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Usama Khaled, Abderrahmane Beroual. AC Dielectric Strength of Mineral Oil-Based Fe₃O₄ and Al₂O₃ Nanofluids. *Energies*, 2018, 11 (12), pp.3505. 10.3390/en11123505 . hal-02127881

HAL Id: hal-02127881

<https://hal.science/hal-02127881>

Submitted on 5 Jun 2019

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Article

AC Dielectric Strength of Mineral Oil-Based Fe₃O₄ and Al₂O₃ Nanofluids

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Received: 27 November 2018; Accepted: 13 December 2018; Published: 15 December 2018



Abstract: This paper deals with an experimental study of the influence of conductive (Fe₃O₄) and insulating (Al₂O₃) nanoparticles 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 permittivity as well as the concentration and size of nanoparticles using scanning electron microscope images of nanoparticles powders and energy dispersive x-ray spectroscopy analysis are presented. Experimental findings reveal that these two types of nanoparticles materials significantly improve AC breakdown voltage and the magnitude of this enhancement depends on the nanoparticle concentration, and the size and nature (material) of nanoparticles. For a given type of nanoparticle, the effect is more marked with the smallest nanoparticles. The conductive nanoparticles offer higher enhancement of dielectric strength compared with insulating nanoparticle based nanofluids. With Fe₃O₄, the breakdown voltage (BDV) can exceed twice that of mineral oil and it increases by more than 76% with Al₂O₃. The physicochemical mechanisms implicated in this improvement are discussed.

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

1. Introduction

The search for increasingly efficient materials of integrated components in electric power transmission and distribution systems to improve the dielectric strength, and reduce their size, weight, and cost is a permanent task. Also, nanotechnologies and, more particularly, dielectric nanofluids (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 for the past 20 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 results from the large volume fraction of interfaces in the bulk of the material and the ensuing interactions between the surface of the charged nanomaterial and the molecular structure of the 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 power transformers especially, 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 power machines and heat generation parts of vehicles); heating buildings and reducing pollution; nuclear cooling systems; space and defense (space stations and aircrafts); and solar absorption for heat-transfer performance. Indeed, they have preferable better thermal characteristics (thermal diffusivity, thermal conductivity, convective coefficient of heat transfer) than the base fluids. These strongly depend on properties and the volume fraction of the added nanomaterial [7,8]. Furthermore, for a given particles volume, the contact area of the solid-liquid surface between nano-size particles and the suspension fluid is greater than that for micro-size particles. Therefore, the shape and size of particles has a clear impact on heat transfer and thermal conductivity 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 composite materials. It is in search of new liquid dielectrics ensuring these functions that the present study fits.

In recent years, research on transformer oil-based NFs have been the subject of 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. Qi Wang et al. [22] observed that the positive lightning impulse breakdown voltage of three types of transformer oil-based Al_2O_3 , TiO_2 , and Fe_3O_4 nanofluids increases with the concentration of nanoparticles up to a critical value of concentration; beyond this concentration, it starts decreasing. The improvement of negative impulse breakdown voltages was less than that of pure transformer oil. An enhancement of 33% to 36.1% with Al_2O_3 and 38.8% to 50% of Fe_3O_4 of pure mineral oil has been reported by Madavan et al. [23]. Dhar et al. [24] reported that the addition of traces of graphene or carbon nanotubes (CNT) improves the AC dielectric breakdown strength of insulating mineral oils by about 70% to 80%. Peppas et al. [25] observed that an NF of surface coated in Fe_3O_4 nanoparticles improves the AC BDV of natural ester (FR3) and mineral (Shell Diala) oils. However, some authors have reported contradictory results [26]. They observed that the AC breakdown voltage, with the conducting particles as Fe_2NiO_4 , decreases when the concentration of nanoparticles increases.

This work is aimed at investigating the effect of two kinds of nanoparticles (insulating and conductive), namely Fe_3O_4 and Al_2O_3 , on the AC dielectric strength of transformer mineral oil. We first present the preparation of nanofluids and then some characteristics, such as the zeta potential, to check the stability of NFs, and the real and imaginary parts of dielectric permittivity. The concentration and size of nanoparticles using scanning electron microscope images of nanoparticles powders and energy dispersive x-ray spectroscopy 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 the breakdown phenomena of nanofluids are discussed.

