RESEARCH ARTICLE

Dielectric and Thermal Response of TiO$_2$ and SiC Natural Ester Based Nanofluids for Use in Power Transformers

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ABSTRACT The improvement of dielectric and thermal properties of insulating oils used in High Voltage (HV) equipment has been a key part of research over the last three decades. In this study, a natural ester oil is used as matrix oil and dispersions of two semi-conducting nanoparticles (NPs), titanium oxide (TiO$_2$) and silicon carbide (SiC), are prepared in 0.004 weight percentage ratio (0.004% w/w). In terms of thermal features, thermal diffusivity and thermal conductivity are measured over a large temperature range between 25 and 90 $^{\circ}$C. By means of the dielectric properties, the Lightning Impulse Breakdown Voltage (LI BDV) of nanofluids (NFs) is measured and compared to the corresponding ones of the matrix. Moreover, the dielectric constant and dielectric dissipation factor (DDF) are determined for the NF and matrix samples in the frequency range of 1 to 10^6 Hz. The obtained results show that the NF containing SiC NPs is characterized from the greatest increase in thermal conductivity, by 58% with respect to natural ester oil's, as well as the lowest dielectric constant in both 25 and 90 $^{\circ}$C, most likely due to their higher permittivity and thermal conductivity. Lastly, the LI BDV of the same NF is the most enhanced, by 10% relatively to the matrix, while a numerical model in COMSOL Multiphysics is developed and is found to successfully verify the LI BDV results.

INDEX TERMS Insulating nanofluid, lightning impulse, dielectric constant, thermal conductivity, numerical model.

I. INTRODUCTION

Dielectric liquids are one of the three types of insulation used in High Voltage (HV) equipment, due to their high dielectric strength, the greater ease of their replacement in relation to solid insulators and mainly thanks to their ability to dissipate heat preventing the temperature rise inside it [1]. The use of mineral oil as dielectric liquid in HV equipment, mainly power transformers, displays certain disadvantages,

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such as toxicity, high flammability, while the combination of paper in the conductors leads to anisomeric stress due to the significant deviation in their dielectric constants [2]–[6].

Therefore, current research interests focus on finding a proper replacement, with natural and synthetic esters being possible candidates, as long they are readily biodegradable, they are characterized from increased flash and fire points and their higher permittivity is critical, as it can match the one of paper insulation in minimizing electric stress during high-load conditions [5]–[9]. However, natural ester oils (NEO) suffer from lower dielectric strength under AC or Lightning

Impulse (LI) voltage [9], [10]. Furthermore, as the temperature rises, their dielectric dissipation factor (DDF) rises as well, implying increasing dielectric losses [12], [13]. As a result, researchers are concentrating their efforts on developing nanofluids (NFs) based on conventional oil and a suitable amount of nanoparticles (NPs), with the goal of eliminating ester oil defects while also increasing their dielectric strength and thermal performance [7]–[19].

The integrated NPs are classified on account of their types i.e., metal oxides [7]–[11], [14]–[17], carbides [13], [19], [20] and nitrides [20]–[22], their shapes, sizes and electrical property, i.e. conductive [9]–[11], [15], [23], semi-conductive [8], [13], [14], [17], [24]–[26] and insulative [11], [15], [21], [22], [25]. Khaled and Beroual [11] reported that adding conductive iron oxide (Fe_3O_4) NPs with a diameter of 50 nanometers (nm) at 0.2 g/L concentration to synthetic ester oil (SEO) can result in a 7.51% of the negative Lightning Impulse Breakdown Voltage (LI BDV). The same authors [15] observed that integrating the same kinds of NPs in NEO can result in a 48 percent increase of the AC Breakdown Voltage (AC BDV). Koutras *et al.* [13] studied the influence of increasing concentration of silicon carbide (SiC) NPs of 50 nm average diameter in AC BDV of NEO FR3. They found that the AC dielectric strength is increased until a threshold level of 0.004 % w/w and decreased after this concentration level. In [17], Koutras *et al.* found that the addition of titanium oxide (TiO₂) NPs with 21 nm average diameter in the same NEO can lead to a 22.4% maximum improvement of the AC BDV at 0.020% volume percentage ratio. Primo *et al.* [27] studied the LI BD performance of mineral oil-based Fe_3O_4 NFs on various concentrations finding that positive LI BD can be augmented by 50%, but the negative one is similar to the matrix's. Mansour *et al.* [28] studied the influence of surface modified aluminum oxide (Al_2O_3) and TiO₂ NPs on AC BDV. They found that with the addition of a suitable amount of surfactant, the same percentage enhancement is recorded, in contrast with the effect of the unmodified NPs, where the integration of TiO₂ led to higher improvement. They also reported similar decrease in dielectric constant for both NFs containing surface-modified NPs, which was attributed to the influence of the augmented interfacial zone. However, the use of surfactants could reduce the heat transfer performance of NFs while simultaneously increasing viscosity [29], [30]. Moreover, Khelifa *et al.* [18] and Atiya *et al.* [31] studied Partial Discharge (PD) activity in SEO and mineral oil-based NFs respectively. The authors of [18] found that the addition of fullerene NPs significantly enhances the Partial Discharge Inception Voltage (PDIV) of SEO, unlike graphene NPs, while the authors of [31] reported that Al_2O_3 NPs could cause a higher improvement to the resistance of NF to PD activity with respect to TiO₂. They attributed this finding to the larger Electrical Double Layer (EDL) thickness around the Al_2O_3 NPs [31]. Beyond liquid insulation, NPs are also widely used to increase the dielectric properties of solid insulators [32]–[34] and in other types, like biosensors [35]–[37].

In this work, two uniform suspensions of SiC and TiO₂ NPs at 0.004% w/w are prepared in NEO FR3. In terms of thermal properties, thermal diffusivity and thermal conductivity are measured and calculated respectively. In terms of the dielectric ones, the relative permittivity and electrical conductivity of the NPs and matrix are measured by employing the Dielectric Relaxation Spectroscopy (DRS) technique. LI BDV under positive polarity, pursuant to IEC 60897 standard is also examined and compared to the matrix performance. Apart from that, the dielectric constant and DDF are determined with the DRS technique. The novelty of this study can be supported from the following points:

- In the literature, the BDV characteristic and mechanism of natural ester-based NFs incorporating metal oxide NPs, particularly TiO₂, have been extensively studied [26], [38]. The impact of carbide NPs, such as SiC, a semiconductor NP like TiO₂, in biodegradable oils, on the other hand, has not been thoroughly investigated [13].

- Most research studies are focused on the dielectric properties of NFs, while the available literature of their effect on thermal diffusivity is limited [21], [22], [39], [40]. Consequently, investigating the dielectric and thermal performance of semiconducting SiC NP doped NF, compared to the corresponding one of NF incorporating TiO₂ NPs in the same concentration leads to valuable findings.

- One of the most significant drawbacks of the NEO is its high dielectric losses. With the assistance of the DRS method, it has been shown that the DDF of both NFs, especially sNF, is reduced mainly at 25 °C.

- Last but not least, the conduction of the LI BDV experiment under positive polarity is accompanied with the development of a numerical model for the simulation of pre-breakdown phenomena regarding the same NFs during LI stress in COMSOL Multiphysics [23], [41] and the results are compared to the experimental ones. The reported results demonstrate that adding SiC NPs to the under-studied oil can result into an improvement of the dielectric properties, while the development of the model in COMSOL Multiphysics provides physical insight on the pre-breakdown phenomena and verifies the LI BDV results.

