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Dielectric Performance of Graphene Oxide Dispersed Biodegradable Transformer Nanofluid under Ageing Conditions

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Abstract— ***Natural ester oil-based nanoparticle dispersed fluids are latest developed oils for insulation purposes in transformers. Different types of nanoparticles have been dispersed in crop extracted oils to synthesize renewable dielectric fluids to replace toxic and non-biodegradable mineral oil. The successful application of developed nanofluids in transformers depends upon the dielectric behavior of these fluids in fresh as well as under thermal ageing conditions. In this research, a crop extracted oil dispersed with graphene oxide nanoparticles has been tested for its dielectric performance before and after being put under thermal ageing conditions. Results have shown that at maximum values, the dielectric breakdown voltage of aged nanofluid samples reduces by 2.7% whereas this reduction is calculated to be 8.4% in the case of base oil samples after ageing. In terms of dielectric constant and loss tangent, the aged nanofluids also perform better as compared to the aged base oil. Moreover, the dispersion of nanoparticles has resulted in a possibility of decrease in the transformer core size by 8.6% as compared to the base oil.***

Index Terms— ***Ageing, graphene oxide, nanofluid, transformer.***

I. INTRODUCTION

Natural ester oils are generally oils extracted from the crop produce. These oils are treated in a particular way in order to enable them to be better liquid dielectrics. Natural ester oils are being studied for their dielectric capabilities in order to use them as dielectric fluids in transformers [1-3]. When compared to the currently used transformer mineral oil which is toxic and non-biodegradable, these vegetable based natural ester oils are completely biodegradable in nature [4, 5]. Being a petroleum based by-product, mineral oil poses serious environmental threats when used at such a large scale around the globe. Its spillage in water bodies can cause massive damage to aquatic life. For example, in 2010 when Deepwater Horizon oil spill happened in off-coast in the Gulf of Mexico during which around 210 million US gallons of petroleum oil was spilled causing extensive damage to the marine and wildlife habitats. A study about the spill was conducted in 2013 and it was reported that various aquatic species which were exposed to the spill developed heart related deformities that would be expected to be fatal [6]. Similarly, in 1989 the infamous Exxon Valdez spill happened in Alaska in which around 40,000 cubic meters, i.e., 10.5 million gallons were spilled in the waters of Alaska causing massive damage to marine life. Likewise, in electrical power systems, explosions caused by inflammable mineral oil in transformers have led to major black-outs every now and then. All these factors along with the depleting natural resources of mineral oil are forcing the researchers to look for alternative oils that can be used in transformers. Alternative vegetable oils have shown promising applications in electrical power systems, especially for dielectric purposes in transformers. Various vegetable based oils like rapeseed oil, sunflower oil coconut oil, palm oil, etc., have been successfully tested for their insulating properties [1-4, 7]. However, the latest emerging trend in the field of dielectrics which has created a boom in research is to enhance the insulating and thermal properties of these vegetable oils by dispersing suitable nanomaterials in them. For this purpose, various nanomaterials like AL_2O_3 [8], Fe_2O_3 [9], TiO_2, ZnO [10], etc., have been dispersed in vegetable oils to improve their dielectric capabilities. This has enabled the engineers to increase the transformer ratings with great success. Any newly synthesized dielectric fluid should be studied for its insulating properties under controlled thermal ageing conditions before its successful operation in transformers. The effects of thermal ageing play a very important part in the overall behavior of the dielectric as well as the thermal properties of these fluids. It is unanimously reported that over a certain period of usage of these dielectric fluids in transformers, their insulating properties keep on degrading [11, 12]. So, it becomes very important to study in detail the dielectric properties of a newly developed insulating fluid before ageing as well as after subjecting that dielectric fluid to the ageing conditions in the laboratory. If the dielectric behavior of the synthesized insulating fluid after ageing is within the acceptable limits as specified by the IEC standards for dielectric oils for insulation purposes in transformers, only then the synthesized fluid should be used in transformers.

In this research, one of the latest and in-trend nanomaterials, i.e., graphene oxide has been chosen for the synthesis of dielectric nanofluid. Often called ‘the wonder material’, graphene oxide is being studied in almost every field of research, however its contribution in insulating systems is yet to be explored. In this research, a soyabean extracted vegetable oil and mineral oil mixed blend has been dispersed with graphene oxide (GO) nanoparticles. The developed oil samples have been subjected to ageing conditions for different time periods in order to understand the behavior of developed nanofluids in transformers over a period of time. The dielectric and physio-thermal properties of aged oil samples have been studied and compared with the newly developed unaged oil samples. Results show that the overall insulating properties of nanofluids were enhanced even after the ageing period and thus enabling the usage of GO dispersed vegetable oil based nanofluid in transformers.

