 145 °C specification of ASTM D3487 standard. The pour point of the produced transformer oil is –20 °C as shown in Table 1 fulfil the pour point specification in accordance with IEEE C57.147 specification, which stipulates that the pour point of natural ester fluids should be less than or equal to –10 °C. The produced transformer oil viscosity at 40 °C is 15.23 mm²/s. This value meets the specification as stipulated in IEEE C57.147 standard which states that the viscosity at 40 °C should be less than or equal to 50 mm²/natural ester fluids to be used as transformer oil.

TABLE 1. PHYSICOCHEMICAL AND ELECTRICAL PROPERTIES OF PRODUCED TRANSFORMER OIL AND SPECIFICATIONS

Property	Specification for Natural Ester Fluids [15], [28]	Specification for Mineral Insulating Oil [29], [28]	Neem seed oil Transformer oil
Density, g/cm ³	≤ 0.96	≤ 0.91	0.893
Water content, mg/kg	≤ 200	≤ 35	155.2
Acidity, mgKOH/g	≤ 0.06	≤ 0.03	0.1436
Flash point, °C	≥ 275	≥ 145	194
Pour point, °C	≤ -10	≤ -40	-20
Viscosity, mm ² /s	≤ 50	≤ 12	15.23
Breakdown voltage, kV	≥ 20	≥ 20	32

The breakdown voltage of NSTO is 16 kV before water treatment. However, the breakdown voltage of NSTO after water treatment is 32 kV as shown in Table 1. The acceptable breakdown voltage in accordance to IEEE C57. 147 specification, for new natural ester fluids have to be equal to or more than 20 kV. Therefore, the NSTO meets the specifications after water treatment. Fig. 1 shows variant of dissipation factor in relation to voltage while Fig. 2 shows measurements of dissipation factor in relation to frequency for NSTO and mineral oil. The limit value for dissipation factor of new natural ester fluids is stipulated as 0.20 % at 25 °C in accordance with ASTM D6871 specification. As shown in the figure this limit value is met by NSTO from 100 Hz to 400 Hz. Mineral oil and NSTO relative dielectric constant were measured in accordance to distinct voltage. The relative dielectric constant of the both oils were also measured from the range 50 Hz to 400 Hz frequency. The change of frequency and voltage does not affect the relative dielectric constant.

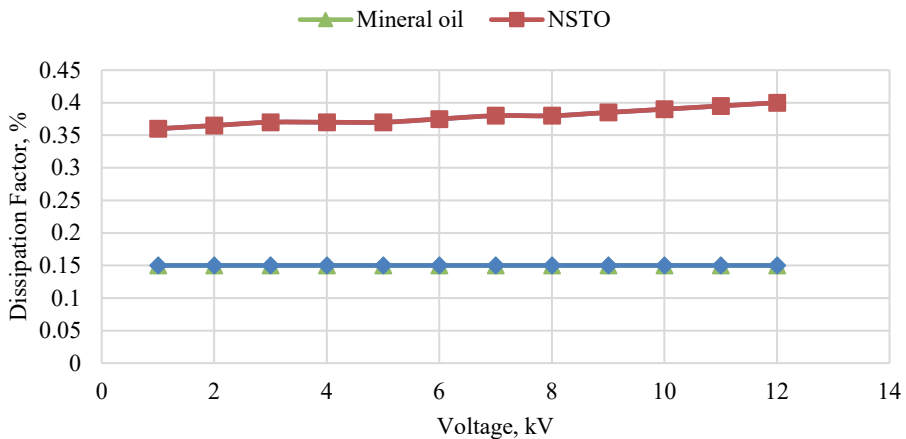


Fig. 1. Tan delta changes in relation to voltage.

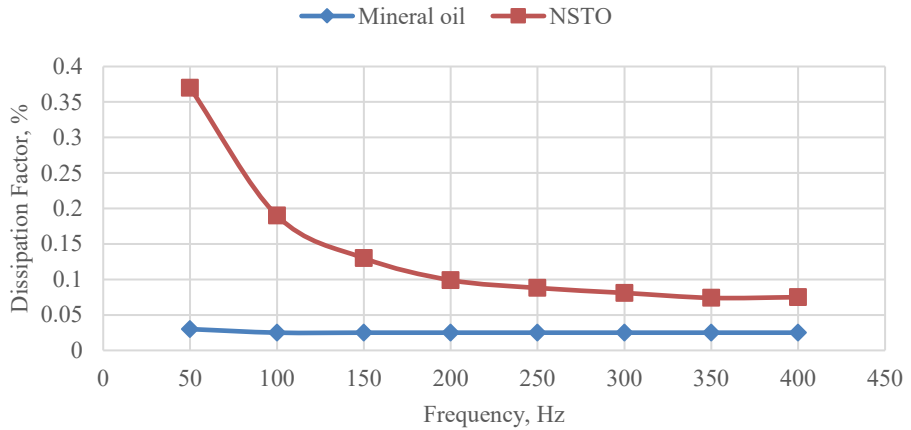


Fig. 2. Dissipation factor changes in relation to frequency.

