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The Effect of Magnetite, Graphene Oxide and Silicone Oxide Nanoparticles on Dielectric Withstand Characteristics of Mineral Oil

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ABSTRACT

In this work, we compare the dielectric withstand performance of nanofluids prepared using Shell Diala D as a base fluid, and magnetite, graphene oxide and silicone dioxide as nanoadditives. The ac withstand capability of the nanofluids was investigated as well as partial discharge inception voltage, partial discharge and repetition rate under ac, dc positive and dc negative voltages. The results indicate that, for all nanofluids, nanoparticle concentrations around 0.2 g/l enhance dielectric withstand properties under quasi uniform fields. Under divergent fields, partial discharge characteristics are improved under ac conditions. Under dc conditions silica nanofluid performs better than mineral oil, but the other two nanofluids do not perform well.

Index Terms — **Power transformer, mineral oil, partial discharge, conductivity, nanofluids.**

1 INTRODUCTION

MINERAL oil (MO) is used in power transformers for both insulation and cooling purposes. Although some manufacturers have started to focalize on esters, MO is still the industry-preferred choice.

Yet, the race for higher power densities in transformers has led researchers around the world to experiment with colloidal suspensions of conductive nanoparticles in oil (nanofluids), with the aim of improving the heat transfer properties of mineral oil [1], [2]. Contrarily to expectations, Segal et al showed that nanoparticles improve the withstand capabilities of mineral oil [3].

A recent paper [4] provides a comprehensive literature survey on the progress made with nanofluids based on transformer oil. It is of particular interest to observe that these improvements were ascribed to the electron trapping capabilities of nanoparticles for both conductive ([5][6]) and semiconductive [7] nanoparticles. The explanation is that, during a discharge, electrons attach to nanoparticles, and the resulting charged nanoparticles are much slower and much less effective than electrons in ionizing neutral molecules. As a result, the discharge is quenched.

Alternatively, [8] presents an interesting alternative based on insulating silica nanoparticles. Their good performance is explained in terms of their hydrophilicity, which “sequester”

water molecules from the oil.

Indeed, most of the research work presented in the literature is focused on thermal conductivity and breakdown voltage under quasi-uniform fields [1]-[9]. Rarely, breakdown and pre-breakdown phenomena (i.e., Partial Discharge, PD, inception, charge and repetition rate) have been dealt with [7]. In this work, we focus therefore on pre-breakdown phenomena under ac and dc divergent fields (although breakdown voltage, BDV, under ac quasi-uniform field is reported as well). To achieve a thorough picture, different concentrations of three different types of nanofluids (obtained using conductive, semiconductive and insulating particles) are investigated. PD Inception Voltage (PDIV), maximum charge (Q), and repetition rate are used to provide a quantitative comparison of the obtained results.

2. PREPARATION OF NANOFLUIDS

Shell Diala D was used as the base fluid. The oil was filtered using a paper filter having pore size of <5 μm .

The conductive nanofluids were prepared by adding magnetite nanoparticles to the oil. This nanofluid is indicated here as ferrofluid (FF). The FF was prepared by mixing oil with a suspension of magnetite nanoparticles (10-50 nm) manufactured by Magnacol (UK). While the manufacturer does not disclose details, it is likely that magnetite nanoparticles are treated on the surface with oleic acid to

prevent agglomeration [10].

A semiconductive nanofluid was prepared using Graphene Oxide (GO) platelets. GO powder was prepared by the Hummer's method from graphite nanoplatelets (purchased from Punto Quantico, Italy) [11]. The GO powder preparation followed the same procedure described by Li et al [12].

Eventually, an insulating nanofluid was obtained mixing a silica nanopowder (purchased from Sigma-Aldrich) with oil. The nanopowder consists of spherical nanoparticles (5-20 nm). It was used as received from the manufacturer. Some of the properties (including the charging relaxation time, [6]) of the nanoparticles used to prepare the nanofluids are reported in Table 1.

Table 1. Properties of individual nanoparticles before preparation of nanofluids.

Nanoparticles	σ (S/m)	ϵ_r	τ (s)	Size (nm)
Magnetite	10^4	80	7.5×10^{-14}	Average: 10 Maximum: 50
GO	10^{-7}	104	9.6×10^{-3}	12 20-50
SiO ₂	1.4×10^{-9}	3.9	51.2×10^{-3}	Average: 5 Maximum 20

Various test samples including pure mineral oil, nanofluids of 0.1 g/l, 0.2 g/l, 0.4 g/l (for SO only) and 0.5 g/l concentrations were prepared. In order to mix the nanoparticles with the base oil, we used a Hielscher UP200St ultra-sonicator. The sonication cycle lasted 15 minutes, with 20 W power and 50 % duty cycle. The samples were dried and degassed by placing them in a vacuum chamber for 48 hours at an absolute pressure lower than 50 Pascal. The final moisture content, below 5 ppm, was determined by a Karl Fischer titration method [13]. The so-obtained samples display great stability (no particle deposition/agglomeration was observed over several days).

3 EXPERIMENTAL SETUP

3.1 BREAKDOWN VOLTAGE MEASUREMENT

The BDV of the samples is measured using an automated test kit with maximum test voltage of 75 kV (Baur DPA 75C). Four sets of measurements, each consisting of 10 tests were carried out to achieve a large statistical significance. The procedure for each "shot" is as specified in std. IEC 60156 [14].