2. Experiment

2.1. Preparation and Characterization of Nanofluid Samples

The nanoparticles used in this study were supplied from Sigma-Aldrich and have a purity of 99.9%. These were conductive (Fe_3O_4) and insulating (Al_2O_3). Their mean sizes were 50 nm for Fe_3O_4 , and 13 nm and 50 nm for Al_2O_3 . Figure 1 gives the distribution of the various nanoparticles we used in mineral oil. It was measured by using a particle size analyzer (NanoPlus, Particulate Systems, USA). Scanning electron microscope (SEM) images show that these nanoparticles were near spherical in shape even if some aggregates can be observed as shown in Figure 2a. Figure 2b shows the peaks corresponding to the species present in the nanoparticles as evidenced by energy dispersive X-ray spectroscopy (EDS) analysis.

The EDS analysis confirms the presence of each sample composition at the atomic percentage. The characteristic parameters of the basic mineral oil we used are shown in Table 1.

Note that because of the 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 were prepared with the same base oil, the comparison of our results/comparison will be done on the same basis.

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

In order to reach a stable suspension of nanoparticles in oil, further ultrasonification was applied for only a 2 min duration using a Sonics Vibra-cell sonicator (750 W power rating, 20 kHz capability, and 0.5 inch probe) to avoid reunion of nanoparticles without the need of adding any surfactant. Then, all samples were moved into a vacuum chamber of 0.16 MPa for 24 h for drying and removal of internal micro bubbles formed during the ultrasonication process.

The stability of NFs we prepared was checked through the measurements of the zeta potential (ζ -potential), by using Malvern Zeta sizer nano ZS 90-UK, which is a key indicator of the stability of suspensions. The magnitude of the ζ -potential indicates the degree of electrostatic repulsion between adjacent, similarly charged particles in a dispersion. For particles that are small enough, a high zeta potential refers to higher stability and, consequently, the dispersion becomes more stable without agglomerations. Table 2 gives the zeta potential for four NFs and mineral oil for comparison. It was observed that the absolute value of the ζ -potential increases with the concentration of nanoparticles. Additionally, for a given concentration (0.3 g/L for instance), this value was higher for Al_2O_3 nanoparticles than that for Fe_3O_4 .

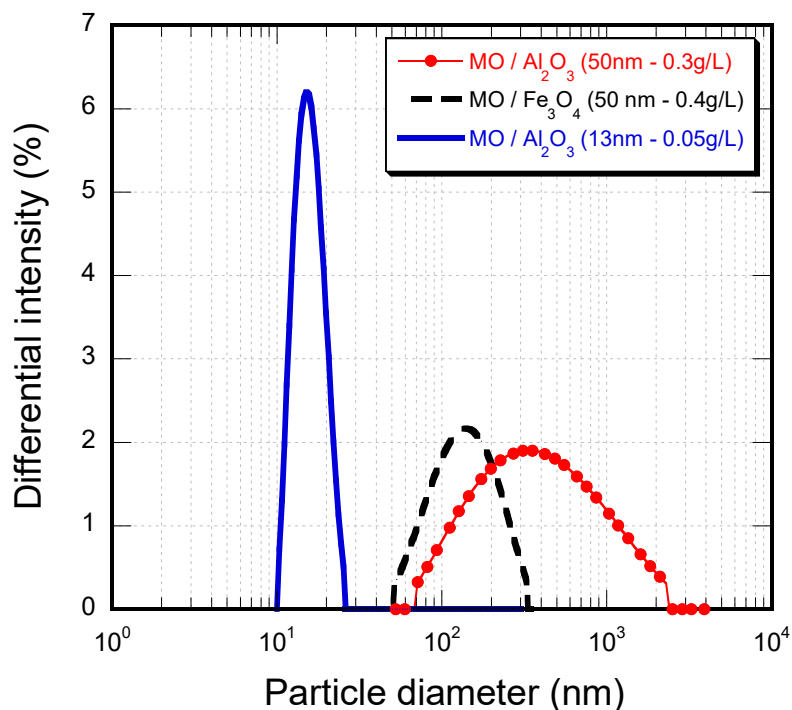


Figure 1. Particles size analysis of nanofluid samples.

