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AC Dielectric Strength of Mineral Oil-Based Fe_3O_4 and Al_2O_3 Nanofluids

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Abstract: This paper deals with experimental study of the influence of conductive (Fe_3O_4) and insulating (Al_2O_3) nanostructured particles at various concentrations on the dielectric strength of transformer mineral oil. The method of preparation and characterization of these nanofluids (NFs) through the measurements of zeta potential, the real and imaginary parts of dielectric constant as well as the concentration and size of nanoparticles using Scanning Electron Microscope (SEM) images of nanoparticles powders and Dispersive x-ray Spectroscopy (EDS) analysis are presented. Experimental findings reveal that these two types of nanoparticles materials significantly improve AC breakdown voltage and the magnitude of this improvement depends on the concentration, size and nature (material) of nanoparticles. For a given type of nanoparticles, the effect is more marked with the smallest nanoparticles. The conductive nanoparticles offer higher enhancement of dielectric strength compared with insulating nanoparticles based nanofluids. With Fe_3O_4, the breakdown voltage (BDV) can exceed twice that of mineral oil and it increases by more than 76% with Al_2O_3. The physicochemical mechanisms implicated in this improvement are discussed.

Keywords: AC dielectric strength; insulating oils; mineral oil-based nanofluids; statistical analysis; Weibull distribution; normal distribution

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1. Introduction

The search for new materials for electrical insulation increasingly efficient to improve the dielectric strength of components integrated in the systems of transport and distribution of electrical energy and reduce their size, weight and costs is a permanent task. Also, nanotechnologies and more particularly dielectric nano-fluids (NFs) constitute an innovative line of research with a promising potential and future.

The effect of nanoparticles on the electrical properties of dielectric materials in particular on their ability to record the initiation voltage of partial discharges and to slow the propagation of electrical discharges; trees in polymers and streamers in liquids; leading to breakdown is the subject of many studies around the world since a twenty years. It appears from the results reported in the literature that some polymers nano-composites are promising materials for high voltage applications [1-6]. The fact that nano-materials present interesting dielectric characteristics would result from the large volume fraction of interfaces in the bulk of the material and the ensuing interactions between the charged nanoparticle surface and the molecular structure of hosting material. However, if the solid dielectrics provide a function mainly of insulation and mechanical support (equipment envelope, support isolators, bushing, etc.), the liquid dielectrics must ensure the thermal transfer for a better cooling of high voltage components and especially power transformers, in addition to their insulating role.

This cooling property (heat transfer) was at the origin of NFs development. Nowadays, NFs are used in several fields like electronics (chips, electronic circuitry components …); transportation (the cooling systems of heavy duty engines and heat generation parts of automobiles …); heating building and reducing pollution; nuclear cooling systems; space and defense (space stations and aircrafts); solar absorption for their heat-transfer performance. Indeed, they possess better thermal properties (thermal conductivity, thermal diffusivity, convective heat transfer coefficient) than the base fluids. These strongly depend on the volume fraction and properties of the added nanoparticles [7, 8]. Furthermore, for a given volume of particles, the solid-liquid surface contact area between nano-scale particles and the suspension fluid is greater than that for micro-scale particles. Hence, the shape and size of particles have a significant effect on thermal conductivity and heat transfer characteristics [9 - 11].

The volume fraction of particles (concentration), their shape and size, and the surface contact area between particles and liquid, are major parameters that influence not only the thermal properties as indicated above, but also have a very great influence on the dielectric properties of composites materials. It is in search of new liquid dielectrics ensuring these functions that the present study fits.

In recent years, researches on transformer oil-based NFs have been the subject of a particular attention. Much effort has been focused on the dielectric and thermal properties of some NFs for use in power transformers [12 - 18]. It has been reported that NFs especially magnetic NFs present better AC and DC breakdown voltages and thermal conductivity than base oils [19-21]. Segal et al [13] observed that the positive impulse breakdown voltage of these types of NFs is almost twice of the base transformer oil. Dhar et al. [22] reported that the addition of traces of graphene or nanotubes carbon (NTC) improves the AC dielectric breakdown strength of insulating mineral oils by about 70 to 80%. G. D. Peppas observed that a NF of surface coated Fe_3O_4 nanoparticles improve AC BDV of natural ester (FR3) and mineral (Shell Diala) oils. However, some authors reported contradictory results [23]. They observed that AC breakdown voltage with conducting particles as Fe2NiO4 decreases when concentration of nanoparticles increases.

This paper is aimed at the influence of two types of nanoparticles (insulating and conductive) namely Fe_3O_4 and Al_2O_3, on AC dielectric strength of transformer mineral oil. We first present the preparation of nanofluids and then some characteristics such as zeta potential to check the stability of NFs, real and imaginary parts of dielectric constant. The concentration and size of nanoparticles using Scanning Electron Microscope (SEM) images of nanoparticles powders and Dispersive x-ray Spectroscopy (EDS) analysis are also conducted. Then, the results of AC breakdown voltage measurements of mineral oil-based Fe_3O_4 and Al_2O_3 nanofluids are presented and the physicochemical processes implicated in breakdown phenomena of nanofluids are discussed.