The electrode cell was modified to operate with a small quantity of NF: 50 ml. The cell contains two stainless steel semi-spherical electrodes having a body diameter of 7.8 mm, and a tip radius of 4 mm, spaced 2.5 mm. For the same fluid, the BDV were, on average, lower than those observed using a standard cell [15].

3.2 NANOFLUIDS TEST CHAMBER

PDIV and PD features were investigated using a point/plane electrode configuration. The point was a tungsten steel needle (1 μ m radius tip, 0.5 mm diameter) manufactured by Fine Science Tools GmbH. The gap spacing was 20 mm. The low voltage electrode was split to realize a guard ring. For PD

detection, the measurement impedance (50 Ω) was connected between the low voltage electrode and the guard ring. To minimize the amount of nanofluid (NF) used in each experiment, the cell capacity was reduced to 10 ml. The cross-sectional view of the cell is shown in Figure 1.

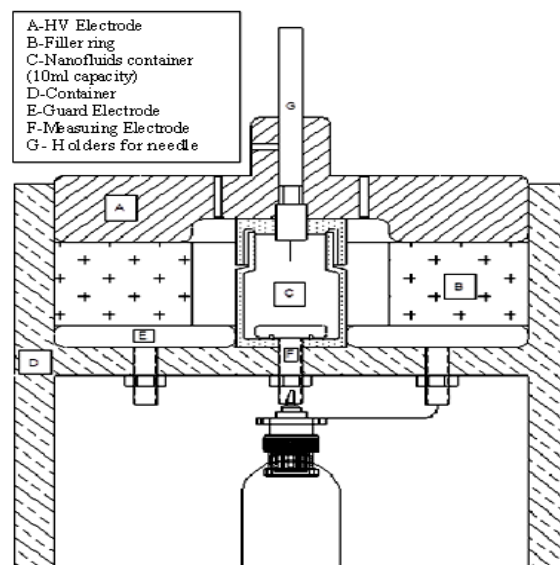


Figure 1. Nanofluids test cell for PD measurements.

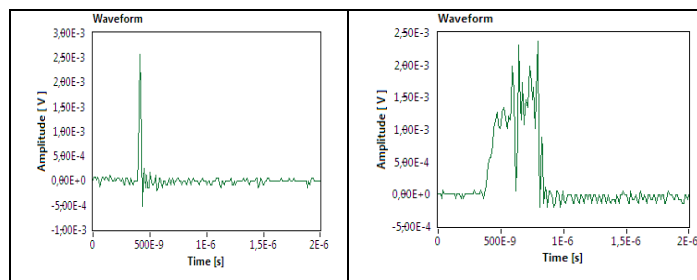


Figure 2. PD waveforms for base mineral oil at different overvoltage levels under DC positive voltage at (left) PDIV=14 kV, (right) V= 26 kV.

3.3 PD MEASUREMENT AND SETUP

The AC test setup consists of a 220 V/30 kV transformer and a capacitive divider (used to measure the applied voltage). PD signals were recorded by a Techimp PDCheck detector. For testing under dc voltages, a Fug - HCN 35-35000 bipolar 35 kV dc source was used. The entire test setup was placed in a shielded cabinet to reduce the external noise. The sensitivity of the system was better than 1 pC. Note that, since the test cell capacitance is very low, one should expect calibration errors [16]. To overcome this problem, the apparent charge was estimated using the following procedure. First, the spectrum of the PD pulse was calculated via the Fast Fourier Transform. The DC component of the pulse (removed by the high pass filter built in the detector input) was estimated as the median of the 20 lowest harmonics of the spectrum (since in the lowest frequency range the pulse harmonics are comparable with the DC component). Eventually, the charge was evaluated as the product of the estimated DC component of the pulse times the time length of the acquisition window.

PDIV was measured starting with an initial voltage of 2kV. The voltage was raised in steps of 1 kV, each lasting 5 minutes, till the occurrence of the first PD pulses. Measurements were repeated to get 5 PDIV values. The above procedure was performed for both AC and DC.

By further increasing the voltage, PD magnitudes increase, but the pulse waveform does not change. However, above a given threshold, the pulse tends to spread in the time domain transferring more charge, as shown in Figure 2 [17], [18]. This suggests the development of more intense streamers. In order to study this behavior, the applied voltage was raised above PDIV until a substantial change in the PD pulse shape could be observed. Since for short gaps (as the one used in these experiments), when streamers are observed, breakdown follows after increasing the applied voltage only slightly, tests were stopped when pulses as the one shown in Figure 2 (on the right) were first observed.

3.4 STATISTICAL DATA ANALYSIS

The data for BDV and PDIV were modeled assuming that their distributions follow a 2-parameter Weibull model, i.e.

$$F(x) = 1 - \exp\left(-\left(x/\alpha\right)^\beta\right) \quad (1)$$

where α and β are the scale and shape parameter of the distribution respectively. The parameters of the distribution fitting the experimental data were obtained through the Median Rank Regression estimate.