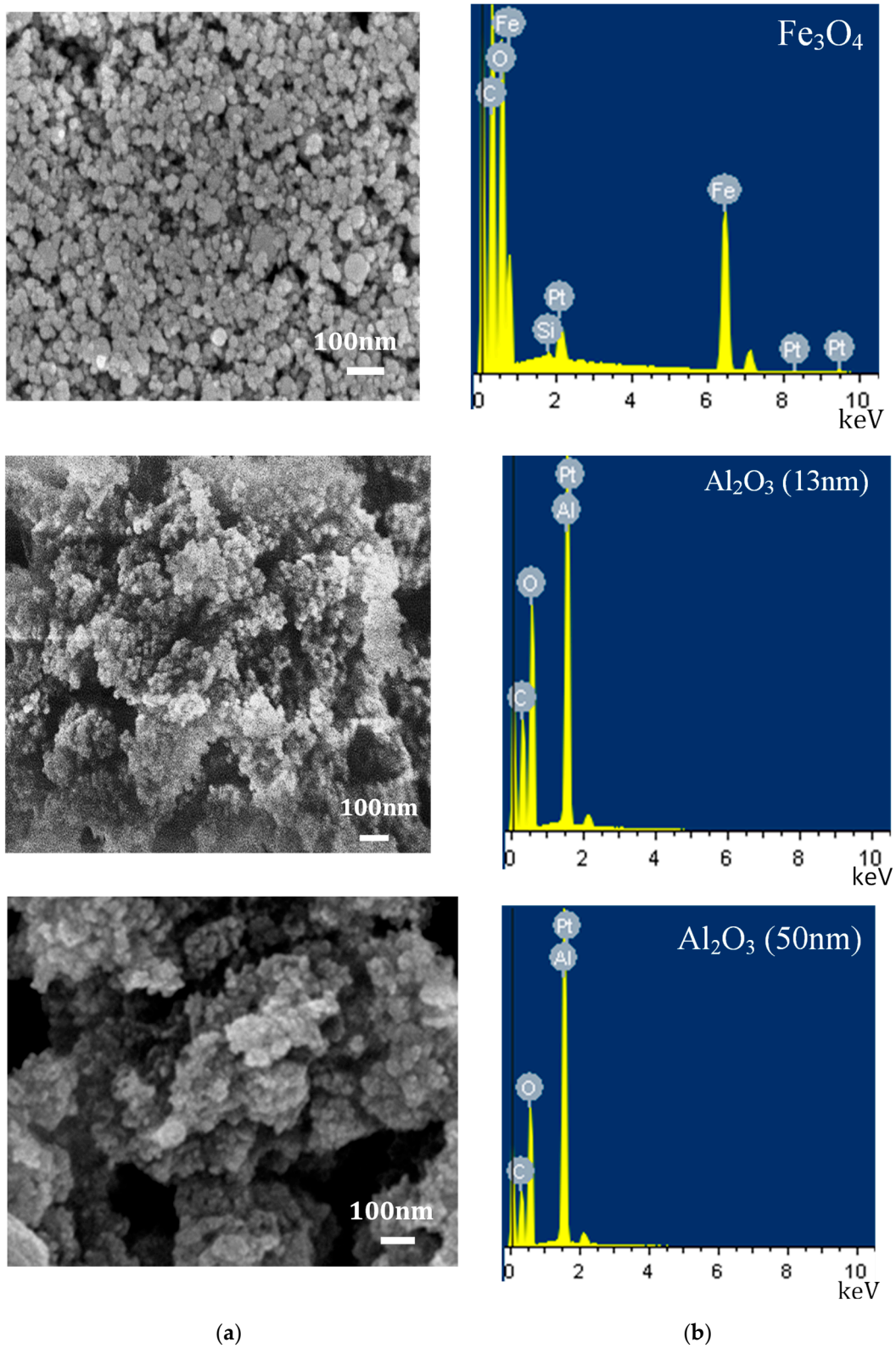


Figure 2. (a) Scanning electron microscope (SEM) and (b) EDS images of nanoparticles.

Table 1. Physicochemical properties of mineral oil.