II. HARACTERIZATION OF THE MATERIALS AND SYNTHESIS OF NANOFLUIDS

Biodegradable NEO FR3^TM has been used as matrix, with a density of 0.92 g/ml, fire point greater than 300 °C and other characteristics that have been discussed elsewhere [10], [17], [42]. The commercially obtained TiO₂ (Sigma Aldrich) and SiC (NanoAmor) NPs are spherical, with 99.5% purity and average nominal diameters of 21 and 50 nm respectively. The thermal conductivity of the integrated TiO₂ and SiC NPs is in the range of 5-11.8 W/(m·K) and 165-220 W/(m·K) respectively. The Field Emission Scanning Electron Microscopy
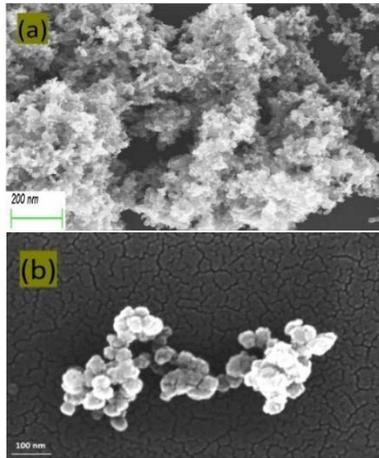
FIGURE 1. FE-SEM images of the NPs in high magnifications: (a) TiO$_2$ NPs, (b) SiC NPs.

(FE-SEM) method was used to determine the nominal size of the NPs. The NPs were drop-casted onto Si substrates and a Zeiss SUPRA 35VP microscope was utilized to monitor their size. Figure 1 depicts the TiO$_2$ and SiC powders as shown by FE-SEM, revealing that their average size is in the range of the nominal one.

A. PREPARATION PROCEDURE OF NANOFLUIDS

For the production of NF samples, a two-step procedure is employed. A 500 mL volume of FR3 has been chosen as the matrix for the production of colloidal suspensions. To accomplish moisture reduction within the allowed limits, each sample is filtered and dried overnight in a hot air oven at 120 ºC. Following the methods described previously [10], the amount of moisture inside each sample after drying was validated using the Karl Fischer titration method [43]. In summary, the residual moisture was measured and validated using a Mitsubishi Chemicals Co. CA-100 moisture meter and a Metrohm KF Coulometer 831, respectively and was found less than 200 ppm.

Following that, suitable proportions of TiO$_2$ and SiC powder are submerged in the matrix dielectric liquid to attain the selected 0.004% w/w concentration. However, the integrated colloids tend to aggregate rapidly after their addition because of the attractive intermolecular forces, creating agglomerates, due to their large surface areas per unit volume [10]–[13], [26]. Consequently, the NF samples are ultrasonicated for 1.5 h using Elma's ultrasonic cleaner Elmasonic S 40 H in order to guarantee initial homogeneous colloidal suspension dispersion. Table 1 displays the labeling of the NFs, as well as a base oil sample that is also investigated for comparison purposes.

B. ELECTRICAL CHARACTERIZATION OF MATERIALS

The DRS method is used to measure the real and imaginary parts of relative permittivity for the NPs and matrix sample. The NPs in form of powder are placed in a special cell with

TABLE 1. Labeling of under-study samples.

Sample Label (n)	NPs Percentage Ratio (% w/w)
Matrix (Base)	0
tNF	NF containing 0.004% TiO$_2$
sNF	NF containing 0.004% SiC
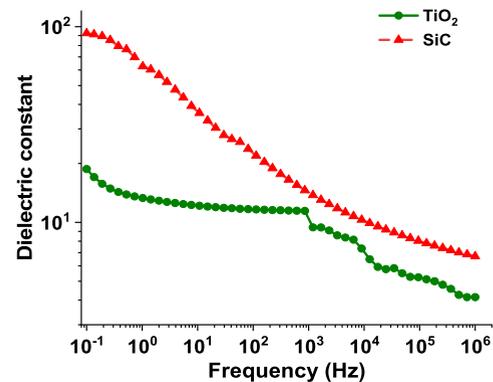
FIGURE 2. Change of dielectric constant versus frequency for: (a) the used NPs, (b) the matrix.

golden sphere electrodes of 20 mm diameter, which is then inserted to the Novocontrol BDS1200 cell for the conduction of the measurements. The liquid samples, on the other hand, are put in a sample holder-capacitor that is cylindrical (BDS1307 cylindrical liquid cell by Novocontrol). Using a Novocontrol Alpha analyzer, an AC voltage is applied to the capacitor sample, and the relative permittivity ε_r, in the form of (1), is recorded as a function of frequency in the range from 1 to 10^6 Hz [44]:

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r'' \tag{1}$$

where, ε_r' represents the real part of relative permittivity expressing the dielectric constant and ε_r'' denotes the imaginary part, which defines the dielectric losses caused by leakage current and dielectric polarization. In addition, the DDF (tanδ), is calculated from (2):

$$tan\delta = \frac{\varepsilon_r''}{\varepsilon_r'} \tag{2}$$

The electrical conductivity σ in S·m^{-1}, on the other hand, is calculated from the measured dielectric losses, according to (3):

$$\sigma = \omega \cdot \varepsilon_0 \cdot \varepsilon_r'' \tag{3}$$

where, ω is the angular frequency in rad/s and ε_0 is the permittivity of vacuum in F·m^{-1}.

Figures 2 and 3 illustrate the dielectric constant and electrical conductivity of the NPs at room temperature, respectively.
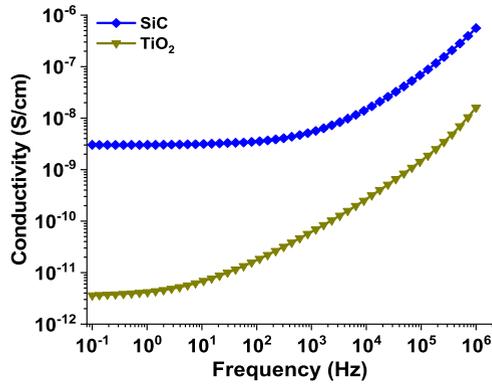
FIGURE 3. Change of electrical conductivity versus frequency for: (a) the used NPs, (b) the matrix.
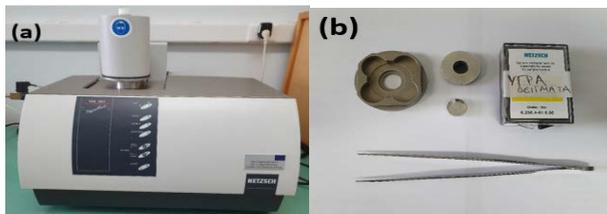
FIGURE 4. (a) NETZSCH LFA 467 Hyper Flash apparatus, (b) LFA test cell along with the liquid samples.

III. EXPERIMENTAL SETUP

Firstly, both the thermal and dielectric measurements were conducted inside the first 48 hours from the synthesis of uniform suspensions, in order to eliminate the negative effects of NPs' ageing which leads to aggregation/sedimentation, thus change in the active NPs' concentration inside the transformer oil [13], [17], [45].