To provide a quantitative comparison of NF performance, the 10th percentile (B10) of the distribution are used in the following. The choice of using B10 stems from the fact that the better the oil, the higher should be the BDV (or PDIV) at low unreliability values, those that should be used as a reference for design purposes [19], [20]. Indeed, given the reduced sample size, the confidence intervals for percentiles below the 10th was excessively large to provide meaningful information. Therefore, the 10th percentile was used as a compromise between conflicting requirements.

The 90% confidence intervals of B10 were evaluated using a Monte Carlo procedure.

4. BREAKDOWN VOLTAGE

Following the standard approach, the first tests carried out on mineral oil and NFs were those aimed at inferring breakdown voltage (BDV) in quasi-uniform fields, according to the standard IEC 60156 [14].

Starting with Ferrofluids (FF), the results of these tests are reported in the Weibull chart of Figure 3, which shows that BDV increases up to 0.2 g/l. In terms of B10, the improvement with respect to mineral oil is about 40% (see Table 2). Larger FF concentrations reduce the NF performance. Figure 3 also emphasizes that, with the optimum concentration (0.2 g/l), data dispersion is minimum.

Similarly to FF, the BDV of nanofluids based on graphene oxide (GO) increases till 0.2 g/l (see Table 2 and Figure 4). Again, larger concentrations lead to worse results. Also in this

case, in correspondence of 0.2 g/l, the dispersion of breakdown voltages is minimum (larger β values).

Notably, the behavior (see Figure 5) of silica-based NFs is similar to that of the other NFs: an increase in value up to 0.2 g/l. Then, increasing further the concentration (up to 0.4 g/l) leads to lower BDV values.

Table 2. Comparison of BDV B10 for mineral oil and NFs.

Fluid	g/l	B10 _{min}	B10	B10 _{max}
Mineral oil	---	36.55	39.15	41.38
FF	0.2 g/l	53.35	54.72	55.89
GO	0.2 g/l	49.22	50.84	52.34
SiO ₂	0.2 g/l	48.35	49.65	50.62

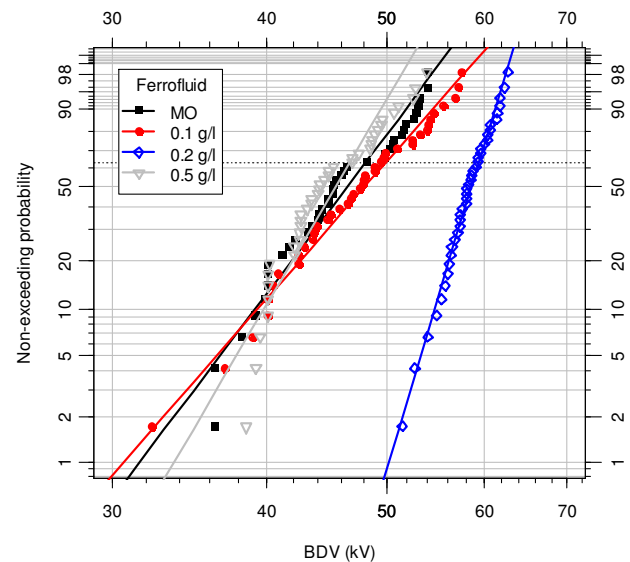


Figure 3. Weibull analysis of BDV values of FF.

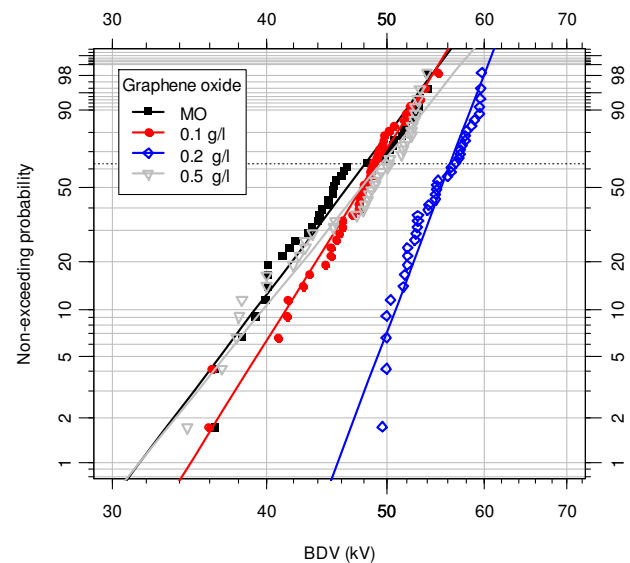


Figure 4. Weibull analysis of BDV values of GO.

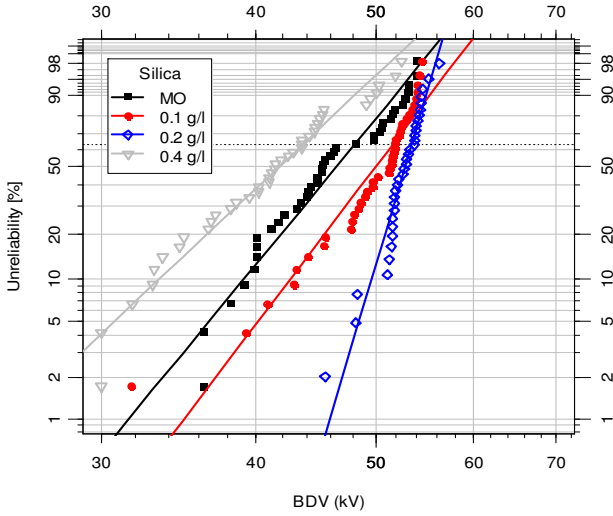


Figure 5. Weibull analysis of BDV values of SiO₂.