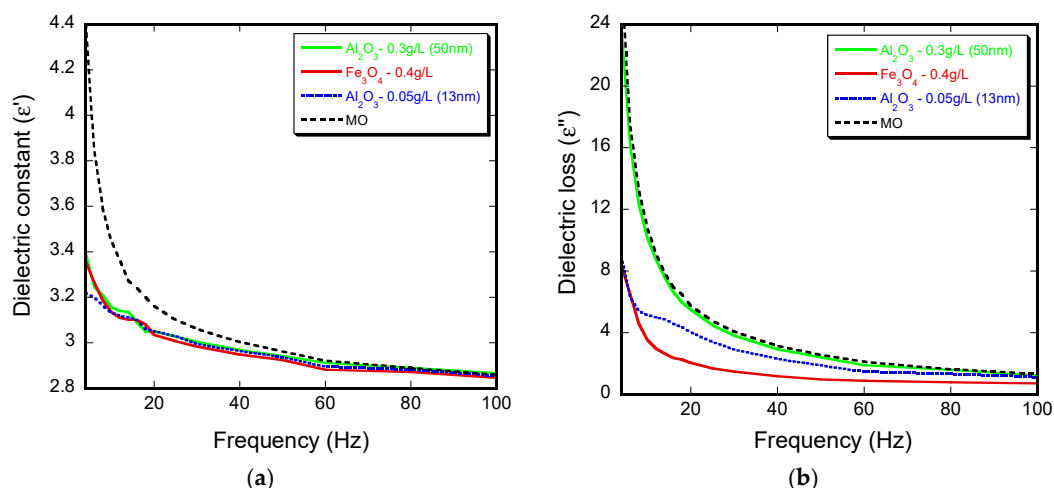
Property	Mineral Oil
Density at 25 °C (g/mL)	0.85
Kinematic viscosity at 40 °C (cSt)	9
Pour point (°C)	−40
Flash point (°C)	150
Fire point (°C)	160
Total acid number (mg KOH/g)	<0.5
Antioxidant content	<0.3%
Water content (ppm)	39
Gassing characteristics (mm ³ /min)	−35–30
Interfacial tension (mN/m)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

Table 2. Zeta potential and electrical conductivity of the tested nanofluids.

Tested Nanofluids	Zeta Potential (mV)	Electrical Conductivity (mS/cm) × 10 ^{−3}
MO	+15.1	0.212
MO/Al ₂ O ₃ (0.05 g/L)	−3.44	1.02
MO/Al ₂ O ₃ (0.3 g/L)	+16.5	0.58
MO/Fe ₃ O ₄ (0.3 g/L)	−10.3	1.21
MO/Fe ₃ O ₄ (0.4 g/L)	−26.9	1.43

The measurements of electrical conductivity show that the Fe₃O₄ based NF were higher than that of Al₂O₃ for the same concentration of nanoparticles, as shown in Table 2. This was due to the fact that Fe₃O₄ conducts more than Al₂O₃, which is known as a good insulating material.

Figure 3 depicts the real and imaginary parts of the dielectric permittivity of the investigated NF versus the frequency up to 100 Hz. These measurements were performed by a HIOKI-LCR meter IM3536, Japan. We observed that the real part of the dielectric permittivity decreases abruptly in the range of 0 Hz–40 Hz and then tends to be constant.

**Figure 3.** (a) Real and (b) imaginary parts of the relative dielectric permittivity vs. the frequency of the investigated nanofluids.

At 50 Hz–60 Hz, which corresponds to industrial frequencies, the real part of the dielectric permittivity of nanofluid samples was practically the same as that of the base oil (mineral oil). Similarly, this was also observed with the imaginary part of the dielectric permittivity.

2.2. Breakdown Voltage Measurement

The breakdown voltage (BDV) measurements were achieved according to the IEC 60156 standard [27] and using a test cell of a 500 mL volume and an oil tester (Foster Oil Test 90 type). The electrode arrangement consisted of two copper hemispheres of a 12.5 mm diameter; the gap distance between the electrodes was kept at 2.50 ± 0.05 mm. Both electrodes and the test cell were also prepared according to the IEC 60156 specifications. The voltage was continuously applied at the electrodes at a uniform rise rate of 2 ± 0.2 kV/s until the breakdown occurred. The breakdown voltage was the average of 16 successive measurements and the time delay between successive measurements was 2 min. Sixteen (16) is a proper number for a Weibull statistical analysis. This enables us to have a power of two ($2^4 = 16$) and to deduce the slope of the Weibull graphs [28].