2. Experiment

2.1 Preparation and characterization of nanofluids samples

The nanoparticles used in this study are supplied from Sigma-Aldrich and have a purity of 99.9%. These are conductive (Fe_3O_4), and insulating (Al_2O_3). Their mean sizes are 50 nm for Fe_3O_4, and 13 and 50 nm for Al_2O_3. Figure 1 gives the distribution of various nanoparticles we used in mineral oil. It was measured by using particle size analyzer (NanoPlus, Particulate Systems, USA). Scanning Electron Microscope (SEM) images show that these nanoparticles powders seem to be spherical and regular in shape even if some aggregates can be observed as shown in Figure 2a. Moreover, the used species are subjected to energy dispersive x-ray spectroscopy (EDS) analysis which is used for elemental analysis of a sample as shown in Figure 2b. The EDS analysis confirms the presence of each sample composition at atomic percentage. The characteristic parameters of the basic mineral oil we used are given in Table 1.

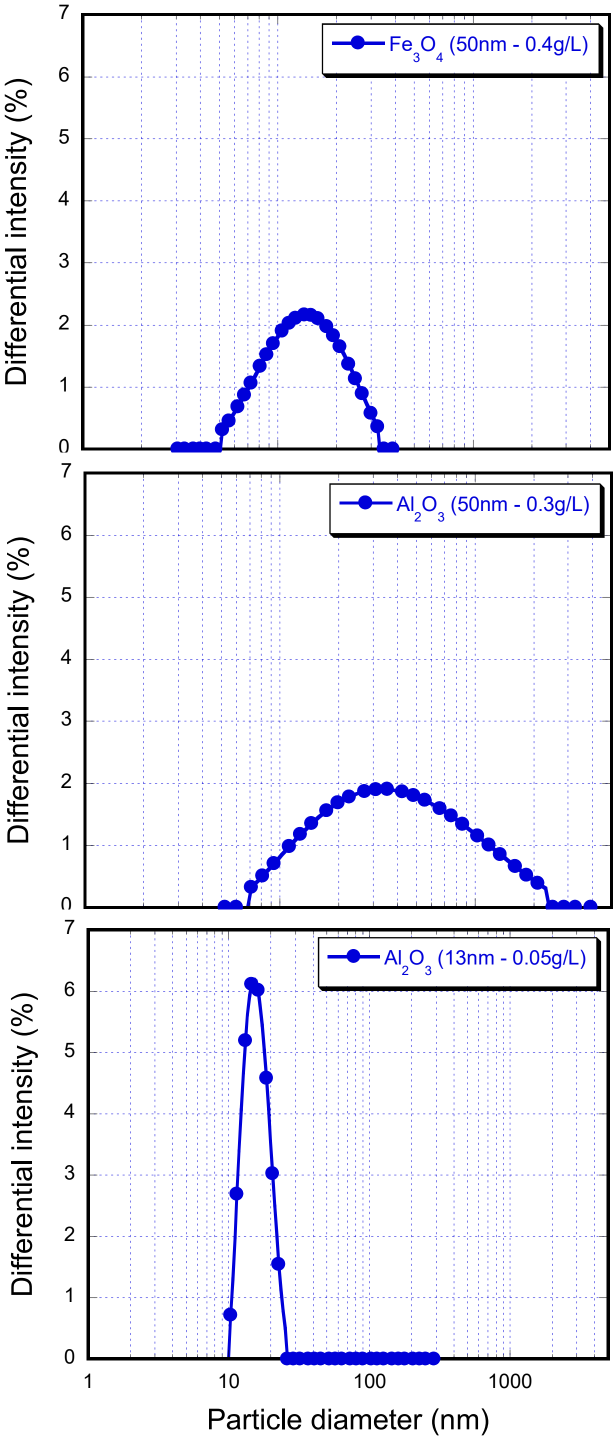
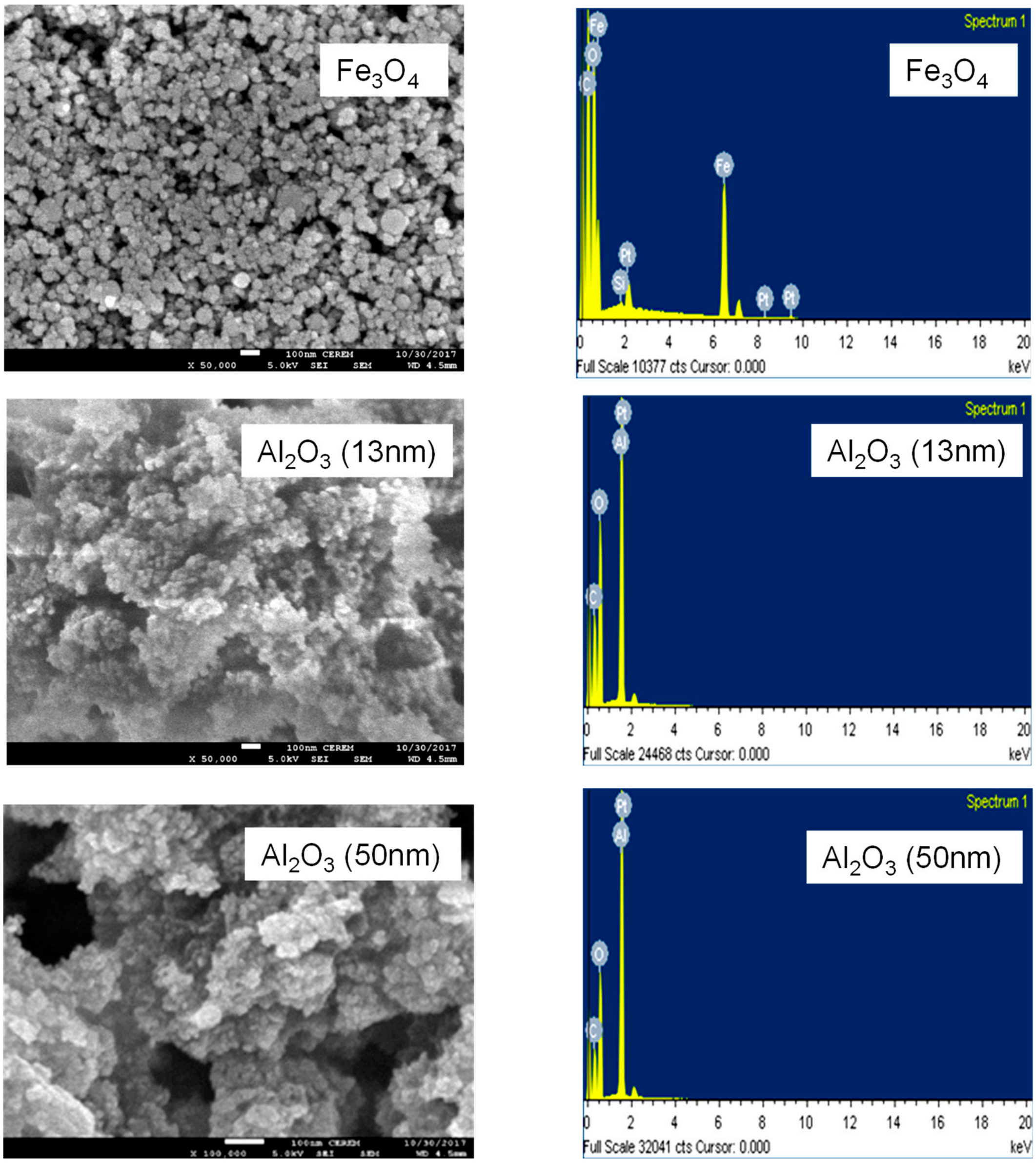