The thermal diffusivity and thermal conductivity are determined using a NETZSCH LFA 467 Hyper Flash apparatus, as depicted in Figure 4(a). The samples are placed in a liquid-specific cell (LFA test cell with diameter of 12.7 mm) illustrated in Figure 4(b). Measurements are performed across a wide range (from 25 to 85 °C in 10-degree increments, with a final temperature of 90 °C). Thermal diffusivity α, in mm^2/s, is directly measured from an average of 5 light pulse shots and calculated via the half-time method (4) [45]:

$$\alpha = \frac{1.38 \cdot L^2}{\pi^2 \cdot t_{1/2}} \qquad (4)$$

where, L is the sample thickness and $t_{1/2}$ is the half-time necessary in order the maximum temperature to reach on the opposite side.

Thermal conductivity λ, on the other hand, is calculated from Fourier's law of heat conduction (5) [42], [45]:

$$\lambda = \alpha \cdot C_p \cdot \rho \qquad (5)$$

where, λ is the thermal conductivity in W/(m·K), C_p is the specific heat capacity in J/(g· K) and ρ is the density of the liquid in g/ml, which has been kept constant and equal to that of the matrix (i.e. 0.92 g/ml).

The dielectric constant and DDF of the NF samples have been determined in the same way as described in the previous section for the NPs and matrix oil with the DRS technique. The NF samples are placed in a cylindrical liquid sample holder-capacitor. An AC voltage is applied to the capacitor sample and the relative permittivity ε_r is recorded as a function of frequency, in the range from 1 Hz to 1 MHz employing a Novocontrol Alpha analyzer.

In terms of LI BDV measurement, this is a critical attribute because it indicates the dielectric insulation coordination of the NF during lightning activity. A two-stage (Marx) Haefely LTD impulse generator with a 400 kV maximum output voltage, 15 nF per stage capacitance and 600 J total energy, is used [46]. The measurements are conducted with a divergent field geometry, under positive polarity, according to the IEC 60897 standard [47]. The test cell consists of point-sphere electrodes and their gap is fixed at 25 mm. The point's radius of curvature is 50 μm and the diameter of the sphere is 12.7 mm.

For the conduction of the experiment, the up and down method is implemented [11], [27], [48]. Starting from a relatively low voltage with respect to the expected LI BDV, 3 voltage applications take place at each voltage level. In case none of the 3 ends in breakdown, the voltage increases by 5 kV. If at least 1 BD occurs, then the voltage is automatically reduced by 2 kV and a 5 min interval is applied before the next shot. The same process continues until 10 BD events are obtained, followed by statistical analysis with their adjustment to the Weibull distribution [11], [15], [17], [27], [48]. The equivalent circuit of the impulse generator is shown in Figure 5(a). The applied LIV (1.2/50 μs) is monitored via the CST capacitive divider (Figure 5(a)) through an oscilloscope Tektronix DPO4104; 1 GHz/5 GS s-1. A typical positive LI BDV waveform obtained from the oscilloscope is presented in Figure 5(b).

For the processing of the results, the Weibull distribution was chosen in order to evaluate the strength of the matrix oil and NF samples in critical failure rates of 1, 10, and 50% since it does not make assertions on skewness and kurtosis [11], [13], [15]. In order for this statistical processing to be feasible, the Anderson-Darling goodness of fit test is used to conclude whether the BD data for each sample can be adjusted to the Weibull distribution by calculating the *p*-value [13] and comparing it to the selected significant level α, which is determined to be 5% ($\alpha = 0.05$).

IV. EXPERIMENTAL RESULTS AND DISCUSSION
A. THERMAL PROPERTIES
This section includes the obtained results from the thermal diffusivity and conductivity measurements, along with the thermal stability study and discussion on them.

1) THERMAL DIFFUSIVITY AND CONDUCTIVITY
The mean values of thermal diffusivity for the NF and base oil samples acquired from five light pulses and calculated

(a)

(b)
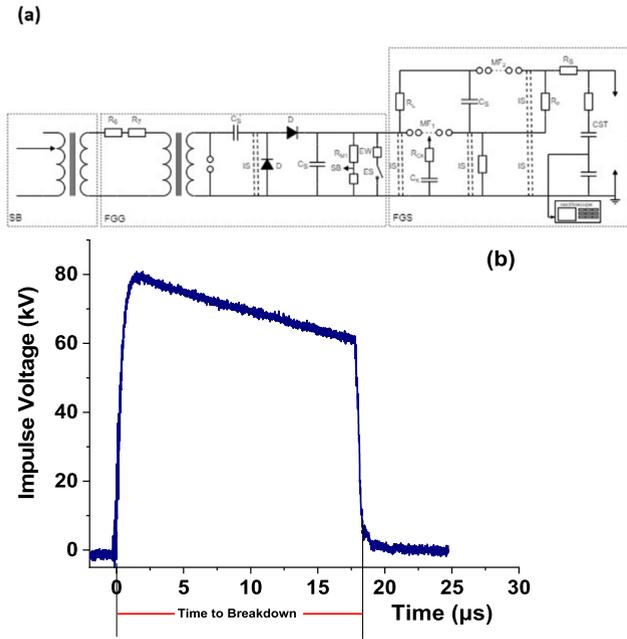
FIGURE 5. (a) Equivalent circuit of the impulse generator, (b) Typical LI waveform (1.2/50 μs) displaying the breakdown time.
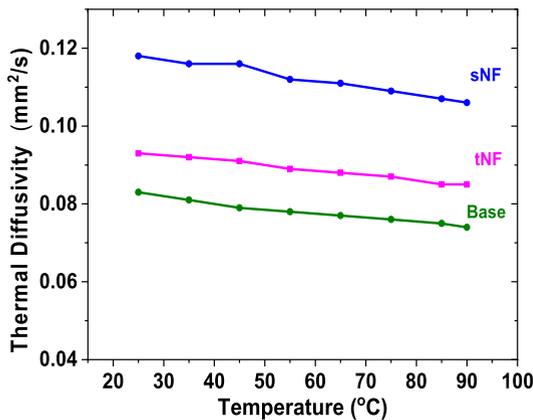
FIGURE 6. Change of the thermal diffusivity of the samples in question as a function of temperature (range 25-90 $^{\circ}$C).

from (4) at each temperature level are demonstrated in Figure 6. It is clear from this graph that both NF samples have improved thermal diffusivity with respect to NEO, with sNF showing the higher improvement throughout the whole temperature range. Beyond that, as the temperature rises, all samples show a decrease in diffusivity [45], [49], [50]. The Matrix oil sample has the greatest impact on the thermal diffusivity behavior of NFs, as evidenced by the experimental results, as long the same trend in thermal diffusivity with temperature increase is also observed for the NFs [50].