3. Experimental Results

Figures 4–6 show the Weibull probabilities 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, Table 3 gives the AC breakdown voltage at breakdown probabilities of 1%, 10%, and 50% for the three investigated NFs, which were read from the distinctive Weibull plots. Further, these tables show the incremental percentage of the mineral oil with different concentrations of nanofluids. The breakdown voltage at the 1% cumulative probability is estimated to be the minimum possible breakdown voltage, and so, refers to the reliability of oil. Generally, the BDV of nanofluids were higher than that of the pure mineral oil whatever the type and size of nanoparticles.

The indications within the inserts are: (1) The shape parameter that is equal to the slope of the line in a probability plot, which affects the shape of the curve; (2) the scale parameter, which is related to the scattering of the data, and indicates the degree of failure; (3) the Anderson–Darling (AD) value that is the Anderson–Darling measure of the area between the fitted line and the empirical distribution function, which is based on the data points; (4) N is the number of breakdown voltage data points; and (5) the p -value is a probability that measures the evidence against the null hypothesis.

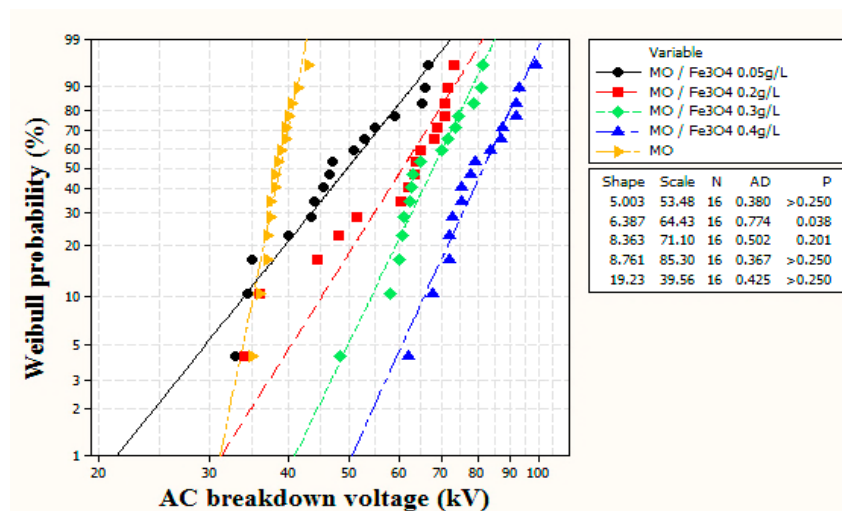


Figure 4. Weibull probability of the average reading of the breakdown voltage of MO/ Fe_3O_4 NFs.

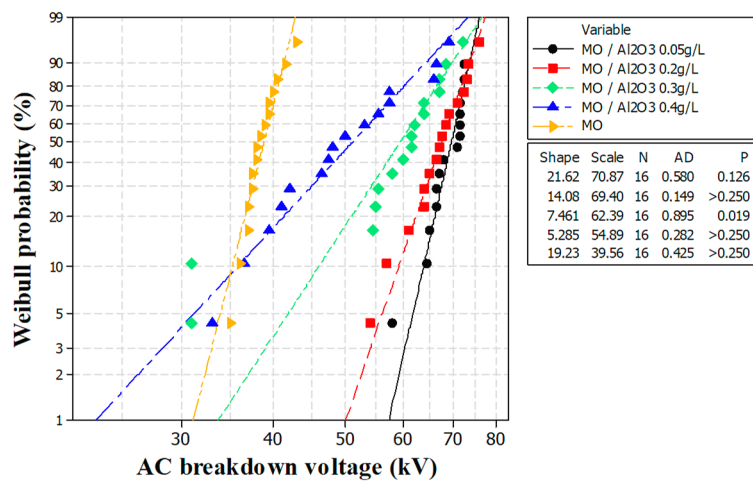


Figure 5. Weibull probability of the average reading of the breakdown voltage of MO/Al₂O₃ (13 nm) NFs.