II. MATERIALS

A. Synthesis of Nanomaterials

GO nanomaterials were synthesized by using modified Hummer’s method. Equal amounts of powdered graphite and sodium nitrate ($NaNO_3$), about 0.5 g each, were mixed into 23 ml of conc. sulfuric acid (H_2SO_4). The complete mixing was done in an ice bath in order to maintain the solution’s temperature less than 10°C after which the reaction was continuously and uniformly stirred for almost 4 hours. After mixing the reaction for the said time period, it was added with about 3 g of potassium permanganate ($KMnO_4$). The new mixture was again stirred uniformly for 1 hour. The entire reaction was transferred into an ice-bath over a magnetic stirrer and 110 ml of distilled H_2O was put into it. While stirring, 5 ml of 30% hydrogen peroxide (H_2O_2) was put in a dropwise fashion into the solution after which the entire reaction was centrifuged with 5% hydrochloric acid (HCl) and ethanol. The leftover residue was kept in a hot air oven till dried to obtain the powdered graphene oxide nanoparticles. To limit the phenomenon of agglomeration of nanoparticles when dispersed in a polar solvent, the nanoparticles are generally surface coated with either stearic or oleic acids. This weakens down the intermolecular forces between the nanoparticles and delays the process of agglomeration. This process is called surface modification of nanoparticles [13]. In this research, synthesized GO nanoparticles were surface modified with oleic acid and the process is explained as follows. 0.5 g of synthesized GO nanoparticles were dispersed into 10 ml of ethanol. The resulting mixture was sonicated for 40 minutes. This resulted in the formation of a homogenous slurry in which 0.3 g of oleic acid (OA) was added. The entire mixture was put on refluxing conditions at 60°C for 30 minutes. The solution was centrifuged, and the ethanol was distilled away leaving behind the precipitate of GO nanoparticles coated with oleic acid. This precipitate was dried in an oven to obtain the final product as oleic acid coated graphene oxide (OA-GO) nanoparticles.

B. Synthesis of Insulating Fluids

Vegetable oil extracted from soyabean crop and conventional mineral oil were purchased commercially with their dielectric parameters meeting the IEC 60296:2003 standards. A vegetable based oil-blend was prepared by mixing the soyabean oil and mineral oil in 80:20 proportions respectively. The reason behind the choosing the volume ratio to be 80:20 is that it has been repeatedly shown in literature that the optimal dielectric performance of mixed oil blends is achieved when the vegetable oil and the mineral oil are mixed in volume proportions of 80:20 [14]. The uniformity in the mixing of both oils was achieved with the help of sonication and magnetic stirrers. The oil-blend was labelled on the basis of percentage of vegetable oil in it, i.e., OB80. Synthesized OA-GO nanoparticles were dispersed in OB80 in five different concentrations of 0.001, 0.003, 0.005, 0.007 and 0.009 g/L in order to make five nanofluid samples having different concentrations of GO nanoparticles. The nanofluid samples were named NF1, NF3, NF5, NF7 and NF9 respectively.

III. EXPERIMENTAL APPROACH

A. Ageing of Nanofluids

The oil blend OB80 and the NFs were aged as per the IEC 60076-7 specifications for accelerated ageing of transformer oils in different ambient temperatures. Six ageing vessels were filled with the oil samples, one with OB80 and the other five with NFs having different concentration values. The ageing vessels were filled only up to 3/4th of their total capacity to let the air remain in the vacant space to match the transformer like conditions. To simulate more approximate transformer-like environment, one pressboard strip and one Kraft paper strip having dimensions 70*10 mm were immersed in each ageing vessel as per the specifications set by IEC 60641-1 standards of transformer solid insulating materials. To replicate the effect of transformer winding on ageing of transformer oil, one copper strip of length 20 mm and diameter 2 mm was also immersed in each ageing chamber. The chambers were air-sealed and put into the hot air oven set at a temperature of 130°C. Oil samples from each ageing vessel were collected on 15th day and 60th day of the starting of ageing process. The analysis of oil samples at 0 days, 15 days and 60 days of ageing corresponds to the study of behavior of these samples at raw unaged conditions, during the onset of the thermal stress conditions and after being subjected to the extreme thermal stress conditions for a prolonged time respectively. The various insulating and physio-thermal properties of these aged oil samples were studied and compared with the respective unaged oil samples. The effect of nanoparticles on the ageing of base oil-blend

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and the nanofluid samples was also studied. The synthesized GO nanoparticles as well the aged and unaged oil samples along with the pressboard and Kraft paper strips immersed in ageing vessel are shown in Fig 1. For simplicity, only OB80 and NF9 samples have been shown in Fig 1.