Fig. 3 displaces the change of relative dielectric constant in relation to the voltage. The relative dielectric constants of NSTO are 3.01. This value meets the specification of between 2.9 and 3.2 as stated in the work of Fernandez *et al.* [29]. Hence, NSTO might be utilized as substitute to mineral oil in transformers. The variant of resistivity in relation to voltage at 50 Hz is shown in Fig. 4 while Fig. 5 shows the variations in resistivity according to frequency at 10 kV. As observed the oils resistivity are in a decreasing trend with increasing voltage and frequency. As seen the resistivity of NSTO is lower than that of mineral oil. ASTM D1169-11 specification which stipulates that the resistivity value acceptable for insulating liquids as $1 \times 10^{12} \Omega \cdot \text{cm}$ hence, NSTO meets the specification of natural esters as transformer oil in terms of resistivity for all frequency and voltage ranges measured.

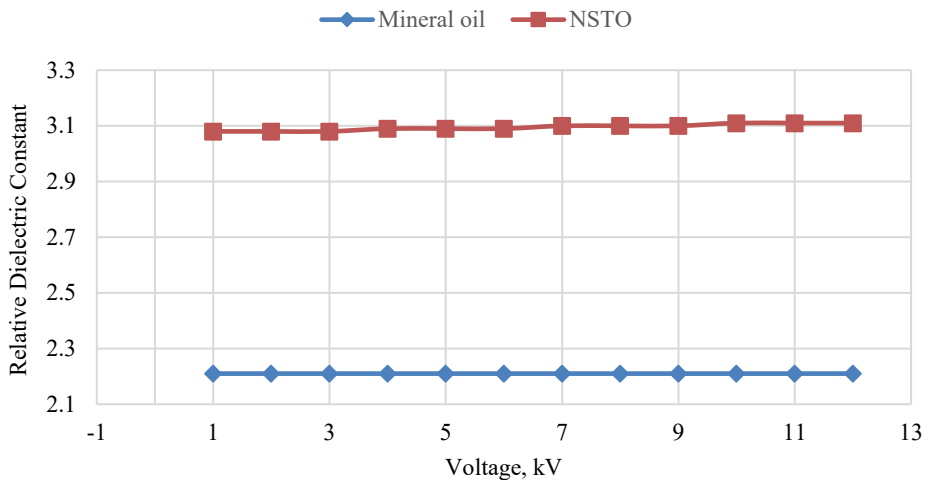


Fig. 3. Relative dielectric constant changes in relation to voltage.