Figure 1. Particles size analysis of nanofluid samples

Note that because of aged mineral oil we used, its water content is higher than that of the fresh oil which generally does not exceed 8 ppm. This fact would affect the experimental results. However, as all nanofluid samples are prepared with the same base oil, the comparison of our results/comparison will be done on the same basis.

Mineral-oil nano-fluids were prepared by dispersion of nanoparticles in concentration ranging from 0.05 to 0.4 g/L. After the magnetic stirring process for 30 min, the NFs samples are submitted to ultra-sonication process (i.e.; NFs are placed in the ultrasonic homogenizer) for 2 h to avoid agglomeration and clusters due to attractive and repulsive forces.



(a) (b)

Figure 2. (a) The Scanning Electron Microscope and (b) EDS images of nanoparticles.

Table 1. Physicochemical properties of mineral oil

Property	Mineral oil
Density at 25°C (g/mL)	0.85
Kinematic viscosity at 40°C (cSt)	9
Pour point (°C)	-40
Flash point (°C)	150
Fire point (°C)	160
Total acid number (mg KOH/g)	<0.5
Antioxidant content	<0.3%
Water content (ppm)	39
Gassing characteristics (mm^3 /min)	-35 to +30
Interfacial tension (mN/m)	40~60
Resistivity (Ω.m)	>3*10^9
Dissipation factor at 90°C	0.1-0.5%

In order to reach a stable suspension of nanoparticles in oil, further ultrasonification was applied for only 2 min duration using Sonics Vibra-cell sonicator; 750 W power rating, 20 kHz capability, and 0.5 inch probe; to avoid reunion of nanoparticles without need of adding any surfactant. Then, all samples are kept under vacuum of 0.16 MPa for 24 hours to eliminate humidity and internal micro bubbles formed during the ultrasonication process.