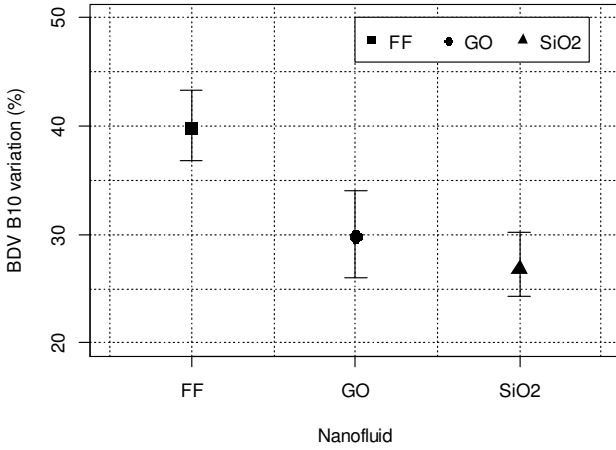


Figure 6. Variation of ac BDV for the three NFs considered in this investigation (concentration: 0.2 g/l in all cases).

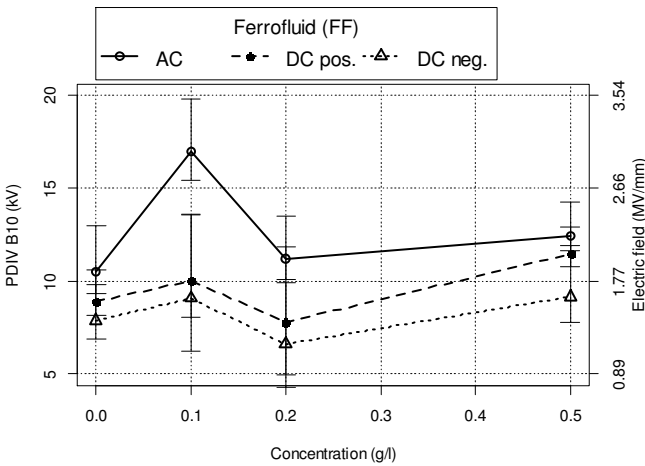


Figure 7. Behavior of 10th percentile (B10) of PDIV as a function of FF concentration. For ac voltages, the peak value of PDIV is reported.

Figure 6 eventually shows the relative values of BDV B10, i.e., the ratio of BDV B10 for the three NFs to the BDV B10. Inspecting the figure, and bearing in mind the data reported in

Table 2, it comes out that, the larger the conductivity, the larger the improvement in BDV B10. However, the dependence is not straightforward, as will be discussed later on.

5. PD INCEPTION VOLTAGE

The results obtained from PDIV tests are summarized below using figures which show PDIV B10 as a function of NF concentration. On the right y-axis, the figures report the peak electric field. The field is estimated through equation (1):

$$E_{tip} = V \cdot \frac{2}{\ln(4 \cdot d / r + 1)} \cdot r \quad (1)$$

where r is the tip radius ($r=1 \mu\text{m}$) and d the gap ($d=20 \text{ mm}$) [21].

For FF PDIV data are reported in Figure 7. Looking at the trends and comparing them with the confidence intervals, it is possible to conclude that ferrofluids do not modify significantly PDIV, except for ac voltages and using a concentration of 0.1 g/l. For dc voltages, FF can have a negative impact on PDIV.

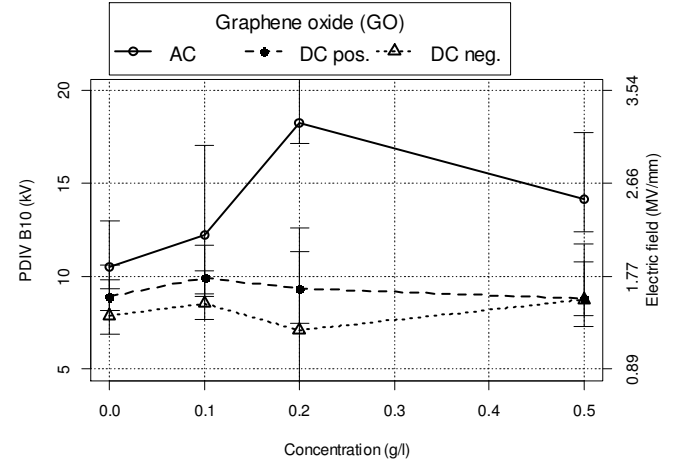


Figure 8. Behavior of 10th percentile (B10) of PDIV as a function of GO concentration. For ac voltages, the peak value of PDIV is reported.