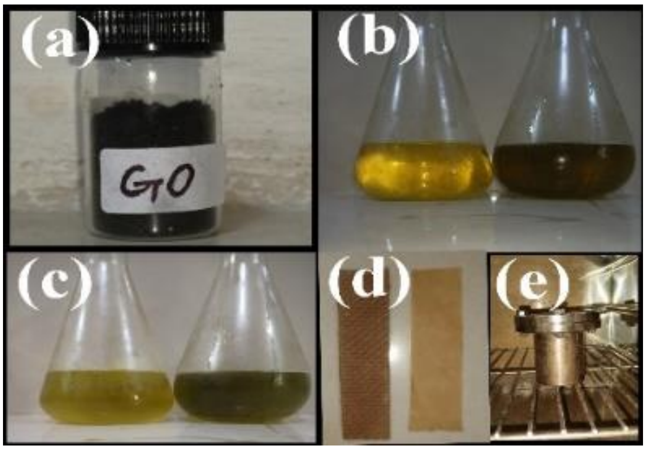

Fig. 1. (a) Synthesized GO nanoparticles, (b) aged and unaged OB80, (c) aged and unaged NF9 sample, (d) pressboard and Kraft paper strip and (e) ageing vessel.

B. Characterization of Nanoparticles

Various characterization techniques were employed to study the structural details as well as the lattice arrangements of the synthesized graphene oxide nanoparticles. The chemical composition of unmodified and modified nanoparticles was also studied using different characterization techniques. X-Ray Diffraction (XRD) method was employed to identify the phases as well as to determine the crystallinity of GO nanoparticles. The structural insight of synthesized nanoparticles was obtained using Fourier Transform Infra-Red (FTIR) spectroscopy. It helped in understanding the various fingerprints of different chemicals and functional groups attached with unmodified as well as modified nanoparticles. The thermal decomposition of surface modified GO nanoparticles was investigated using Thermogravimetric Analysis (TGA). It helped in understanding the behavior of nanoparticles at various temperature values that can be attributed to the temperature range of any typical working transformer, thus providing information about the thermal stability of the compound in the transformer oil. Raman spectra were used to investigate the D and G bands of OA-GO nanoparticles and to calculate the band intensity ratio of the synthesized compound.

C. Insulating Properties of Nanofluids

Various insulating and physio-thermal characteristics of unaged and aged oil samples were studied and compared using corresponding IEC and ASTM standards for transformer insulating oils. Ac breakdown voltage (BDV) was measured using IEC 60156 specifications in which a semi-automatic kit having highest voltage limit of 100 kV and a rising rate of 2 kV/s was used. Two spherical electrodes with a 2.5 mm gap were installed in 400 ml test cell and the values were recorded at 25°C and 90°C. A total of 15 readings were recorded and the mean values were recorded as the resultant values.

Number The relative permittivity, or dielectric constant, and the dissipation factor, or the tangent delta, of the oil samples were measured according to the IEC 61620 and ASTM D924 standards. The values were measured at 25°C and 90°C to correspond to the temperature values of a transformer under different loading conditions. The interfacial tension (IFT) of the oil samples was investigated to study their hydrophobic capabilities. IFT values were recorded at 25°C using Du-Nouy Principle fulfilling the ASTM D0971-99 specifications. The flash point values of oil samples were recorded using ASTM D93 standards according to which the oil samples were continuously heated in an air tight chamber until a burning flash could be seen and the corresponding temperature was recorded. In order to understand the cooling behavior of the aged oil samples, their viscosities were measured at 25°C and 90°C as per the ASTM D-445 specifications. The applied shear rate was varied from 6/s to 120/s with uniform numerical intervals of 6 and the average viscosity of the oil samples was measured at shear rate of 50.5/s.