Figure 7 shows the thermal conductivity of the samples under investigation as a function of temperature. It appears that for both NF and matrix oil samples, it remains nearly constant (with very slight variances) over the whole temperature range. The highest thermal conductivity augmentation is
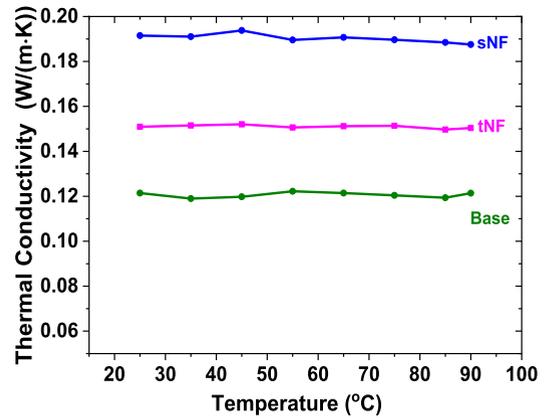
FIGURE 7. Change of the thermal conductivity of the samples in question as a function of temperature (range 25-90 $^{\circ}$C).

seen in the sNF sample, which is around 58% with respect to the NEO's, while the corresponding increase regarding tNF is about 25%. Therefore, it is obvious that at this weight percentage, the integration of this type of NPs has led to significant thermal conductivity enhancement of the dielectric liquid. This finding is consistent with several published research works regarding the influence of nanometric scale particles in thermal conductivity [20], [21], [39], [40], [50]. The high thermal conductivity of the SiC NPs (165-220 W/(m· K)) with respect to TiO$_2$ could be responsible for the higher improvement concerning the sNF sample [51], [52]. Thermal behavior of the two NF samples at these concentration levels is directly affected from the NPs, since they do not suffer from rapid aggregation, as it has been shown in our previous works [13], [17], [50], [53]. When NPs cluster, they lose their ability to increase heat transmission, alter the structural distribution of the suspension system, and ultimately break the irregular movement laws [52].

B. DIELECTRIC PROPERTIES
This part includes the obtained results from the permittivity and DDF measurements, as well as the analysis of positive LI BDV and PD activity results.

1) DIELECTRIC CONSTANT AND DISSIPATION FACTOR
Figures 8(a)-(b) show the spectral change in the real part of relative permittivity for the under-studied samples at 25 and 90 $^{\circ}$C, respectively. The corresponding change of DDF versus frequency at the same temperatures is depicted in Figures 10(a)-(b) respectively.

From Figures 8(a)-(b) it is apparent that both NF samples, especially sNF, are characterized from lower dielectric constant for the whole frequency range, including the power frequency of 50 (60) Hz, mainly attributed to interfacial polarization of Maxwell-Wagner-Sillars type and to electrode polarization [42]. The decreased values of dielectric constant could be a sign for improved dielectric strength of this sample [13], [28], which is also reflected in the following LI
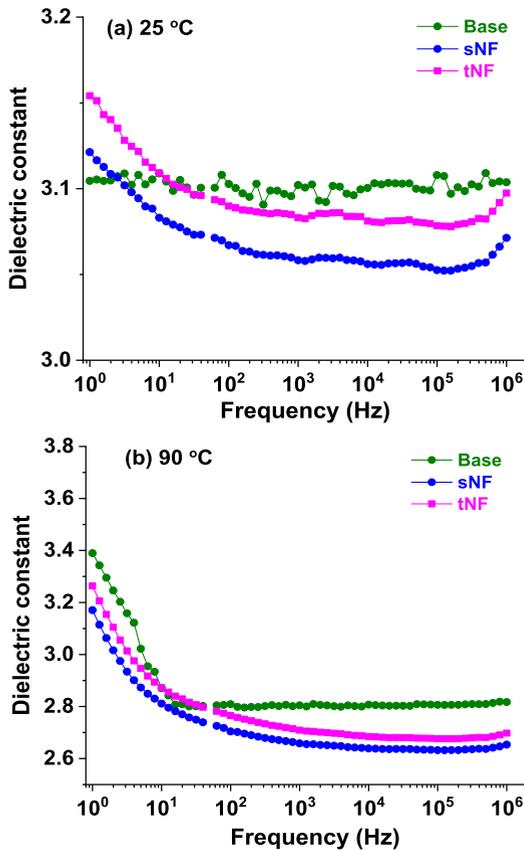
FIGURE 8. Spectral change of dielectric constant of the samples in question (range 1-10^6 Hz): (a) at 25 °C, (b) at 90 °C.

FIGURE 9. Spectral change of dissipation factor of the samples in question (range 1-10^6 Hz): (a) at 25 °C, (b) at 90 °C.

BDV results. It is also obvious that the dielectric constant falls with increasing temperature in all under-study samples, therefore they are more insulative with temperature rise. The temperature effect could be attributed to the fact that, dipolar orientation polarization processes taking place in this frequency range, are proportional to 1/T [50]. Assuming that this type of polarization is the main contributor to polarizability, the recorded dielectric constant reduction with temperature increase can be explained.

Figures 9(a)-(b) show that both NF samples exhibit lower DDF over the whole frequency range, with a greater reduction at room temperature. This is a key characteristic because the elevated DDF in relation to mineral oil is one of the NEO's most serious shortcomings [12], [13]. It should be emphasized, however, that all samples show an increase in DDF as the temperature rises. The DDF, directly correlated with the electrical conductivity of both NFs is mainly driven from the matrix dielectric liquid electrophoresis conductivity [50].

2) LIGHTNING IMPULSE BREAKDOWN VOLTAGE

Table 2 presents the mean values of LI BDV in kV and time to breakdown in μs, regarding the samples in question. In the same Table, the standard deviation of them, is also demonstrated. Scale and shape parameters are crucial for

TABLE 2. Descriptive statistics of the lightning impulse breakdown experiment.

Sample type	LI BDV (kV)		Breakdown Time (μs)		Breakdown velocity (km/s)		
	Mean (kV)	Std. Dev. (kV)	Mean (μs)	Std. Dev. (μs)	U$_{50\%}$	U$_{10\%}$	U$_{1\%}$
Matrix oil	75.3	2.6	17.5	1.3	1.47	1.64	1.91
tNF	79.5	2.0	18.4	1.0	1.35	1.48	1.65
sNF	82.7	2.4	19.1	1.1	1.30	1.43	1.63

the Weibull distribution and represent the failure at a rate of 63.2% and the dispersion of the events (Weibull slope), respectively [11], [13], [15]. The breakdown velocities at 50, 10 and 1% probability levels after fitting the breakdown times to the Weibull distribution are also presented in Table 2. Table 3 also contains the corresponding values of p-value, where it seems that all of them are higher than the significant level, meaning that the possibility that the BD events follow the Weibull distribution cannot be rejected, as well as the values of scale and shape parameters.

Consequently, from the Weibull plot of the LI BDV of Matrix oil, tNF and sNF samples (Figure 10), the BDV at failure probabilities of 1, 10 and 50% are calculated and included in Table 4. According to the processing of the LI BDV results, it is evident that the mean LI BDV is augmented with the addition of SiC NPs at 0.004% w/w with respect

TABLE 3. Weibull distribution parameters.

Sample Label (n)	p-value	Scale (kV)	Shape
Matrix oil	0.07	76.6	28.9
tNF	0.25	80.4	48.0
sNF	0.18	83.9	33.0

TABLE 4. Withstand voltages based on the weibull distribution.