The stability of NFs we prepared has been checked through the measurements of zeta potential (*ζ-potential*), by using Malvern Zeta sizer nano ZS 90-UK, which is a key indicator of the stability of dispersions. The magnitude of ζ-potential indicates the degree of electrostatic repulsion between adjacent, similarly charged particles in dispersion. For particles that are small enough, a high zeta potential will confer stability, i.e., the dispersion will resist aggregation. Figure 3 gives zeta potential for four NFs and mineral oil for comparison. It was observed that the absolute value of ζ-potential increases with the concentration of nanoparticles. And for a given concentration (0.3 g/L for instance), this value is higher for Al_2O_3 nanoparticles than that for Fe_3O_4.

The measurements of electrical conductivity shows that the Fe_3O_4 based NF are higher than that of Al_2O_3, for the same concentration of nanoparticles as shown in Figure 4. This is due to the fact that Fe_3O_4 is more conducting than Al_2O_3 which is known as a good insulating material.

Figures 5 depicts the real and imaginary parts of investigated NF versus the frequency up to 100 Hz. These measurements are performed by HIOKI-LCR meter IM3536, Japan. We observe that the real part of dielectric constant decrease abruptly in the range 0 – 40 Hz and then tends to be constant.

At 50 - 60 Hz what corresponds to industrial frequencies, the real part of dielectric constant of nanofluid samples is practically the same as that of the base oil (mineral oil). Similarly, it's happened with the imaginary part of dielectric constant.

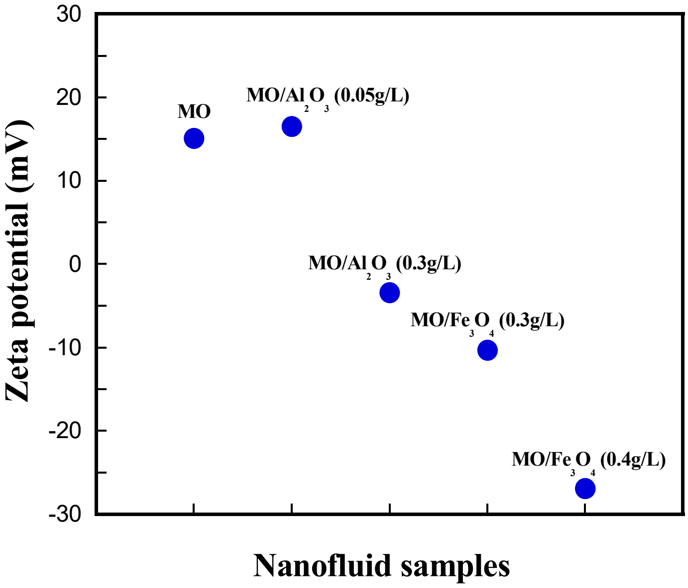


Figure 3. Zeta potential of the tested nanofluids

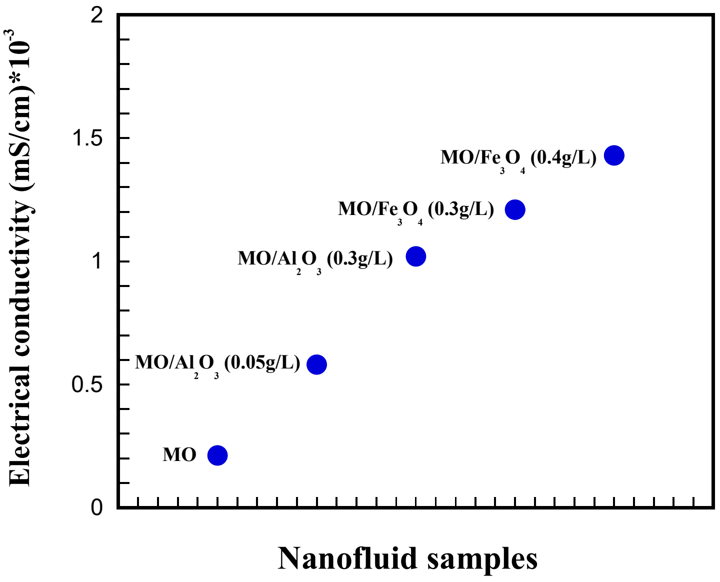
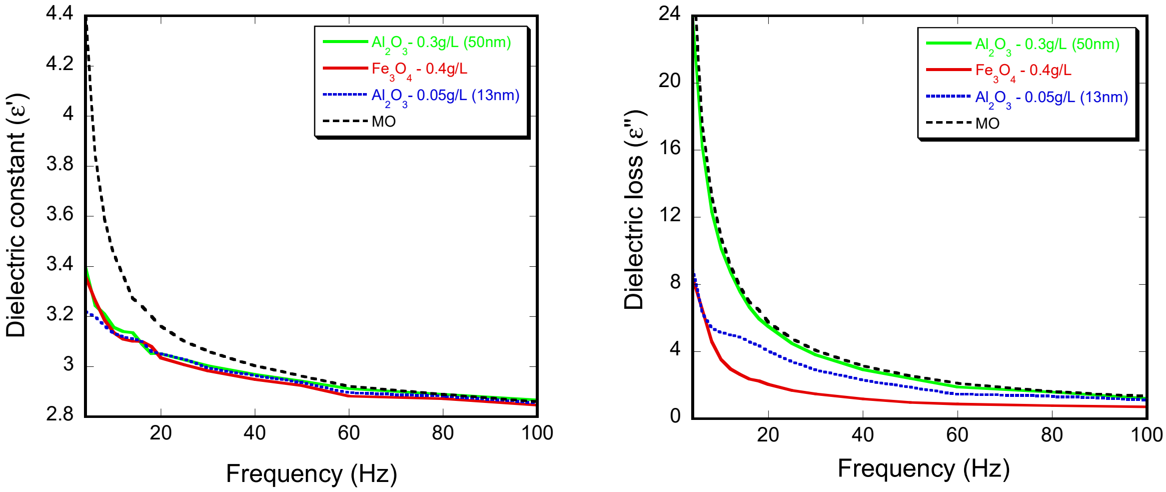