IV. RESULTS AND DISCUSSIONS

A. Characterization of Nanoparticles

XRD: A typical graphene oxide sample shows a conventional XRD peak somewhere between 2θ=10° and 2θ=11° [15]. These planar values suggest the overall enhancement in the interplanar distance between the graphene oxide nanoplatelets. The distance can be a result of the increase in the number of oxygenated groups attached in the process of transformation of graphene oxide pristine graphite. However, in XRD spectra of synthesized GO as shown in Fig 2a, a peak at 2θ=14° can be seen with the planar values of d002 = 0.63 nm. This 3° shift in the diffraction peak of synthesized GO from a typical peak value is caused due to the extra thermal reduction of GO during the process of its synthesis. The second peak at 2θ=27° is due to the leftover graphite residue whereas the third peak at 2θ=43° is due to the GO nanoparticles undergoing turbostratic disorder.

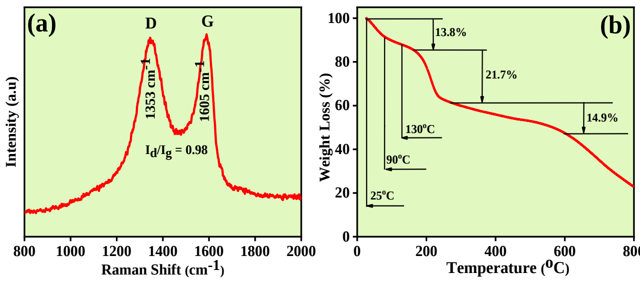

Fig. 2. (a) The XRD spectrum of synthesized GO and (b) FTIR spectra of synthesized GO and OA-GO.

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FTIR: The FTIR spectra of synthesized and surface modified is shown in Fig 2b. In synthesized GO, the stretching vibrations in OH hydroxyl group are indicated by a wide band ranging from 3400 cm^{-1} to 2400 cm^{-1}. These vibrations occur because of absorption of H_2O molecules [16]. The leftover graphite residue stays unoxidized in the final product and causes a peak to appear at 1620 cm^{-1} because of its C=C stretching mode. C=O as well as C-OH stretches of carboxyl and alcoholic groups are represented by the peaks at 1727 cm^{-1} and 1170 cm^{-1} respectively. The stretching vibrations of C-O bond in C-O-C group are represented by the diffraction peak at 1050 cm^{-1}. When compared to the OA-GO, a couple of extra peaks can be seen at 2920 and 2850 cm^{-1} which appear because of the CH stretching mode of different C_nH_{2n+1} chains. Diffraction peaks in OA-GO have less intensity as compared to the peaks in GO which means that the vibrations in OA-GO are comparatively less.

Raman Spectroscopy: A conventional Raman spectrum for pure graphene oxide nanoparticles shows a D band peak around 1350 cm^{-1} and a G band peak around 1600 cm^{-1} [17]. The former signifies the structural defects whereas the latter signifies the in-plane vibrations of sp^2 C-atoms. However, as can be seen in Fig 3a, the measured D value of synthesized OA-GO nanoparticles is seen at 1353 cm^{-1} which can be said to be appearing slightly earlier on x-axis when compared to the conventional D band value. The G band is seen at 1605 cm^{-1} which also appears slightly earlier on x-axis as compared to the conventional G band peak value, however, it lies more closer to it than the recorded D band value. The degree of disorder, structural defects as well as the purity of the synthesized OA-GO can be estimated using the band intensity ratio which is given by the ratio I_d:I_g and is calculated to be 0.98 in this case. In carbon based products, the higher value of this ratio means more structural disorders in the product.

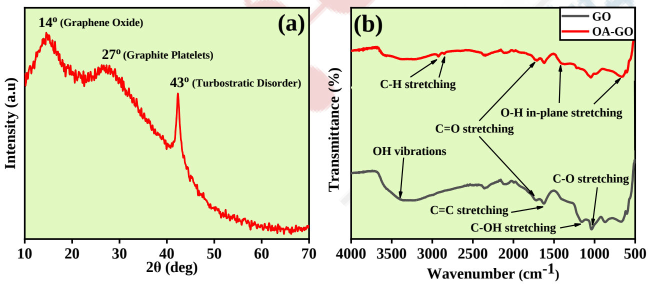

Fig. 3. (a) Raman spectroscopy of OA-GO and (b) TGA of OA-GO nanoparticles.