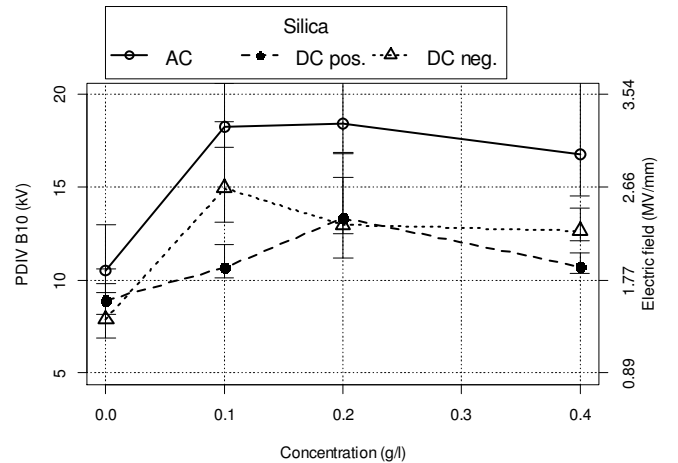


Figure 9. Behavior 10th of percentile (B10) of PDIV for SiO₂ as a function of SiO₂ concentration. For ac voltages, the peak value of PDIV is reported.

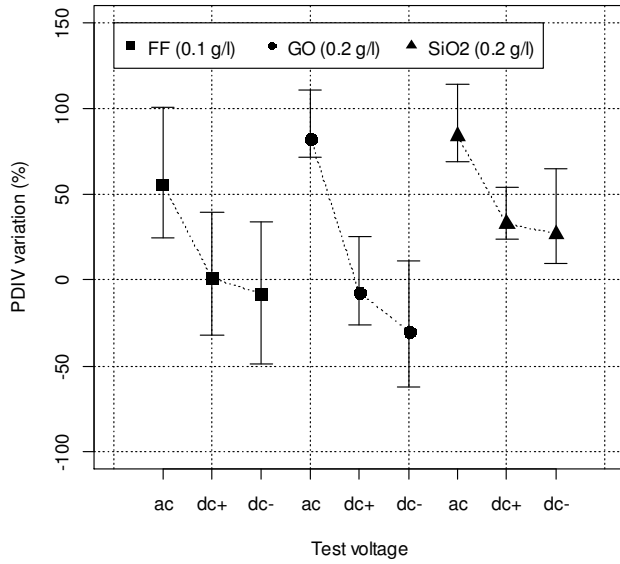


Figure 10. Variation of PDIV for the three NFs considered in this investigation.

For graphene oxide, results are summarized in Figure 8. The results hint that GO-based nanofluids with 0.2 g/l concentration could have a slightly better performance than mineral oil for ac discharges. For other types of supply voltages and concentration, the effect of GO cannot be appreciated.

Differently from the former NFs, Figure 9 shows that silica can improve PDIV consistently, independently of test voltage type. The samples with concentration of 0.2 g/l show enhanced performance for all test voltage types.

As a summary of the data reported in this section, Figure 10 shows the relative PDIV B10 for the concentrations showing the largest PDIV improvements under ac voltages. The figure emphasizes that NFs can improve ac PDIV to a large extent. For dc voltages, the only NF improving PDIV is that based on silica. In general, the NF based on the most conductive particles (FF and GO) have a detrimental effect of dc PDIV.

6. DEPENDENCE OF PD ON VOLTAGE

Experiments were performed with three NFs and three different concentrations, producing a large amount of data. We shall try to summarize here these results focusing on a single concentration for each NF. We have already observed considering PDIV that 0.1 g/l is the optimum concentration for FF, while 0.2 g/l seems the best one for GO and silica. Therefore, in the following, we shall discuss only the results obtained with these concentrations. The data not reported here for the sake of brevity confirm that these concentrations are indeed the optimum ones, something worth further theoretical investigation.

To investigate the behavior at voltages higher than PDIV, the voltage was raised above PDIV till the transition from

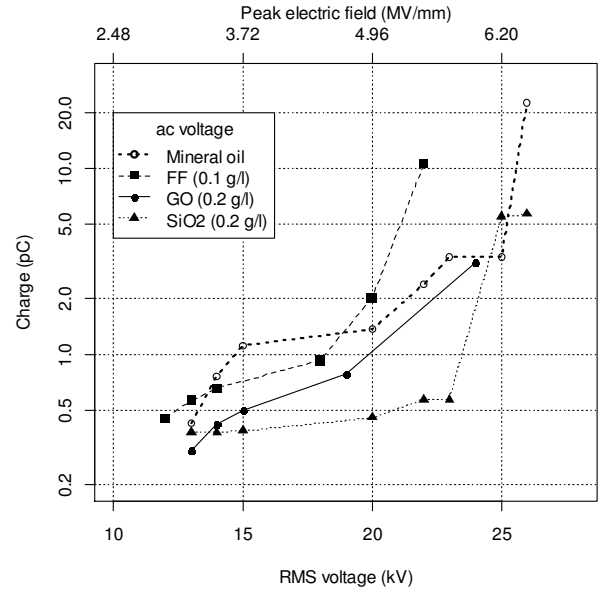


Figure 11. Trend of PD magnitude under ac voltages.