Sample Label (n)	$U_{50\%}$ (kV)	$U_{10\%}$ (kV)	$U_{1\%}$ (kV)
Matrix oil	75.6	70.8	65.3
tNF	79.8	76.8	73.1
sNF	83.0	78.3	73.0
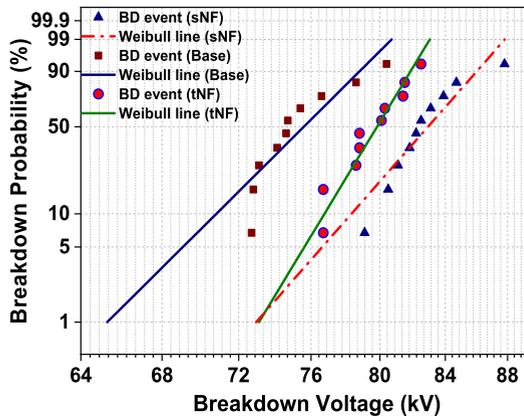
FIGURE 10. Weibull plot of the LI breakdown events for the matrix and the two nanofluid samples under investigation.

to NEO's by 9.8%, while the addition of TiO₂ NPs at the same weight percentage ratio leads to lower improvement, by 5.6%. Furthermore, on account of the withstand voltages calculated following adjustment of the BD events to the corresponding Weibull distribution, it has been found that $U_{10\%}$ and $U_{1\%}$ of sNF the highest improvement, by 10.6% and 11.8% with respect to the matrix oil. The importance of these values is critical for the insulation coordination and the design of a high voltage insulation system with operational reliability and reproducibility of the dielectric insulator's performance. Particularly the $U_{10\%}$ and $U_{1\%}$, corresponding to the lower BDV level, guarantee the insulation level of the system [54], [55].

BDV at 10% cumulative probability provides an assessment for the lowest possible BDV, therefore it is an indication for the transition of streamer to the 2nd mode [10], [11], [13], [15], [18], [24], [56], while $U_{1\%}$ refers to the transformer's voltage limit for safety and continuing functionality, which is a vital parameter for power system insulation coordination during lightning and surge incidents [57], [58].

The average breakdown velocity u in m·s⁻¹, across the gap of 2.5 cm can be calculated from (6) [27], [59], [60]:

$$\bar{u} = \frac{d}{t} \tag{6}$$

where, d is the gap length in m, i.e. 2.5 cm, and t is the mean time to BD in s, as it is demonstrated for each sample in Table 2. The average breakdown velocity for Matrix oil, tNF and sNF is calculated as 1.47, 1.36 and 1.31 km·s⁻¹ respectively corresponding to slow streamers propagation (2nd mode) [59], [60].

In order to interpret the obtained LI BDV results, the NP mechanism as shallow traps of the charge carriers, will be employed [23], [25]. Under the application of an external electric field E_0 in V·m⁻¹, if the dielectric constant of the integrated NPs and the matrix differs significantly, based on (7), induced or polarized charges are created at the NPs' interface. As long both kinds of NPs are semi-conductive, the existence of surface charges is due to dielectric polarization [25]. These charges produce a potential well, which decreases with the distance from the NP's radius. Therefore, the fast electrons at the streamer's tip are captured in shallow traps and the streamer propagation is slown down; as a result, a greater electric field value should be provided to allow the electrons to be freed and span the gap. The potential well distribution for both kinds of NPs is expressed from (7), while (8) corresponds to the charge captured by each of them as a function of time [23], [25], [53]:

$$\varphi_{(r)} = \frac{\varepsilon_2' - \varepsilon_1'}{2\varepsilon_1' + \varepsilon_2'} R^3 E_0 \frac{1}{r^2} \tag{7}$$

where, ε_1', ε_2' are the dielectric constants of matrix oil and NPs, respectively, R is the radius of the NP in nm and r is the distance from the NP's surface in nm.

$$Q_{(t)} = Q_s \frac{\frac{t}{\tau_{pc}}}{1 + \frac{t}{\tau_{pc}}} \tag{8}$$

where, Q_s is the saturation charge captured by each SiC or TiO₂ NP, expressed by (9), and τ_{pc} is the time constant of NP charging, given by (10) [23], [25]:

$$Q_s = -12\pi \varepsilon_1' E_0 R^2 \frac{\varepsilon_2'}{2\varepsilon_1' + \varepsilon_2'} \tag{9}$$

$$\tau_{pc} = \frac{4\varepsilon_1'}{|\rho_e|\mu_e} \tag{10}$$

where, ρ_e is the electron charge density in C·m⁻³ and μ_e is the electron mobility in m²·V⁻¹·s⁻¹.

Thus, based on Eq. (7) and (9), as long the measured value of dielectric constant of SiC is higher than the one of TiO₂ for the whole frequency range, pursuant to the results of DRS technique (Figure 2(a)), a higher potential well distribution, i.e. a higher local field, is created, especially close to their surface, where more electrons are captured and reduce their mobility, so the streamer propagation is hindered. In order to investigate the latter, a model of a single spherical NP is examined in COMSOL Multiphysics graphic environment. A DC external field has been applied to the y-axis and the electric field distribution has been recorded graphically (Figure 11) and in 3D (Figure 12(a)-(b)). It is apparent that higher electric field values are recorded at the surface of SiC NPs, as expected.
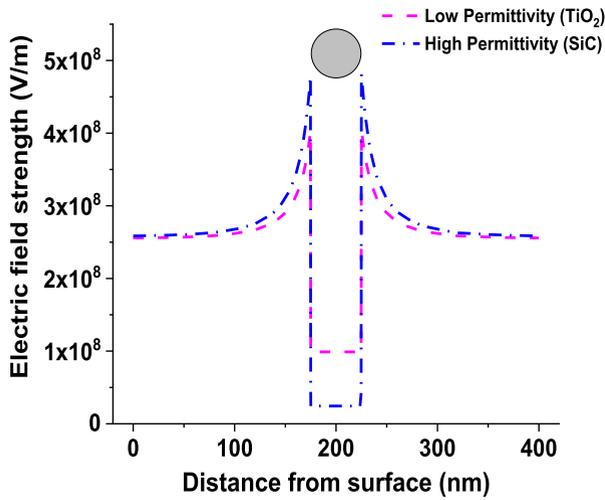
FIGURE 11. Simulated electric field distribution around the surface of SiC and TiO$_2$ NP.

TABLE 5. Main simulation parameters.

Symbol	Quantity	Value (S.I.)
μ_+	positive ion mobility	10^{-9} m^2·V^{-1}·s^{-1}
μ_-	negative ion mobility	10^{-9} m^2·V^{-1}·s^{-1}
μ_e	electron mobility	10^{-4} m^2·V^{-1}·s^{-1}
μ_{np}	nanoparticle mobility	10^{-9} m^2·V^{-1}·s^{-1}
R_{pn}	ion-ion recombination rate	1.13×10^{-17} m^3·s^{-1}
R_{pe}	ion-electron recombination rate	5.65×10^{-13} m^3·s^{-1}
α	molecular separation distance	3×10^{-10} m
h	Planck's constant	6.626×10^{-34} m^2·kg·s^{-1}
m^*	effective electron mass	9.11×10^{-32} kg
n_0	number density of ionizable species	10^{-21} m^{-3}
Δ/e	ionization potential	7.1 eV
τ_α	electron- oil molecule attachment time constant	2×10^{-7} s
τ_{np}	nanoparticle attachment time constant	2×10^{-9} s