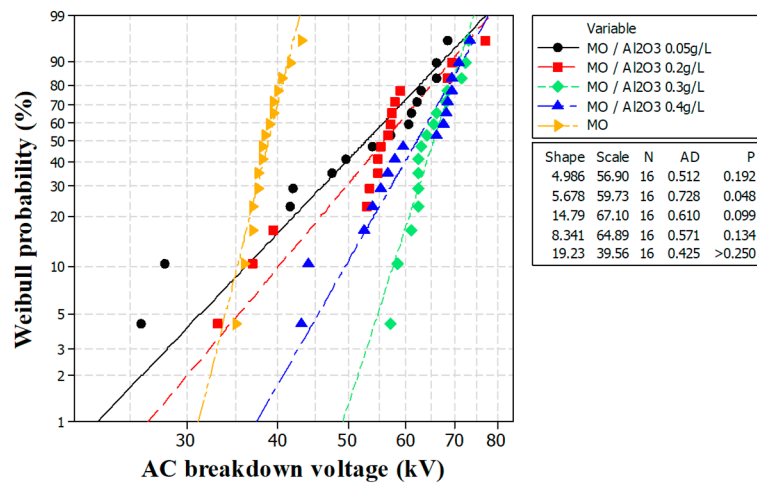


Figure 6. Weibull probability of the average reading of the breakdown voltage of MO/Al₂O₃ (50 nm) NFs.

Table 3. AC breakdown voltage at different breakdown probabilities of various nanofluids.

BDV Probability (%)	MO		0.05 g/L		0.2 g/L		0.3 g/L		0.4 g/L	
	BDV (kV)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	BDV (kV)	Increment (%)	
MO/Fe₃O₄										
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	
MO/Al₂O₃ (13 nm)										
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	
MO/Al₂O₃ (50 nm)										
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	

It was observed that the BDV of mineral oil-based Fe_3O_4 increased with the concentration of nanoparticles as shown in Figure 4. With a concentration of 0.4 g/L, the average BDV of NF exceeds twice that of mineral oil alone (Figure 7). This is of a great interest for the oil-filled apparatus, especially for the power transformer. While for mineral oil-based Al_2O_3 with nanoparticles of 13 nm, the average BDV increased up to a maximum value (optimal), which was reached at a small concentration of 0.05 g/L; this increase is of 72% with respect to that of mineral oil and then decreased, but remained higher than that of mineral oil (Figure 8). While with nanoparticles of 50 nm, the optimal average BDV was reached at a higher concentration of 0.3 g/L and the increase was of 69% (Figure 9). Table 4 summarizes the median and average breakdown voltages of the tested MO/NFs samples.

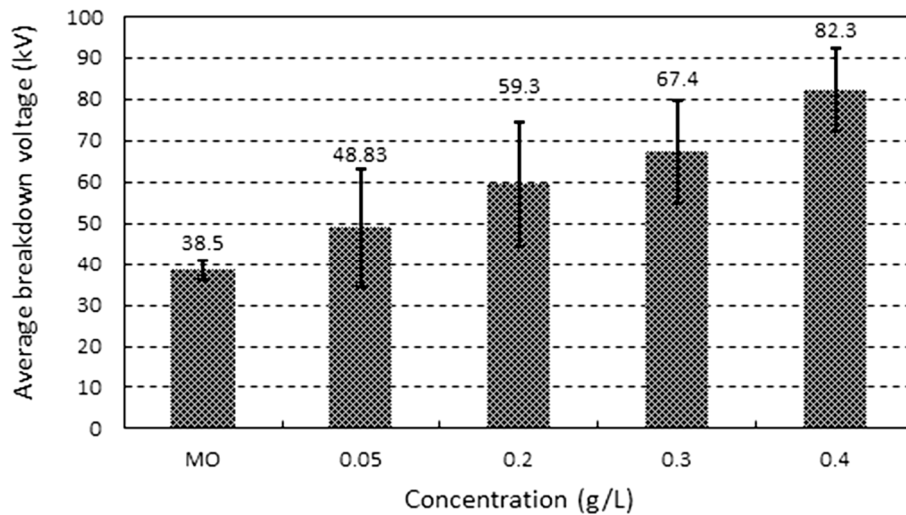


Figure 7. The breakdown voltages of MO/ Fe_3O_4 NFs.