Figure 4. Electrical conductivity of tested nanofluids



(a) (b)

Figure 5. (a) Real and (b) imaginary parts of dielectric constant *vs*. frequency of investigated nanofluids

2.2 Breakdown measurement

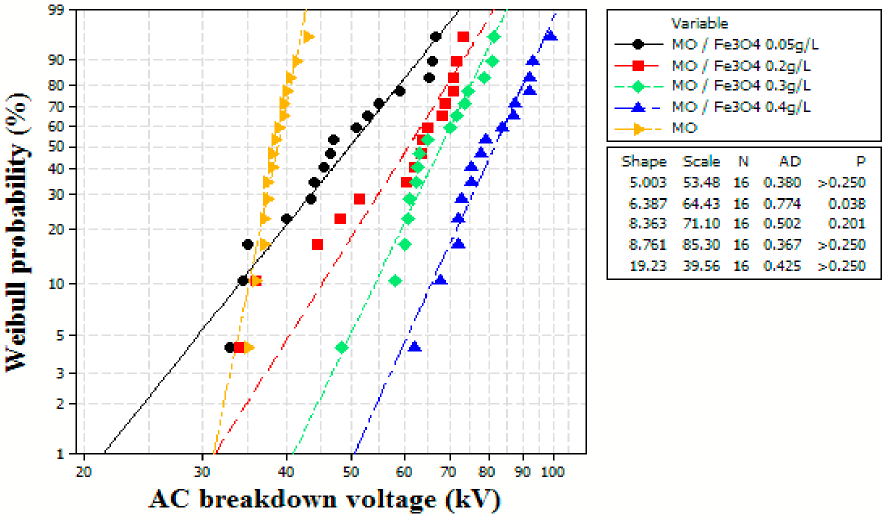
The dielectric breakdown (BDV) measurements were achieved according to the IEC 60156 standard [24] and using a test cell of 500 mL volume and an oil tester (Foster Oil Test 90 type). The electrode arrangement consists of two brass hemispheres of 12.5 mm diameter, separated by a distance of 2.50 ± 0.05 mm. The test cell and electrodes were also prepared according to IEC 60156 specifications. The voltage is continuously applied at the electrodes at a uniform rise rate of 2 ± 0.2 kV/s until breakdown occurs. The breakdown voltage is the average of 16 successive measurements and the time delay between successive measurements is 2 min.

3. Experimental results

Figures 6 to 8 show the Weibull plots of the AC breakdown voltage of mineral oil-based Fe_3O_4 and Al_2O_3 nanofluids for different concentrations of particles and size. In addition, Tables 2 to 4 give the

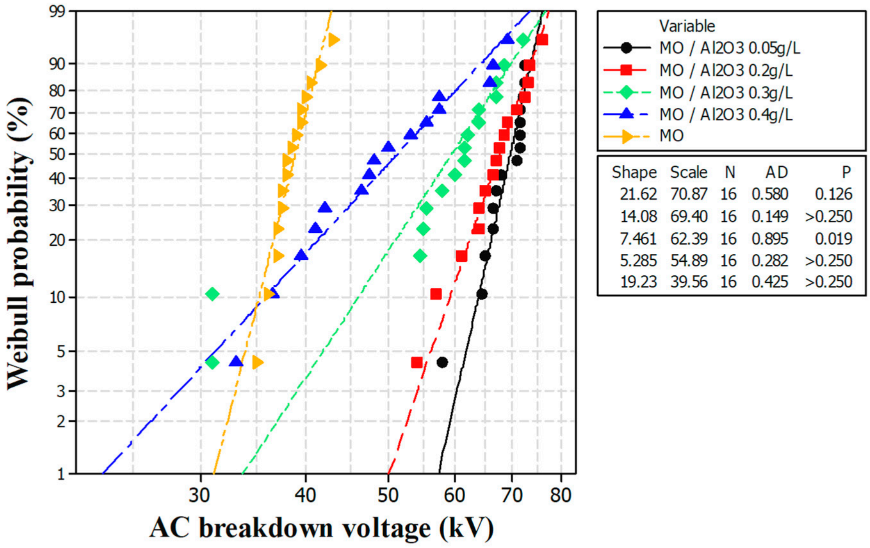
AC breakdown voltage at 1%, 10% and 50% breakdown probabilities for the three investigated NFs. Further, these tables show the incremental percentage of the mineral oil with various concentrations of nanofluids. Breakdown voltage at 50% cumulative probability is an indication to the mean value. The breakdown voltage at 10% cumulative probability is an estimate to the lowest possible breakdown voltage, and so, gives an indication about the reliability of oil. Generally, BDV of nanofluids are higher than that of mineral oil whatever the type and size of nanoparticles.