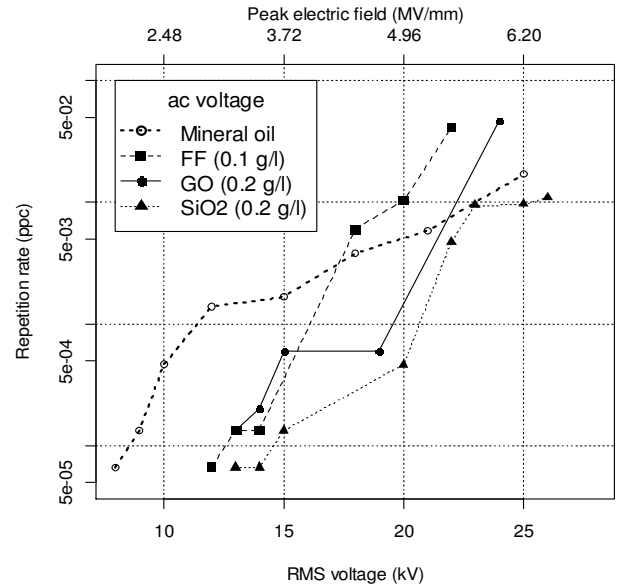


Figure 12. Trend of PD repetition rate under ac voltages.

“fast” to “slow” streamers, i.e., from pulses having a time length of roughly 100 ns to pulses having a time length of some hundreds of ns (i.e., 600 ns as in Figure 2). The maximum discharge magnitude and the repetition rate at the different voltage levels was recorded. Given the low repetition rate of PD in oil, the results are somehow erratic.

6.1 AC VOLTAGES

The results of the tests under ac voltages are summarized in Figure 11 and Figure 12. Against expectations, the most conductive NF, i.e. FF (which has the shortest charge relaxation constant, 7.5×10^{-14} s) shows the poorest performance: starting from 15 kV, PD magnitudes are larger than those observed in mineral oil. In addition, the transition to more energetic streamers, i.e., from “fast” to “slow” discharges, occurs at 22 kV, while all other fluids have thresholds above 25 kV.

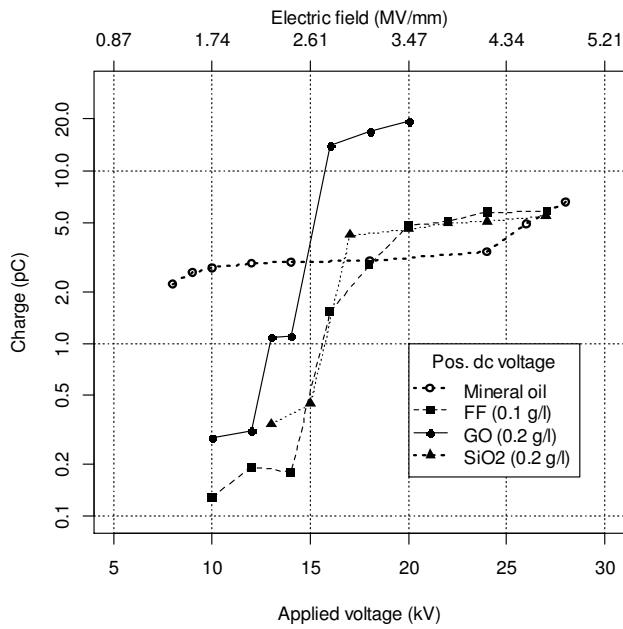


Figure 13. Trend of PD magnitude under positive dc voltages.

The best NF is that based on silica, which provides discharges below those of both mineral oil and GO. Indeed, the streamer transition occurs at the same voltage level for MO and the silica-based NF. Note that PD magnitudes tend to show a linear behavior in semilog y axis (except for the silica-based NF), that is, they increase exponentially with applied voltage.

The behavior observed for PD magnitudes is confirmed for repetition rate: FF is the NF with the highest repetition rates, while the other two fluids tend to behave in a similar way, although the silica-based NF is better than GO-based NF.

It is noteworthy that the relationships between PDIVs (see Figure 10) are reflected in the behavior of PD charge and repetition rate: FF performs worse than GO and SiO₂, these two behaving in a similar way (indeed, SiO₂ seems to perform slightly better than GO).

6.2 POSITIVE DC VOLTAGES

For positive dc voltages, the three NFs tend to provide results much better than mineral oil up to a threshold, which depends on the NF type (see Figure 13). The NF which provide the worst results in terms of PD magnitude is GO, which displays a transition from sub-pC to a few tens of pC discharges around 12 kV. Also Silica-based and FF-based NFs display similar results, although the transition occurs at somehow larger voltage levels (15 kV) and toward discharges of lower magnitude (about 5 pC).

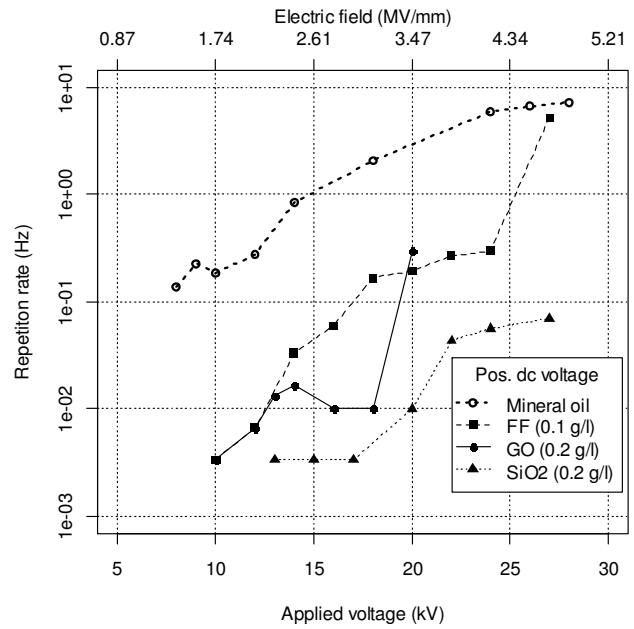


Figure 14. Trend of PD repetition rate under positive dc voltages.