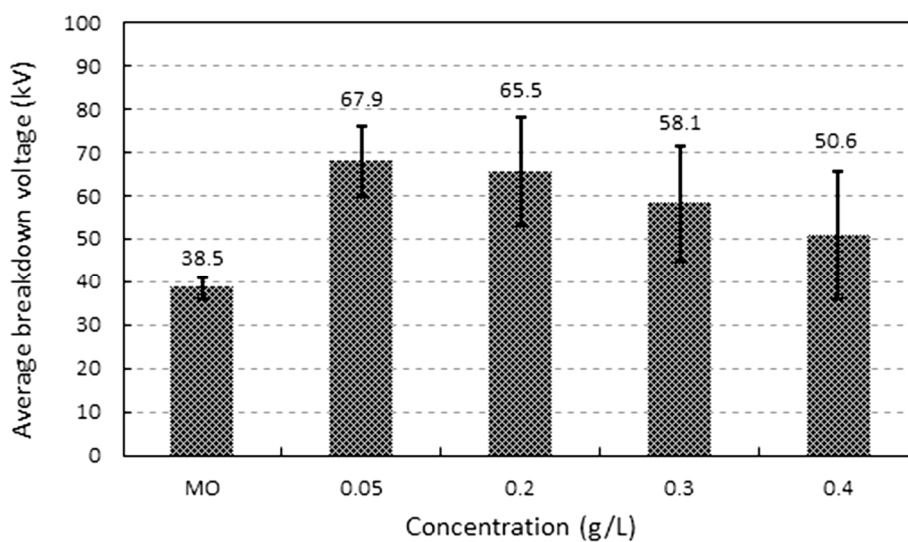


Figure 8. The breakdown voltages of MO/ Al_2O_3 (13 nm) NFs.

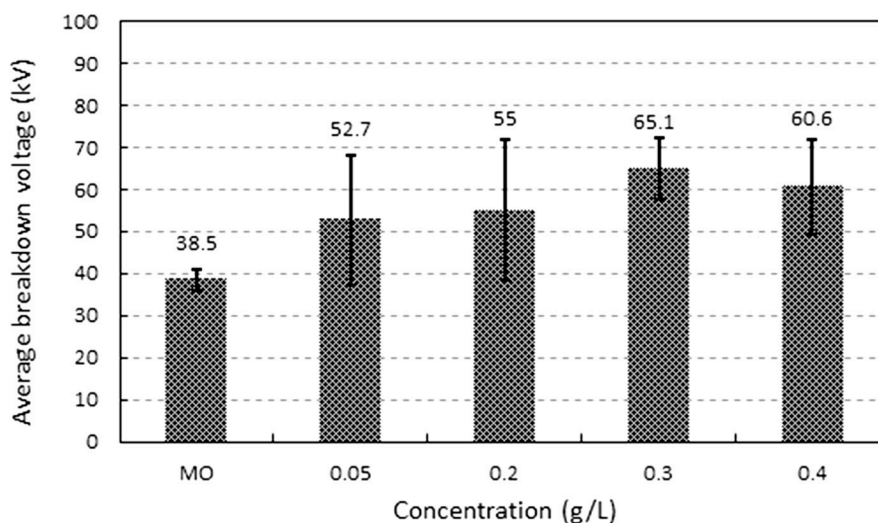


Figure 9. The breakdown voltages of MO/Al₂O₃ (50 nm) NFs.

Table 4. The median and average breakdown voltages of tested MO/NFs samples.

NF Samples and Concentrations	MO	0.05 g/L	0.2 g/L	0.3 g/L	0.4 g/L
Median BDV (50% probability), kV					
MO/Fe ₃ O ₄		50.00	60.80	68.00	81.00
MO/Al ₂ O ₃ (13 nm)	38.80	69.70	67.60	59.40	51.20
MO/Al ₂ O ₃ (50 nm)		52.90	56.00	65.50	62.10
Average BDV, kV					
MO/Fe ₃ O ₄		48.83	59.30	67.40	82.30
MO/Al ₂ O ₃ (13 nm)	38.50	67.90	65.50	58.10	50.60
MO/Al ₂ O ₃ (50 nm)		52.70	55.00	65.10	60.60

4. Discussion

It appears from Table 4 that at concentrations of 0.05 g/L and 0.20 g/L, the highest average breakdown voltage was obtained with Al₂O₃ nanoparticles of 13 nm; the average BDV was increased by 76.6% with respect to mineral oil. While with Al₂O₃ nanoparticles of 50 nm, the increase was 36.3 % for the same concentration. This increase was only 28.9% with Fe₃O₄.