It was observed that BDV of mineral oil-based Fe_3O_4 increases with the concentration of nanoparticles as shown in Figure 6. With a concentration of 0.4 g/L, the average BDV of NF exceeds twice that of mineral oil alone (Figure 9). This is of a great interest for oil-filled apparatus especially for power transformer. While for mineral oil-based Al_2O_3 with nanoparticles of 13 nm, the average BDV increases up to a maximum value (optimal) which is reached at a small concentration of 0.05 g/L; this increase is of 72% with respect to that of mineral oil and then decreases but remaining higher than that of mineral oil (Figure 10). While with nanoparticles of 50 nm, the optimal average BDV is reached at a higher concentration of 0.3 g/L and the increase is of 69% (Figure 11).



Shape	Scale	N	AD	P
5.003	53.48	16	0.380	>0.250
6.387	64.43	16	0.774	0.038
8.363	71.10	16	0.502	0.201
8.761	85.30	16	0.367	>0.250
19.23	39.56	16	0.425	>0.250

Figure 6. Weibull plot of the average reading of breakdown voltage of MO/Fe_3O_4 NFs



Shape	Scale	N	AD	P
21.62	70.87	16	0.580	0.126
14.08	69.40	16	0.149	>0.250
7.461	62.39	16	0.895	0.019
5.285	54.89	16	0.282	>0.250
19.23	39.56	16	0.425	>0.250

Figure 7. Weibull plot of the average reading of breakdown voltage of MO/Al_2O_3 (13 nm) NFs

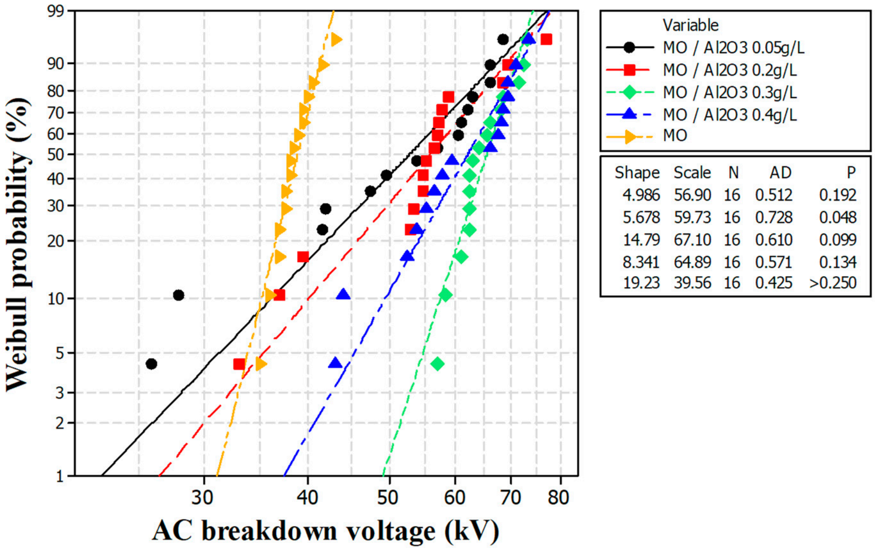


Figure 8. Weibull plot of the average reading of breakdown voltage of MO/Al_2O_3 (50 nm) NFs

Table 2. AC breakdown voltage at different breakdown probabilities of MO/Fe_3O_4

	MO	0.05 g/L		0.2 g/L		0.3 g/L		0.4 g/L	
BDV probability (%)	BDV (kV)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)
1.0	31.1	21.3	-31.6	31.3	0.5	41.0	31.7	50.5	62.2
10.0	35.2	34.1	-3.1	45.3	28.7	54.3	54.3	66.0	87.5
50.0	38.8	50.0	28.8	60.8	56.7	68.0	75.2	81.0	108.7

Table 3. AC breakdown voltage at different breakdown probabilities of MO/Al_2O_3 (13nm)

	MO	0.05 g/L		0.2 g/L		0.3 g/L		0.4 g/L	
BDV probability (%)	BDV (kV)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)
1.0	31.1	57.3	84.0	50.0	60.6	33.6	7.9	23.0	-26.2
10.0	35.2	63.9	81.4	59.1	67.9	46.1	31.1	35.9	1.8
50.0	38.8	69.7	79.6	67.6	74.2	59.4	53.1	51.2	31.9

Table 4. AC breakdown voltage at different breakdown probabilities of MO/Al_2O_3 (50nm)