Considering repetition rates (see Figure 14), the situation is different. In fact, repetition rates in the three NFs are consistently lower than those observed in mineral oil. Nevertheless, repetition rate trends confirm what already observed for PD charges: GO is the NF with the largest repetition rates, while the other two NFs tend to perform in similar ways. However, instead of FF, in this case silica is the NF exhibiting the best performance.

6.3 NEGATIVE DC VOLTAGES

Under positive dc voltages, NFs perform better than mineral oil when subjected to negative dc voltages: both PD magnitude and repetition rate are improved by adding nanoparticles to mineral oil, see Figure 15 and Figure 16. For this type of applied voltage, the silica-based NF is the one showing the lowest PD magnitudes and repetition rates. The GO based NF shows the lowest performance in negative dc voltages both in magnitude of charge and repetition rate compared to other NFs.

7 DISCUSSION

The current understanding of discharge phenomena in liquids is still incomplete. In fact, pre-breakdown and breakdown mechanisms in liquids are difficult to study because electrical and thermal phenomena interact with fluid dynamics. Also, we have no experimental data to quantify the influence of dielectrophoretic and electro-hydrodynamic forces on the distribution of nanoparticles under strongly divergent fields.

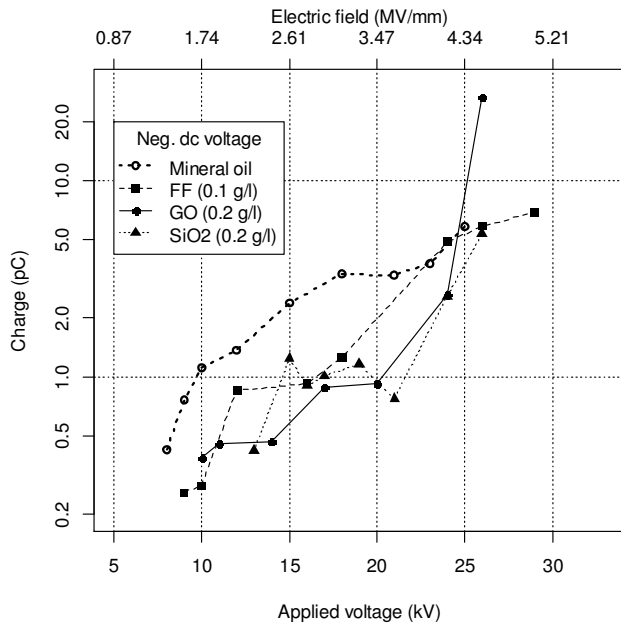


Figure 15. Trend of PD magnitude under negative dc voltages.

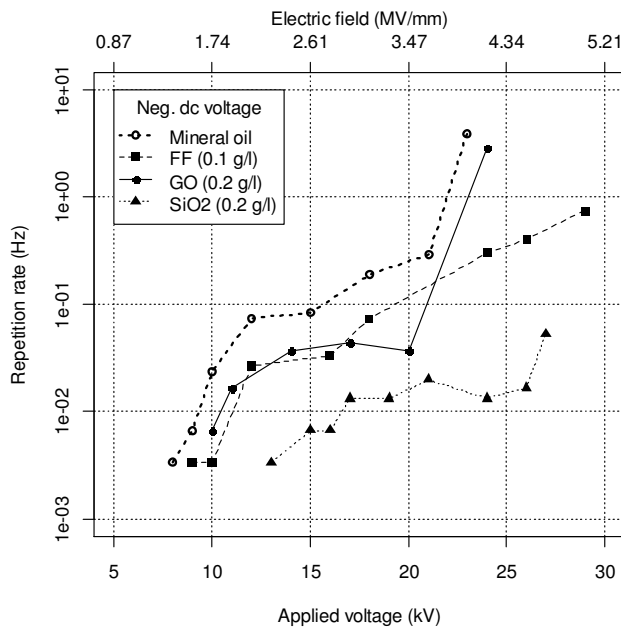


Figure 16. Trend of PD repetition rate under negative dc voltages.

The following points are worth being recalled:

1. Under quasi-uniform fields, the postulated breakdown mechanism for industrial-grade oils is that suspended polar particles are attracted towards microscopic protrusion at the electrodes through dielectrophoretic forces. If a partial bridge is formed between the electrodes, this can become the site at which breakdown takes place. For highly purified oils, an electron avalanche is usually assumed [22].
2. Under divergent negative dc fields, the hydrostatic pressure of the oil [22][23], and its humidity [24]–[26] affect remarkably BDV. Therefore, localized bubbles close to the needle tip are likely at the basis of

discharge inception. Given the large electrical fields, Fowler-Nordheim injection of hot electrons from the needle tip to the oil explains the formation of the initial bubble [23], [27][28][29].