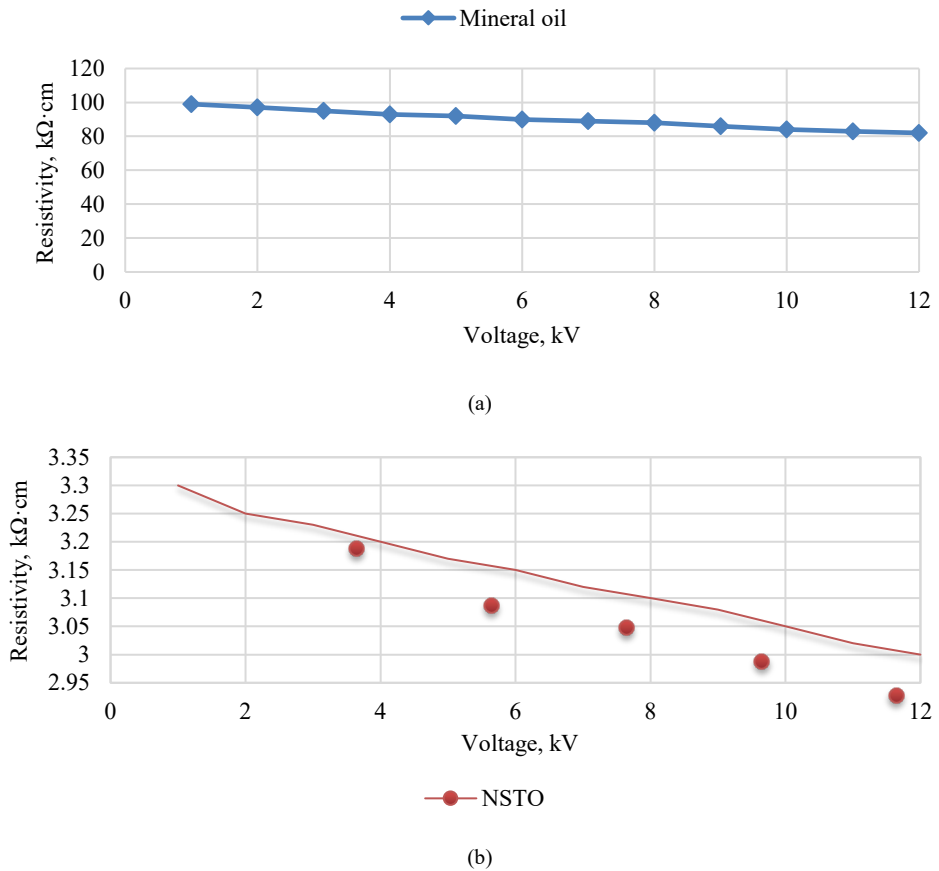
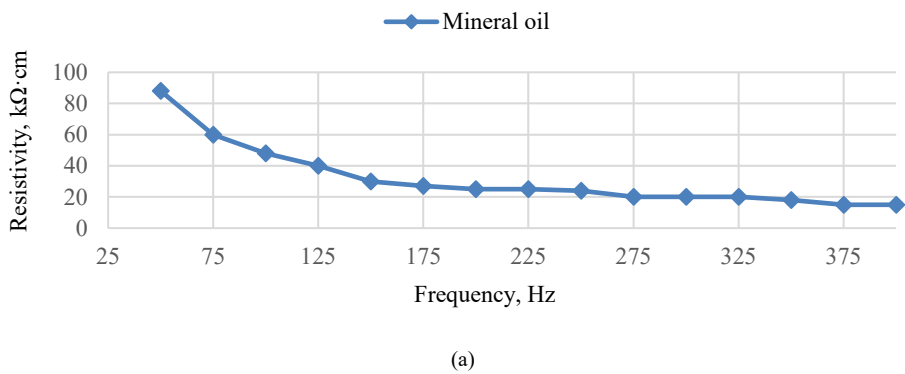


Fig. 4. Resistivity changes in relation to voltage: a) Mineral oil; b) NSTO.



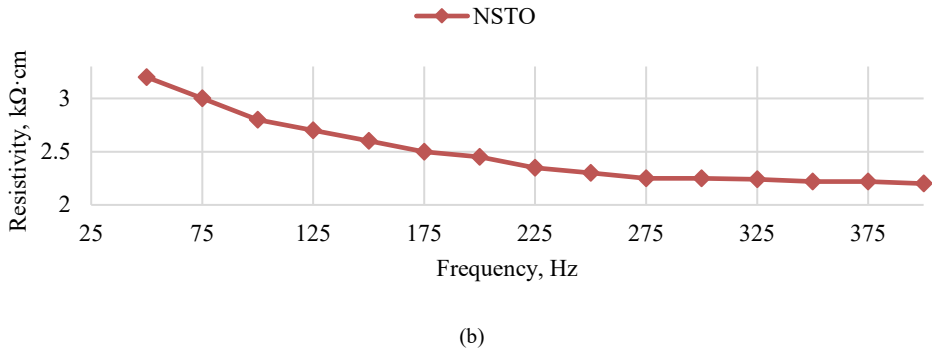


Fig. 5. Resistivity changes in relation to frequency: a) Mineral oil; b) NSTO.

4. CONCLUSION

An eco-friendly transformer oil from neem seed oil using periwinkle waste shell as catalyst was produced. The effect of degumming prior to esterification and water treatment of the produced ester also help to improve some of the properties of the produced NSTO. The breakdown voltage of NSTO was improved from 16 kV to 32 kV. The produced natural ester transformer oil met the density, water content after water treatment process, pour point and viscosity requirement as a substitute to mineral oil for used in transformers. It was discovered that the breakdown voltage of mineral oil was lower than that of NSTO. Increasing the voltage was found to increase the dissipation factor of oils, but increasing the frequency from 50 Hz to 400 Hz decreases the dissipation factor of oils. The results also show that changes in frequency and voltage does not affect the relative dielectric constant of the oils. There is decrease in resistivity of the oils with rising voltage and frequency. From properties exhibited by NSTO from the results obtained after degumming and water treatment processes neem seed oil ester using periwinkle shell as catalysis are good feed stock for the production of natural esters for use as insulating liquid in power equipment.

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