FIGURE 12. 3D plot of electric field distribution around each NP: (a) TiO$_2$ NP, (b) SiC NP.
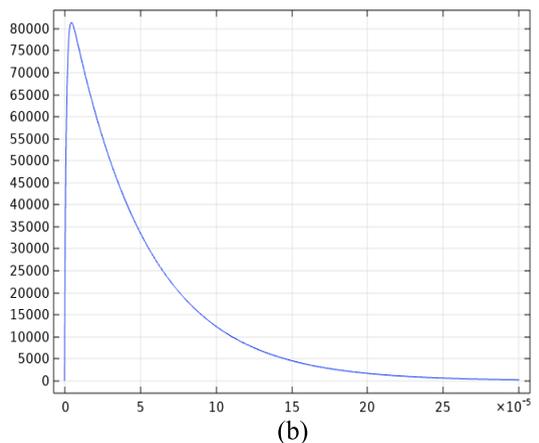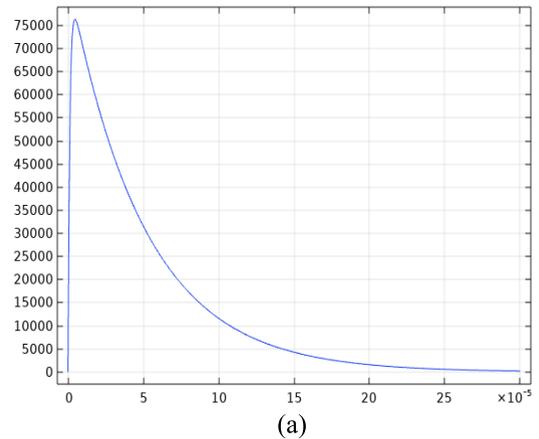
FIGURE 13. Lightning Impulse waveforms for the two simulation cases: (a) 75 kV, 1.2/50 μs, (b) 80 kV, 1.2/50 μs.

V. NUMERICAL MODEL

For the conduction of the simulations, the measured values of dielectric constants from the DRS method have been used for the transformer oil, SiC and TiO$_2$ NPs for the first return stroke at 25 kHz, based on IEC 62305-4 standard, i.e. 3.1, 9.2 and 5.8 respectively [61]. A point-sphere model in conso-

nance with the IEC 60897 standard was inserted in COMSOL Multiphysics software and the interfaces *Electrostatics* and *Transport of Diluted species* were used. The charge continuity equations for positive ions (12), negative ions (13), electrons (14) and charged NPs (15) that are related to Gauss'
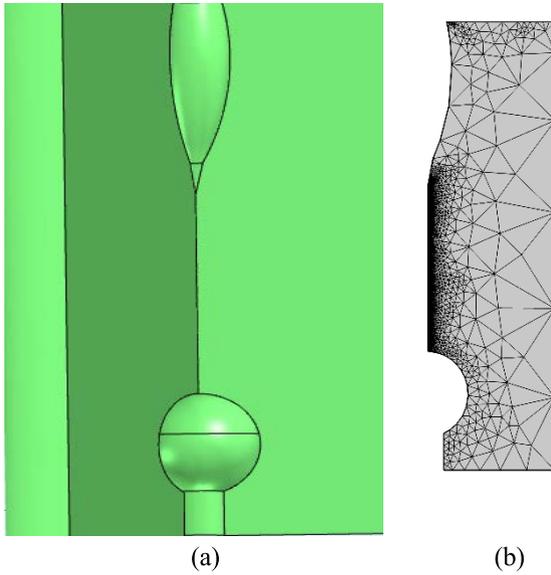
FIGURE 14. (a) Point-sphere gap in COSMOL Multiphysics environment before the application of Lightning Impulse Voltage, (b) Mesh distribution across the gap.

law (11) were introduced to model the LI behavior of pure oil and the corresponding NFs [23], [25], [41], [62], [63]:

$$\nabla \cdot (\varepsilon_1' \cdot \vec{E})$$
$$= \rho_+ + \rho_- + \rho_e + \rho_{np} \tag{11}$$

$$\frac{\partial \rho_+}{\partial t} + \nabla \cdot (\rho_+ \cdot \mu_+ \cdot \vec{E})$$
$$= G_I(|\vec{E}|) + \frac{\rho_+ \cdot \rho_e \cdot R_{pe}}{e} + \frac{\rho_+ \cdot \rho_- \cdot R_{pn}}{e} \tag{12}$$

$$\frac{\partial \rho_-}{\partial t} - \nabla \cdot (\rho_- \cdot \mu_- \cdot \vec{E})$$
$$= \frac{\rho_e}{\tau_\alpha} - \frac{\rho_+ \cdot \rho_- \cdot R_{pn}}{e} \tag{13}$$

$$\frac{\partial \rho_e}{\partial t} - \nabla \cdot (\rho_e \cdot \mu_e \cdot \vec{E})$$
$$= -G_I \left(\left| \vec{E} \right| \right) - \frac{\rho_+ \cdot \rho_e \cdot R_{pe}}{e}$$
$$- \frac{\rho_e}{\tau_\alpha} - \frac{\rho_e}{\tau_{np}} [1 - H(p_{np,sat} - p_{np})] \tag{14}$$

$$\frac{\partial \rho_{np}}{\partial t} - \nabla \cdot (\rho_{np} \cdot \mu_{np} \cdot \vec{E})$$
$$= \frac{\rho_e}{\tau_{np}} [1 - H(p_{np,sat} - p_{np})]$$
$$- \frac{\rho_+ \cdot \rho_{np} \cdot R_{pn}}{e} \tag{15}$$

where, ρ_+, ρ_-, ρ_e and ρ_{np} stand for positive ion, negative ion, electron, and charged NP densities, respectively. The charging process of the NPs is characterized by the Heaviside function $H(\rho_{np,sat}\text{-}\rho_{np})$ (16), and the saturation charge density $\rho_{np,sat} = n_{np} \cdot Q_s$, where n_{np} is the NPs' number density and Q_s is expressed from (9). The TiO₂ and SiC NPs' charging density limit was calculated as -277 and -359 C/m³ respec-
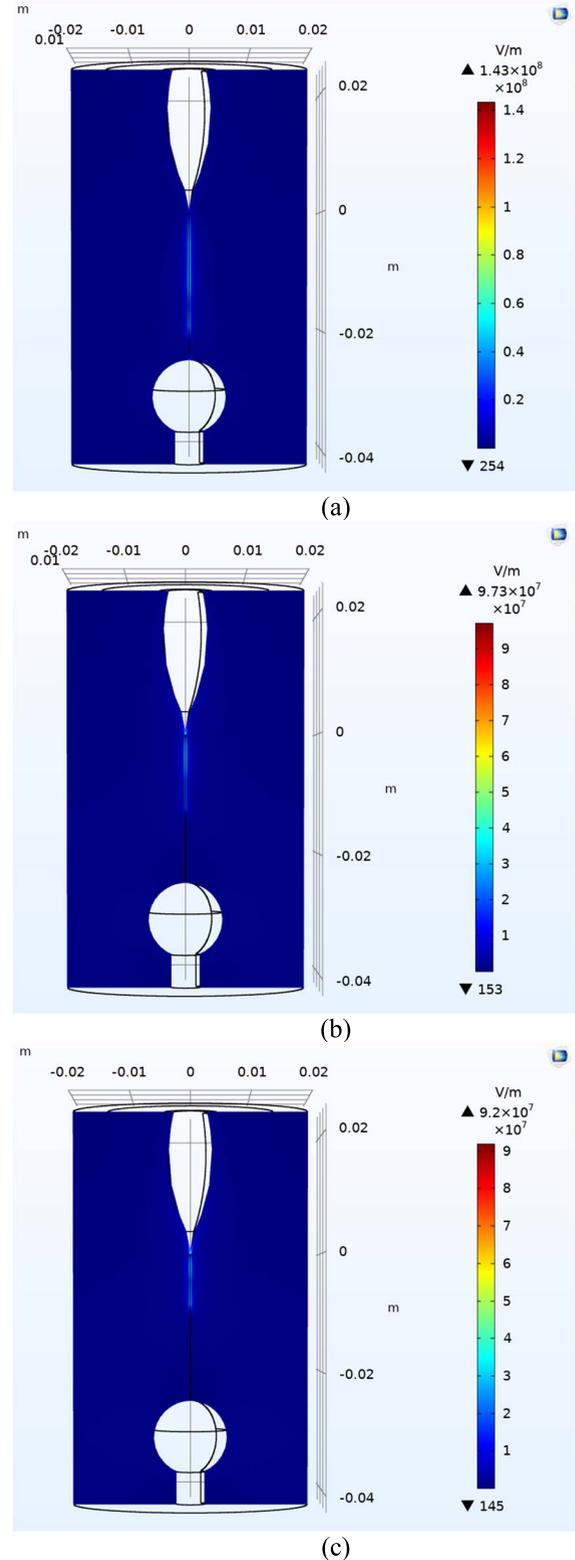
FIGURE 15. Electric field distribution in 3D across the gap at t = 15 μs after the application of the 75 kV LIV for: (a) Matrix oil, (b) tNF, (c) sNF.