Thermo-gravimetric Analysis (TGA): Fig 3b shows the TGA of OA-GO nanoparticles. Synthesized OA-GO shows thermal stability throughout the entire temperature range of any working transformer as is shown by the weight loss of nanoparticles at 25°C and 90°C. The nanomaterials show a slight decrease in their weight around 200°C, however, this temperature value lies beyond the maximum possible temperature in a transformer during overloading conditions.

B. Insulating Properties of Nanofluids

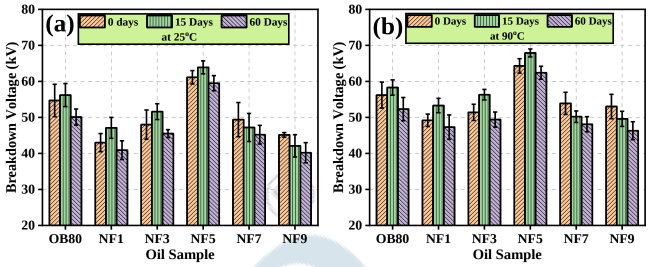

Fig. 4. (a) and (b) BDV of new and aged oil samples at 25°C and 90°C, respectively.

Ac BDV and Impulse BDV: The ac BDV of OB80 and NFs has been shown graphically in Fig 4. At room temperature conditions of 25°C, the BDV value of newly synthesized unaged base oil OB80 is measured to be 54.6 kV. After being subjected to 15 days of ageing period, the BDV of this sample increases to 56.3 kV. As the ageing process is continued for 60 days, the BDV value reduces to 50.2 kV. A comparable behavior of enhanced BDV after 15 days and reduced BDV after being put under ageing conditions for 60 is shown by the NF samples having comparatively lower concentration values. The BDV values of higher concentration NF samples, i.e., NF7 and NF9, started to decrease directly as the ageing period is started. When the BDV of NF samples is measured at a temperature of 90°C, a similar pattern can be seen where the BDV values of lower concentrated NF samples are seen to be enhanced slightly at the end of 15 days and then reduced significantly at the end of 60 days of ageing. At both the temperature values, the maximum BDV after ageing is reported for NF5 sample, i.e., 63.9 kV and 59.5 kV after the ageing periods of 15 and 60 days respectively when measured at 25°C, and 67.9 kV and 62.4 kV after 15 and 60 days of ageing respectively when measured at 90°C. The percentage decrease in BDV of 60 days aged oil blend and nanofluid samples with respect to corresponding unaged samples has been shown in Table 1.

Table 1. Percentage Decrease in BDV of 60 Days Aged Samples with Respect to Corresponding Unaged Samples

Parameter	Percentage Decrease (%)					
	OB80	NF1	NF3	NF5	NF7	NF9
BDV, 25°C	8.4	4.8	5.2	2.6	8.3	10.8
BDV, 90°C	6.9	3.8	3.9	2.9	10.7	12.6
IBDV, 25°C	7.3	9.1	2.8	2.9	9.5	13

Relative Permittivity: The dielectric constant, or relative permittivity values, of aged oil samples follow almost similar trend as that of newly prepared oil samples at corresponding temperatures as shown in Fig 5a and Fig 5b. At 25°C, the maximum value of permittivity for new as well as aged oil samples is recorded for NF7 whereas at the temperature of

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90°C, the maximum value of relative permittivity is attained for NF3 sample which is 2.6. At 90°C, the relative permittivity of 60 days aged OB80 is comparatively at 1.8. These deteriorated permittivity values after ageing of the oil samples show a direct dependency upon the permittivity values of the newly synthesized oil samples with almost similar rate of deterioration at corresponding temperature values.

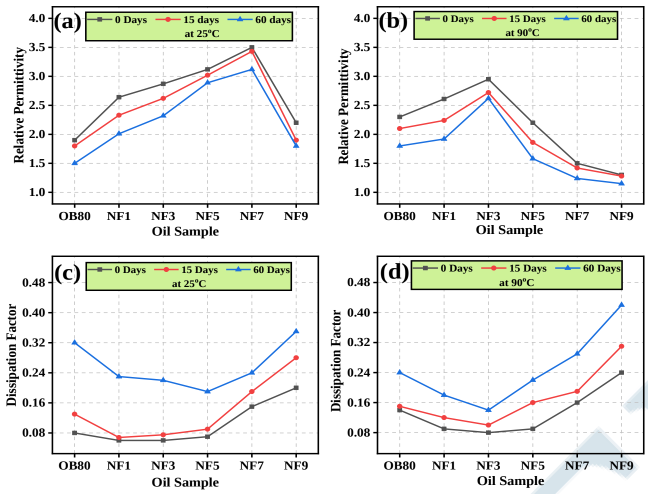

Fig. 5. (a) and (b) Relative permittivity of unaged and aged oil samples at 25°C and 90°C respectively, (c) and (d) Dissipation factor of unaged and aged oil samples at 25°C and 90°C, respectively.