	MO	0.05 g/L		0.2 g/L		0.3 g/L		0.4 g/L	
BDV probability (%)	BDV (kV)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)
1.0	31.1	22.7	-27.1	26.5	-14.9	49.1	57.7	37.4	20.1
10.0	35.2	36.2	2.8	40.2	14.2	57.6	63.6	49.5	40.6
50.0	38.8	52.9	36.3	56.0	44.3	65.5	68.8	62.1	60.0

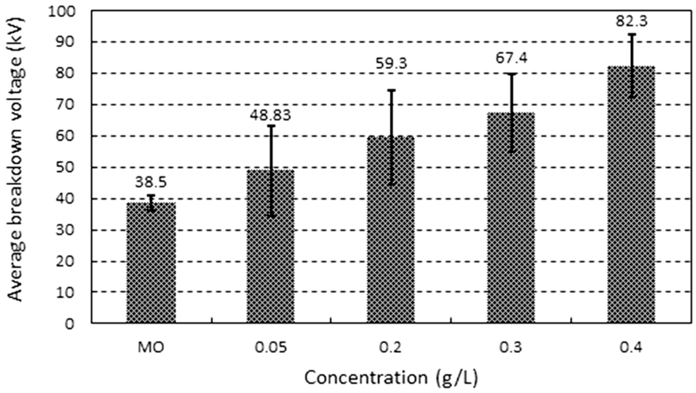


Figure 9. The breakdown voltages of MO/Fe_3O_4 NFs

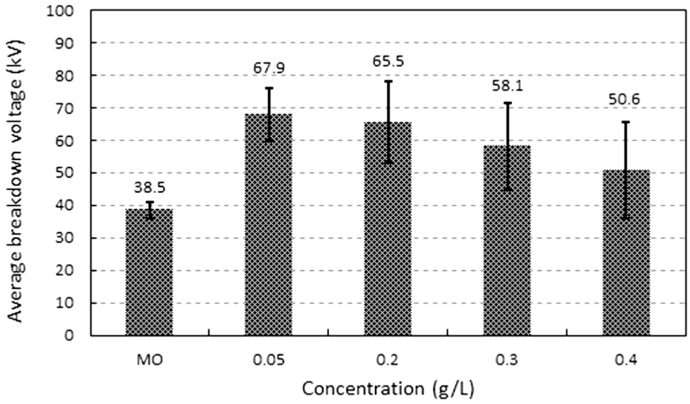


Figure 10. The breakdown voltages of MO/Al_2O_3 (13 nm) NFs

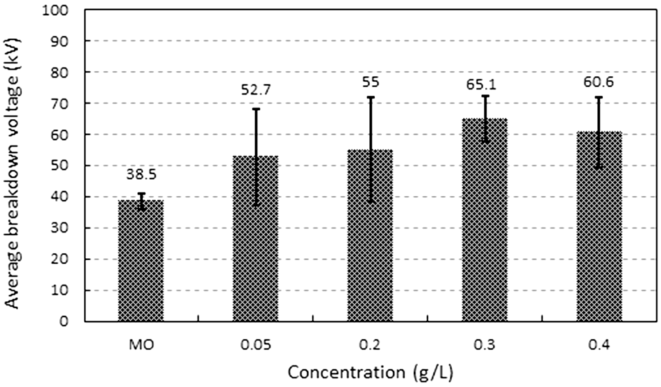


Figure 11. The breakdown voltages of MO/Al_2O_3 (50 nm) NFs

4. Discussion

Table 5 gives a comparison of the investigated NFs for different types of nanoparticles and concentrations. It was observed that at concentration of 0.05 and 0.20 g/L, the highest average breakdown voltage is obtained with Al_2O_3 nanoparticles of 13 nm; the average BDV is increased by 76.6% with respect to mineral oil. While with Al_2O_3 nanoparticles of 50 nm, the increase is of 36.3 % for the same concentration. This increase is only 28.9% with Fe_3O_4.

With concentrations of 0.3 and 0.4 g/L, the maximum average BDV is obtained with Fe_3O_4 nanoparticles. The improvement is of 75.3% and 108.8% with respect to mineral oil, respectively. Table 4 presents a comparison of different NFs of different volume concentrations with mineral oil. The fact that with the same kind of nanoparticles and concentration, the average BDV is higher with smaller nanoparticles is due to the large volume fraction of interfaces in the bulk of the material and the ensuing interactions between the charged nanoparticle surface and the liquid molecule.

The increase of breakdown voltage of liquid (MO) when adding some amounts of nanoparticles results of their influence on the physicochemical processes evolving during pre-breakdown phase that are mainly the conduction, the initiation and/or propagation of streamers. The question is: How and which mechanism(s)? In the following, we analyze the possible implication of each one of these mechanisms.