tively.

$$H(p_{np,sat} - p_{np}) = \begin{cases} 0, & |\rho_{np}| \leq |\rho_{np-sat}| \\ 1, & |\rho_{np}| > |\rho_{np-sat}| \end{cases} \tag{16}$$
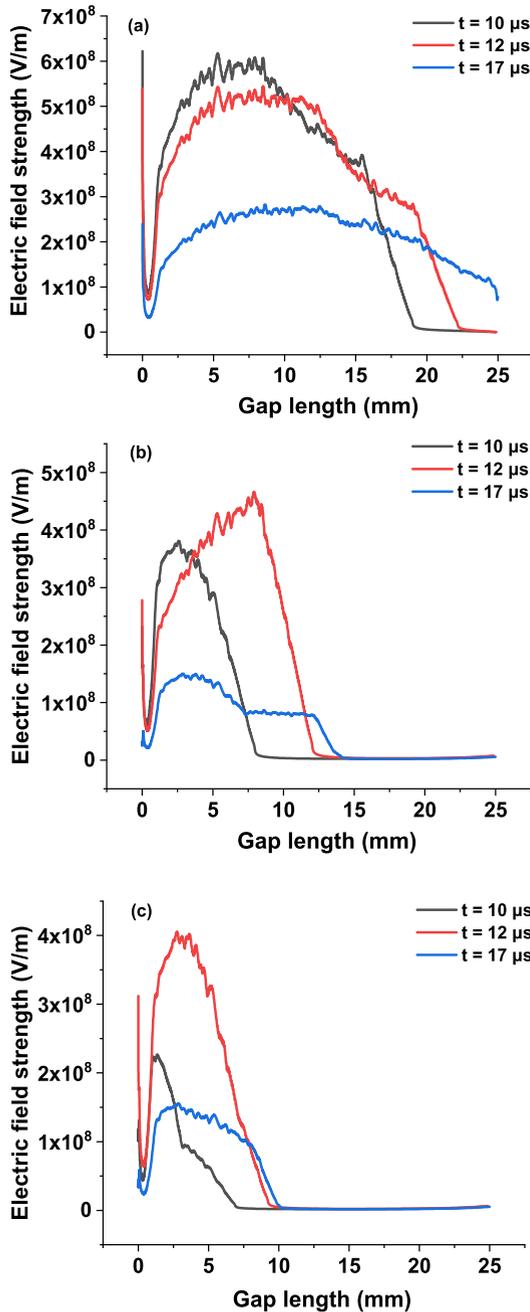
FIGURE 16. Electric field distribution across the gap at t = 10, 12 and 17 μs after the application of the 75 kV LIV for: (a) Matrix oil, (b) tNF, (c) sNF.

FIGURE 17. Space charge density across the gap at t = 10, 12 and 17 μs after the application of the 75 kV LIV for: (a) Matrix oil, (b) tNF, (c) sNF.

The ion-ion, R_{pn}, and ion-electron, R_{pe}, recombination rates have been calculated from (17) and (18) respectively [23]:

$$R_{pn} = \frac{e(\mu_+ + \mu_-)}{\varepsilon'_1} \qquad (17)$$

$$R_{pe} = \frac{e(\mu_+ + \mu_e)}{\varepsilon'_1} \qquad (18)$$

Lastly, the electric field ionization charge density rate $G_I\left(\left|\vec{E}\right|\right)$ has been calculated based on Zener formula (19)

[23], [25]:

$$G_I\left(\left|\vec{E}\right|\right) = \frac{e^2 n_0 a \left|\vec{E}\right|}{h} \exp\left(-\frac{\pi^2 m^* a \Delta^2}{e h^2 \left|\vec{E}\right|}\right) \qquad (19)$$

where, $\left|\vec{E}\right|$ is the electric field ionization threshold, determined to be 1×10^8 V/m in this work.

The definition of the variables that appear in the aforementioned Equations is provided in Table 5, along with their values used for the simulation scenarios based on previous works [23], [25]. For every simulation case, a LI voltage (1.2/50 μs) is applied at the positive point electrode with peak
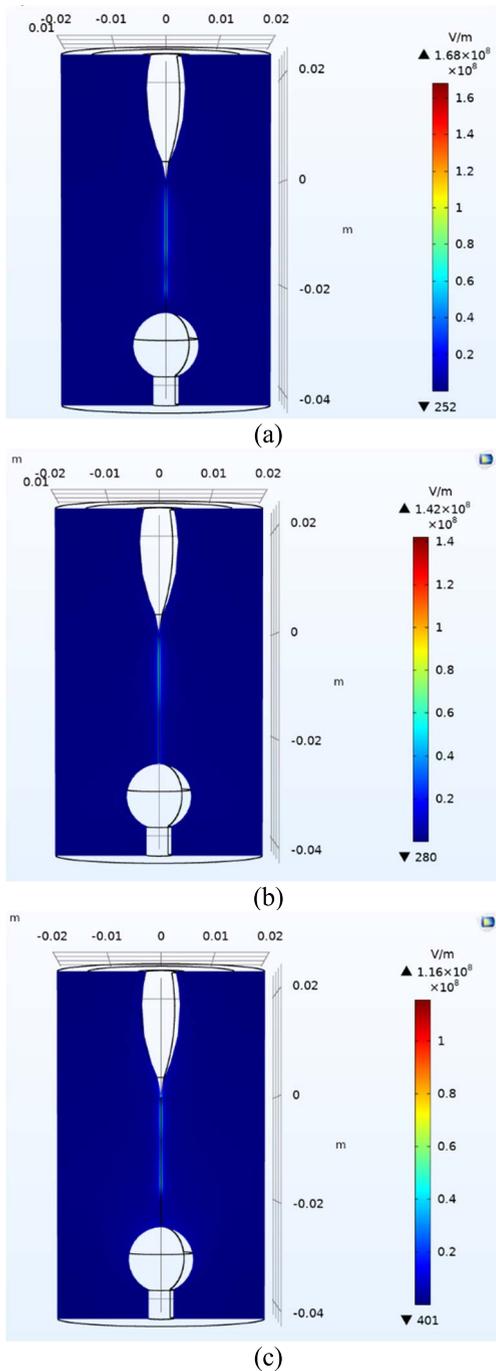
FIGURE 18. Electric field distribution in 3D across the gap at t = 15 μs after the application of the 80 kV LIV for: (a) Matrix oil, (b) tNF, (c) sNF.