Dissipation Factor: The dissipation factor of aged oil samples at temperature of 25°C show a slight distortion in the expected trend when compared to the dissipation factor of newly synthesized oil samples as can be seen in Figure 5c and 5d. Whereas, the minimum dissipation factor value for new oil samples at 25°C was measured for NF3 sample, this peak shifts to NF1 sample after 15 days of ageing and then to NF5 sample after 60 days of ageing. However, at 90°C, the dissipation factor values of aged oil samples follow the similar trend as newly synthesized oil samples with the minimum values falling for NF3 samples in both cases.

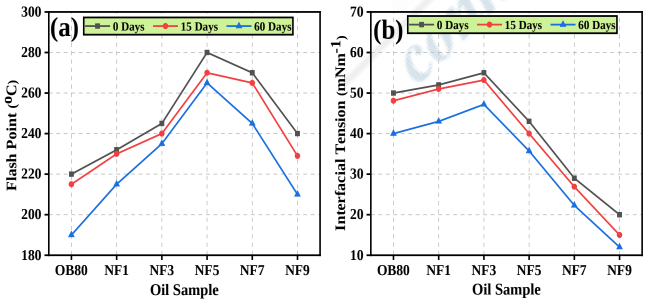

Fig. 6. (a) Flash point of new and aged oil samples, (b) IFT values of new and aged oil samples.

Flash Point: The flash point of OB80 sample, as shown in Fig 6a, falls drastically from 220°C of newly synthesized sample to 190°C of 60 days aged sample. The flash point of NF samples also decreases with ageing, however, at lower concentration values, the decrease in the flash point of NF samples is comparatively quite lesser. The flash points of NF1, NF3 and NF5 samples decreased by only 17°C, 10°C and 15°C respectively as compared to the decrease of 30°C in case of OB80 sample. With the further increase in concentration values of GO in the oil-blend, the flash point also started to decrease considerably with 25°C and 30°C decrease in NF7 and NF9 samples.

Interfacial Tension: The interfacial tension values of new and aged oil samples are shown in Fig 6b. IFT values of aged oil samples also show some deterioration as compared to new oil samples but only after showing a slight increase in the IFT values of oil samples after ageing of 15 days. However, the IFT values of aged NF samples having lower concentration of GO nanoparticles, i.e., NF1 to NF5, still fall within the acceptable limits if IFT values for any useable transformer insulating oil.

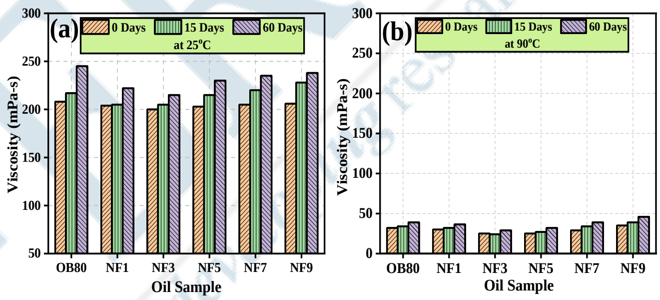

Fig. 7. (a) and (b) Viscosity of new and aged oil samples at 25°C and 90°C, respectively.

Viscosity: The viscosity values of new and aged oil samples are measured at 25°C and 90°C and shown in Fig 7a and 7b. With the increase in temperature, the viscosity of oil samples decreased considerably as expected, however, the viscosity of aged oil samples increased at respective temperatures when compared to the viscosity of the newly synthesized oil samples. At 25°C, NF1 and NF3 oil samples show the least increase in their viscosities after an ageing period of 15 and 60 days. At 90°C, the viscosities of NF3 and NF5 samples show the least increase after the ageing periods.