3. For divergent positive dc fields, the role of pressure and humidity are less marked. Thus, bubbles are not the main reason for BD. Reference [22] postulates that a weak flux of electrons is injected at the low voltage electrode. Travelling towards the anode, electrons enhance the field at the needle tip, leading eventually to oil ionization. When the discharge is incepted, due to the different mobility of positive ions and electrons, a positive space charge forms in front of the needle tip, helping streamer propagation. The opposite does not occur under negative dc voltages, given the large electron mobility [28].
4. According to Zahn et al [5], conductive nanoparticles act as electron scavengers: electrons are trapped on their surfaces, thus reducing their speed. As a result, they become less effective in ionizing new molecules. This quenches the discharges. In a similar way, Yu Duefan et al [7] explain the behavior of TiO_2 nanoparticles.
5. The positive effect of silica, a hydrophilic nanoparticle, was already reported in [8]. It was explained considering that silica nanoparticles can “sequester” moisture, creating a water shell on the nanoparticle surface.

7.1 QUASI-UNIFORM FIELDS

As shown in Figure 6, conductive nanoparticles enhance BDV under quasi-uniform field conditions. The most likely explanation for FF and silica have been reported above. GO and silica tend to provide similar results. Since their relaxation constants are comparable and too large to affect discharge dynamics, the mechanism postulated for silica can likely be advocated for GO.

The reduced performance of NFs having concentrations in excess of the optimum one can be explained bearing in mind that nanoparticles tend to enhance the electrical field in the oil. Above some critical concentration, this phenomenon can become predominant. Indeed, with larger concentrations it is also possible that agglomeration probability increases.

7.2 DIVERGENT FIELDS

It is probably better to recollect the results collected in the previous sections in a schematic way. In practice, we have observed that:

- In terms of PDIV, for both positive and negative dc voltages, conductive nanofluids (FF and GO) perform generally worse than mineral oil. On the contrary, silica performs better than the other two NFs.
- PDIV values are often larger for positive dc voltages, except for silica, where the situation is somehow reversed.

- All NFs improve PDIV under ac voltages. Yet, the most conductive NF (FF) is the one providing the smallest increment with respect to mineral oil.
- With positive dc voltages, conductive nanoparticles behave well up to some voltage threshold. Above this threshold, PD magnitudes tend to increase markedly. A similar threshold was not found dealing with negative dc voltages.
- When dealing with dc voltages, GO is always the worst NF.

Despite the low water content of the oil, the first observation supports the conclusion that transferring water molecules from the oil to the nanoparticles might be more efficient in suppressing partial discharges than the electron scavenging mechanism postulated for conductive particles. Indeed, the evidence that the improvement is comparable for positive and negative dc voltages is at odd with what was found in [26], that is, the influence of moisture on PDIV is significant for negative dc voltages, much less important for positive dc voltages. Therefore, [26] findings would support a more important influence of silica NF on negative PD inception.

Also, the not satisfactory performance of NFs based on conductive particles under dc voltages has to be explained. Let us focus on FF and disregard GO, for the alignment of GO platelets under dc fields might explain its bad performance.

Since the NF performance decreases above the optimum 0.2 g/l concentration, it is possible that dielectrophoretic (DEP) forces drive the FF nanoparticles towards the high field region leading to a suboptimal concentration of magnetite particles at the proximity of the needle tip [30]. For positive dc voltages, the nanoparticles might trap cathode-emitted electrons reinforcing the field at the needle tip (a similar phenomenon was observed adding to mineral oil SF₆, a strongly electronegative molecule [25]). For negative dc voltages, a conductive particle in proximity of the needle tip can increase the field in the region between the tip and the nanoparticle itself, lowering the barrier for electron emission. An increased current density at the needle tip would favor the formation of a gaseous region and, eventually, PD inception. Indeed, this explanation is somehow at odds with the fact that PDIV with larger concentrations is sometimes improving.

To explain what happens under ac voltages, the water shell argument can be invoked for silica. NFs based on conductive nanoparticles pose a challenge, as their low performance under dc voltages is at odds with what happens under ac voltages. To explain this, one needs to postulate that the two phenomena advocated above (i.e., the formation of a negatively charged nanoparticle cloud and bubble formation) do occur after a formative time lag. If not, the peak values of PDIV under ac voltages would be similar to those observed under dc voltages.

For the improved behavior under ac voltages, a possible explanation is that, during the negative voltage half cycle, electrons are injected into the oil and are trapped on the

nanoparticles. Thanks to electro-hydrodynamic motion, the charged nanoparticles would then move away from the needle tip area, where neutral nanoparticles can arrive. During the positive half wave of the voltage, neutral nanoparticles may get attracted towards the needle tip. In close proximity of the tip, tunneling of electrons from the nanoparticle to the tip might become possible, and a cloud of positively-charged might surround the tip. In this way, nanoparticles might shield the needle tip and improve PIV.

8 CONCLUSION

The results reported in the paper highlight that nanofluids can provide significant improvements in the dielectric withstand capability of mineral oil, even under divergent fields. However, they might not be a universal panacea, particularly in the case of pre-breakdown phenomena under divergent fields for equipment working with dc or mixed ac+dc voltage waveforms (e.g., hvdc converter transformers).

Since different research groups have obtained results hinting that nanofluids could improve the dielectric behavior of mineral oil, it would be interesting to start a more systematic investigation with the aim of standardizing the tests and verify results by, e.g. round robin testing involving a number of labs worldwide.

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