With concentrations of 0.3 g/L and 0.4 g/L, the maximum average BDV was obtained with Fe₃O₄ nanoparticles. The improvement was 75.3% and 108.8% with respect to mineral oil, respectively. Table 3 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 was 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 nanomaterial surface and the liquid molecule.

It was observed that improvements with the same nanoparticles, such as Madawan et al. [23], are two times higher than the ones reported by these authors. Also, our findings confirm those reported by a number of authors [13,19–23] and contradict others [26] regarding concerns of the influence of Al₂O₃ and Fe₃O₄ nanoparticles on the breakdown voltage of mineral oil.

The increase of the breakdown voltage of liquid (MO) when adding some amounts of nanoparticles results from their influence on the physicochemical processes evolving during the 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. 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 its breakdown voltage increases.

Table 2 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 [29–32]. However, the interpretations concerning the effect of this streamer acceleration on the breakdown voltage were contradictory. Most authors deduced that the fact that the streamers are more rapid leads to the decrease of the breakdown voltage [29]. Beroual [31,33], and Beroual and Aka [34] showed that the electronic scavengers' additives increase the initiation threshold voltage of streamers, thus increasing the voltage and therefore its energy: The streamer, being more energetic, results in its velocity also being higher [31].

In our case, the nanoparticles acted 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 increased and the breakdown voltage was higher; and (2) at the nanoparticles/hosting liquid interfaces by constituting double layers. The charge carriers were trapped up to saturation. The fact that there was an optimum concentration of NPs is likely due to some saturation of NPs/hosting oil interfaces. On the other hand, the dielectric permittivity (real part) of Fe_3O_4 being significantly higher than that of Al_2O_3 results in a higher surface charge with the Fe_3O_4 NPs/base oil. Fe_3O_4 , being more conductive, could easily be charged and polarized than Al_2O_3 . These phenomena (polarization, double layer, and trapping) explain the greater improvement with conductive nanoparticles.

According to Hwang et al. [35], nanoparticles with different conductivity or permittivity than those of the matrix oil enhance the breakdown voltage strength while the insertion of nanoparticles with a conductivity and permittivity comparable to those of the matrix oil results in the decrease of the 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. [36] and Makmud et al. [37] to explain the improvement of the breakdown voltage. They attributed this improvement to the effective electron scavenging by the nanostructures, which results in delaying the development of streamers and reducing their propagation velocity.

The mechanism of trapping electrons has also been proposed by some researchers [26,38,39] 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 of the streamer propagation (i.e., reduction of streamer velocity) and therefore increasing the breakdown voltage.

As indicated above, such an interpretation through the slowing propagation of streamers remains a subject of discussion; the electronic scavenger additives accelerate the propagation of streamers [29–32]. Another possibility, which would explain the improvement of the breakdown voltage, is the result of electron trapping by nanoparticles: Conductive nanoparticles trap electrons by charge induction, such is the case of Fe_3O_4 , while nonconductive nanoparticles, which are Al_2O_3 , trap electrons due to polarization [39].

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

5. Conclusions

In this work, the AC breakdown voltages of mineral oil-based Fe₃O₄ and Al₂O₃ 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, and the size and type of nanoparticles. For a given type of nanoparticles, the effect is more marked with the smallest nanoparticles. The conductive nanoparticles (Fe₃O₄) offer a higher enhancement of the dielectric strength compared to insulating nanoparticles based nanofluids. With Fe₃O₄, the breakdown voltage (BDV) can exceed twice that of mineral oil and it increases by more than 76% with Al₂O₃. The possible mechanisms implicated in the improvement of BDV were discussed.

Therefore, these nanofluids not only have very good cooling performances, but they can also be considered for use in high voltage power transformers.

Author Contributions: Both authors contribute to conceptualization; Methodology and Software; Validation; Formal Analysis; Investigation; Writing-Review & Editing; Project Administration and Funding Acquisition, U.K.

Funding: This research was funded by King Saud University grant number ISPP#47.

Acknowledgments: The authors extend their appreciation to the International Scientific Partnership Program (ISPP) at King Saud University for funding this research work through ISPP#47.

Conflicts of Interest: The authors declare no conflicts of interest.

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