Table 5. Incremental of different additives material with different volume concentrations compared with mineral oil

	MO	Fe_3O_4	Al_2O_3 (50nm)	Al_2O_3(13nm)
BDV with increment (%)			MO / 0.05 (g/L) NFs	
	38.8	50.0	52.9	**69.7**
	-	28.8%	36.3%	**79.6%**
			MO / 0.2 (g/L) NFs	
	38.8	60.8	56.0	**67.6**
	-	56.7%	44.3%	**74.2%**
			MO / 0.3 (g/L) NFs	
	38.8	68.0	65.5	**59.4**
	-	75.2%	68.8%	**53.1%**
			MO / 0.4 (g/L) NFs	
	38.8	81.0	62.1	**51.2**
	-	108.7%	60.0%	**31.9%**

If the nanoparticles (NPs) act on the conduction, they likely act as charge carriers' scavengers. Thus, by reducing the number of charges by trapping and consequently the total space charge, the liquid becomes less conductive and sees its breakdown voltage increased. Figure 4 seems to contradict that hypothesis since the conductivity of liquid increases when increasing the amount of nanoparticles. Therefore the nanoparticles would act on the streamers phenomena. The influence of electronic scavenger additives (especially halogenated molecules) has been investigated by many researchers and it was established unanimously that such additives accelerate the streamer propagation velocity [25- 28]. However, the interpretations concerning the effect of this streamer acceleration on the breakdown voltage were contradictory. Most of authors deduced that the fact that the streamers are more rapid leads to the decrease of breakdown voltage [25]. Beroual [27, 29] and Aka and Beroual [30] showed that the electronic scavengers' additives increase the initiation threshold voltage of streamers and then, the voltage being higher and therefore its energy: the streamer being more energetic, its velocity will be higher [27].

In our case, the nanoparticles would act at two levels: (1) at the electrodes interfaces by constituting a barrier which reduces the injected charges into the liquid and their mobility. Therefore, the initiation threshold voltage of streamers increases and the breakdown voltage will be higher; and (2) at the nanoparticles/hosting liquid interfaces by constituting double layers. The charge carriers will be trapped up to saturation. The fact that there is an optimum concentration of NPs is likely due do to some saturation of NPs/hosting oil interfaces. On the other hand, the dielectric constant (real part) of Fe_3O_4 being significantly higher than that of Al_2O_3 results in higher surface charge with Fe_3O_4 NPs/base oil. Fe_3O_4 being more conductive, it can be easily charged and polarized than Al_2O_3. These phenomena (polarization, double layer and trapping) would explain the greater improvement with conductive nanoparticles.

According to Hwang et al [33], nanoparticles with different conductivity or permittivity than those of matrix oil enhance breakdown voltage strength while the insertion of nanoparticles with conductivity and permittivity comparable to those of matrix oil results in the decrease of breakdown voltage. It is difficult to accept such an interpretation because in some cases, beyond a certain concentration (optimal concentration) of nanoparticles, the breakdown voltage decreases.

The role of nanoparticles as electronic scavengers has been also advanced by Peppas et al [31], and Makmud et al [32] to explain the improvement of breakdown voltage. They attributed this improvement to the effective electron scavenging by the nanostructures what results in delaying the development of streamers and reducing their propagation velocity.

The mechanism of trapping electrons has been also proposed by some researchers [23, 34 - 36] to explain the higher breakdown strength of conducting nanofluids compared to base oils. The conductive nanoparticles capture very rapidly fast moving electrons and convert into slow negatively charged nanoparticles resulting in the slowing streamer propagation (i.e.; reduction of streamer velocity) and therefore increasing the breakdown voltage.

As indicated above, such interpretation through the slowing propagation of streamers remains a subject of discussion; the electronic scavenger additives accelerate the propagation of streamers [25-28]. Another possibility which would explain the improvement of breakdown voltage would be the result of electron trapping by nanoparticles: conductive nanoparticles trap electrons by charge induction that is the case of Fe_3O_4 while nonconductive nanoparticles that are Al_2O_3 trap electrons due to polarization [35].

On the other hand, the fact that for the same concentration of nanoparticles, the dielectric strength are higher for smaller nanoparticles is due to the large volume fraction of interfaces (more surfaces for charges accumulation) in the bulk of liquid and the ensuing interactions between the charge nanoparticles surface and the molecular structure of liquid. There are less displacements towards the opposite electrode thus slowing the propagation of streamers.

5. Conclusion

In this work, the AC breakdown voltages of mineral oil-based Fe_3O_4 and Al_2O_3 nanofluids (NFs) were investigated. The preparation and physicochemical characterization of these nanofluids were presented.

It has been highlighted that the AC breakdown voltage of these NFs are higher than that of mineral oil. The magnitude of this improvement depends on the concentration, size and type of nanoparticles. For a given type of nanoparticles, the effect is more marked with the smallest nanoparticles. The conductive nanoparticles (Fe_3O_4) offer higher enhancement of dielectric strength compared to insulating nanoparticles based nanofluids. With Fe_3O_4, the breakdown voltage (BDV) can exceed twice that of mineral oil and it increases by more than 76% with Al_2O_3. The possible mechanisms implicated in the improvement of BDV have been discusses.

Therefore, as they also have very good cooling performances, these nanofluids can be considered for use in high voltage power transformers.

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