FIGURE 19. Electric field distribution across the gap at t = 10, 12 and 17 μs after the application of the 80 kV LIV for (a) Matrix oil, (b) tNF and t = 10, 12, 17 and 20 μs for (c) sNF.

values of 75 (Fig. 13 (a)) and 80 kV (Fig. 13 (b)) for the three under study oil samples. The gap with the point-sphere geometry according to IEC 60897 standard, imported in COMSOL Multiphysics, is depicted in Figure 14(a). The free triangular mesh distribution used to distinguish the geometry is presented in Figure 14(b). The duration of each simulation case was 20 μs with a time step of 0.5 μs. For every LIV voltage level, the electric field distribution and space charge density across the 2.5 cm gap have been recorded at selected

time intervals, i.e. at 10, 12 and 17 μs after the application of the LIV at the positive point electrode. Apart from that, the electric field strength across the gap at t = 15 μs has been illustrated in 3D for every under-study sample at each LIV level.

A. LIGHTNING IMPULSE VOLTAGE LEVEL OF 75 KV
Figure 15(a)-(c) depicts the electric field distribution across the gap 15 μs after the application of the positive LIV in terms of the matrix, tNF and sNF samples respectively. From
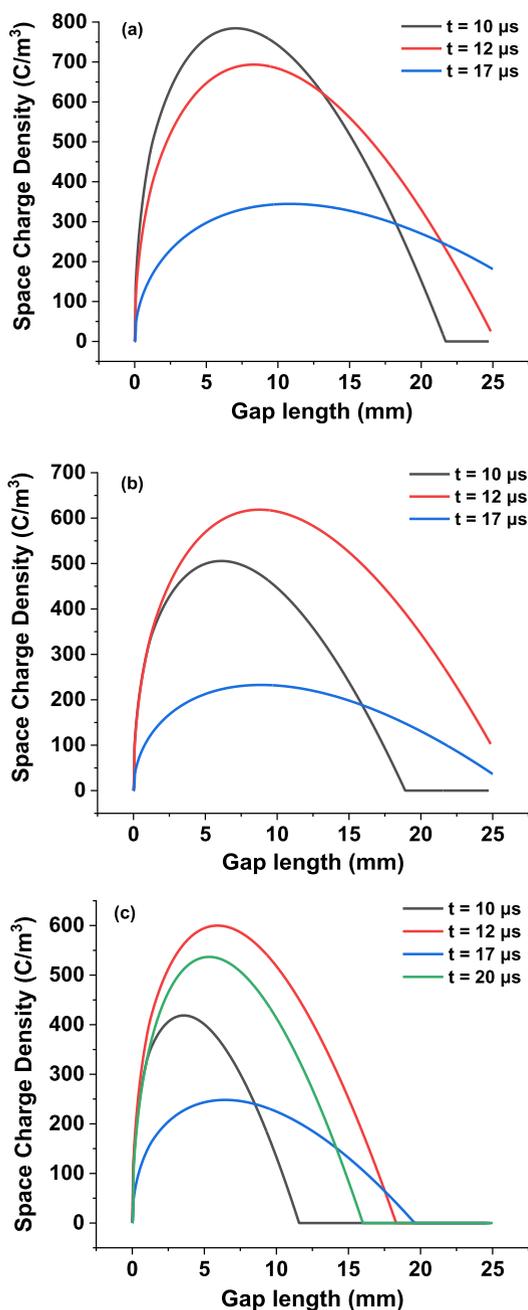
FIGURE 20. Space charge density across the gap at t = 10, 12 and 17 μs after the application of the 75 kV LIV for: (a) Matrix oil, (b) tNF, (c) sNF.

these plots, it is evident that, in the case of the matrix oil, the streamer has propagated significantly towards the negative sphere electrode and could cause breakdown, unlike the two NFs. This is clearer from the electric field and space charge plots of Figures 16(a)-(c) and 17(a)-(c) respectively. Higher electric field and space charge values across the gap are recorded for the matrix oil case, which extend across the whole gap. In fact, based on Figure 16(a) and 17(a) it seems that at t = 17 μs the gap is bridged, so the 75 kV LIV could be a possible candidate for BDV. In terms of the two NF samples, the electric field and space charge density are weakened and

restricted to 1.25 cm and 1 cm gap for the tNF and sNF respectively, with their values being more decreased for the sNF. It seems that the 75 kV LIV is not enough to cause breakdown in both NF samples due to the ability of NPs to act as electron scavengers, while the higher ability of SiC NPs is also demonstrated in simulation results through lower values of electric field and space charge density leading to delay of streamer propagation.

B. LIGHTNING IMPULSE VOLTAGE LEVEL OF 80 kV

Figure 18(a)-(c) depicts the electric field distribution across the gap 15 μs after the application of the positive LIV for the matrix oil, tNF and sNF samples respectively. Higher electric field values are recorded in relation to the first case, as expected, with matrix oil characterized from the highest, facilitating the breakdown, as observed from Figure 18(a). Figures 19(a)-(c) and 20(a)-(c) demonstrate the electric field and space charge density across the gap for the under-study samples. From Figures 19(b) and 20(b), which are referred to tNF, it seems that this voltage level is enough to cause the breakdown at about 17 μs after the LIV application. The space charge density across the gap significantly exceeds their saturation charge density, consequently the remaining charge is enough to bridge the gap and cause breakdown. Unlike the tNF sample, this voltage level does not cause breakdown of the sNF sample. As depicted in Figures 19(c) and 20(c), lower space charge density is accumulated in the gap hindering the streamer propagation, which however has bridged about 2 cm at 17 μs. For this reason, the depiction of electric field and space charge density distributions at 20 μs is necessary. Based on the same graphs, streamer propagation has receded to 1.5 cm, therefore it can be concluded that the breakdown with the application of 80 kV LIV has been avoided for the sNF sample. The results of the simulation at 75 and 80 kV LIV show a good match with the experimental ones presented in Section IV.

VI. CONCLUSION

In this work, a comparative study has been conducted regarding the influence of TiO$_2$ and SiC NPs at the same weight percentage ratio (0.004% w/w) on the thermal and dielectric performance of NEO FR3. The most important findings are summarized as follows:

- Both prepared NFs demonstrate great improvement of the thermal diffusivity and conductivity, with sNF's conductivity mostly enhanced, by 58%, while the enhancement in terms of tNF is about 25%.
- The dielectric constant of both NF samples is decreased relatively to the matrix oil for a frequency range from 1-10^6 Hz, while all samples show decreased values of dielectric constant with temperature increase. sNF sample is characterized from the lowest dielectric constant, which is an indication that the addition of these kinds of NPs leads to a more insulating dielectric liquid.

- The DDF of both NF samples under room and high temperature is decreased with respect to NEO. However, DDF is increased for all of them with temperature rise.
- sNF sample shows the most improved LI BD strength under positive polarity, by around 10% with respect to the NEO.
- With the development of a numerical model, it has been shown that greater values of electric field and space charge density are recorded at LIV level of 75 kV for the Matrix oil, while the sNF sample demonstrates the lowest ones both at 75 and 80 kV levels, verifying the experimental results.

As part of future work, it would be of major interest to compare the numerical simulation results to the ones of NFs suffering from rapid aggregation, in order to verify the influence of NPs' agglomeration on the reduction of dielectric strength.

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