C. Mechanism of Improvement

The significant improvement in the dielectric as well as the physio-thermal properties of the nanofluid samples is caused by the transfer of charge to a streamed path formed by the Gouy-Chapman-Stern (GCS) layer. The layer is formed due to the residual products formed over the period of time at the common boundary, i.e., an interface between the nanoparticles and the oil molecules. As a result of formation of GCS layer, the conductive path via the interface increases which in turn increases the conduction along the interface. This results in an overall increased shallow trap density [18-20]. Higher shallow trap density means higher number of shallow traps being present in the nanofluids. The shallow traps enhance the phenomenon of electron trapping by accumulating the charge carriers. As a result, the de-trapping of electrons takes place at a slower rate in nanofluid samples when compared to that in base oil samples and thus resulting in significantly improved insulating and physio-thermal properties of nanofluids.

D. Design Considerations

It is well established that the temperature difference between the center of the core and the outer surface at a distance of x from the center is given by Equation (1) [21].

$$\theta = \frac{q\rho x^2}{2} \ ^\circ\text{C} \quad (1)$$

$$or,\ x^2 = \frac{2\theta}{q\rho} \quad (2)$$

$$or,\ x^2 = k\theta \quad (3)$$

$$or, x = k\sqrt{\theta} \quad (4)$$

where q is the heat produced per unit volume in W/m^3, ρ is the thermal resistivity of the core along the direction of heat flow in Ωm and k=2θ/q. Since, the temperature reduces in case of more uniform electric stress distribution in solid and liquid insulations, i.e., with the increase in the ratio of liquid to solid insulation permittivity [16], thus θ can be replaced by the term $1/(\epsilon_{liquid} : \epsilon_{solid})$. Thus, Equation (4) becomes,

$$x = k'\sqrt{\frac{1}{\epsilon_{liquid} : \epsilon_{solid}}} \quad (5)$$

Where, ϵ_{solid} is the relative permittivity of pressboard sample impregnated with the corresponding oil. Since, the dielectric stress is more for aged insulation system when compared to new insulation system, so the relative permittivity of the corresponding aged samples measured at 90°C are chosen to calculate the ratios. The relative permittivities of 60 days aged OB80 oil and the pressboard impregnated with OB80 (P-OB) are 1.8 and 4.1 respectively whereas the relative permittivities of 60 days aged NF3 oil and the pressboard impregnated with NF3 (P-NF3) are 2.6 and 5 respectively as discussed in results section. The NF3 sample has been selected since it shows the highest value of the relative permittivity amongst all the aged oil samples at 90°C. Thus, putting the values of liquid to solid permittivity ratio to be 0.36 in case of OB80 and 0.52 in the case of NF3 sample, Equation (5) becomes,

$$x_{OB80} = k'\sqrt{\frac{1}{\epsilon_{OB} : \epsilon_{P-OB}}} = k'\sqrt{(1/0.44)} = 1.5k' \quad (6)$$

$$x_{GNF} = k'\sqrt{\frac{1}{\epsilon_{GNF} : \epsilon_{P-GNF}}} = k'\sqrt{(1/0.52)} = 1.37k' \quad (7)$$

Thus, from the Equations (6) and (7), the relative decrease in percentage can be calculated as,

$$\frac{(1.5k' - 1.37k')}{1.5k'}$$

$$= 8.6\%$$

Thus, the distance from the center of the core can be reduced by 8.6% in the case of NF filled transformer when compared to the OB80 filled transformer.

V. CONCLUSION

Graphene oxide dispersed vegetable based nanofluids with different concentrations have been made to undergo the accelerated thermal ageing process. The dielectric and physio-thermal properties of nanofluid samples have been studied after 15 and 60 days of ageing and the properties have been compared with the newly developed respective nanofluid samples. As expected, the insulating properties of NF samples deteriorated after being subjected to the ageing period of 60 days. However, the most essential dielectric property, i.e., ac BDV of NFs with lower concentration values (NF1, NF3 and NF5) showed a significant increase at the end of the ageing period of 15 days after which the breakdown voltage started to decrease with the increase in ageing period. The rate of deterioration of dielectric and physio-thermal properties of aged NF samples is also reported to be much lesser than in the case of aged base oil OB80 sample. In terms of design consideration, the dispersion of GO nanoparticles in the vegetable oil led to the possibility of reduction of core size by 8.6%, thus making the design more compact. The future scope of this research will include the simulation of a transformer model based upon these equations and verify the results.

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