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Effect of the Coexistence of Al_2O_3 Nanoparticles and Water on Transformer Oil Electrical Performance

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ABSTRACT It has been found that the introduction of nanoparticles (NPs) into transformer oil can alleviate the destructive effect of water on the insulation properties. Researchers have begun to explore the mechanism behind this phenomenon and try to provide possible explanations. However, the effect of water on the electrical properties of NF has not yet been explained definitively due to many factors, such as the NP concentration and external electric field. In this paper, mineral oil-based Al_2O_3 NFs with a wide range of NP concentrations and moisture content are prepared and tested to study the effects of their coexistence on the electrical properties of transformer oil. Diagnostic techniques (including ac breakdown voltage, thermally stimulated current, and dielectric spectroscopy) have been used to measure the dielectric properties of samples with varying relative humidity. The experimental results show that different NP concentrations and relative humidity can obviously affect the electrical performance of transformer oil. The breakdown voltage of the NF significantly improved compared with that of pure oil with the introduction of Al_2O_3 NPs at varying relative humidity. The shallow trap density in NF significantly increases due to the introduction of NPs with different relative humidity. The proper mechanisms of the water forms with different water contents are discussed based on the results of the experiment. The study of the electrical properties of NFs with different relative humidity is a good complement to current studies in this field.

INDEX TERMS Dielectric spectroscopy, nanofluid, relative humidity, thermally simulated current.

I. INTRODUCTION

Mineral oil is one of the key insulation liquids for high-voltage equipment worldwide, and its high dielectric strength provides insulation support for the normal operation of high-voltage devices [1]. The development of transformer oil with high dielectric strength is extremely important because of the demand for transformers with a small volume and a high voltage rate in electrical systems [2], [3]. A particularly novel work aiming to improve the insulating characteristic of transformer oil is the development of dielectric nanofluids (NFs) by suspending nanoparticles (NPs) in transformer oil. In 1998, Segal *et al.* [4] first added magnetic NPs to transformer oil and measured the resulting dielectric properties. The results of the electrical breakdown show that, for positive streamers, the breakdown voltage of NFs is almost twice that of the base oils during lightning impulse tests. The results are very encouraging and arouse the attention of many researchers toward the dielectric properties of NFs. Subsequent researchers prepared NFs by dispersing NPs composed of semiconductors [5], [6], insulators [7], [8], and materials with special properties [11], [12] and tested their electric properties. It has been demonstrated that these NFs have much greater dielectric breakdown strengths than the base transformer oil.

In the studies mentioned above, the water content in the NF remains constant or is not considered. Water is one of the most important factors affecting the insulation of transformer oil. Water generation is inevitable in the high-voltage and high-temperature environment of the transformer. It has been found that the probability of multimolecular water clusters will significantly increase under the high relative humidity in transformer oil, resulting in the accumulation of space charge, which distorts the internal local electric field and creates weak links [13], [14]. Many studies have shown that water not only affects the insulation characteristics of transformer oil but also has high practical value [15], [16]. In addition, it is noteworthy that the introduction of magnetic NPs (Fe_3O_4) can effectively alleviate the damage of water on dielectric properties [4]. It opens a door for the introduction of additives into transformer oil to reduce the destructive effect of water on the dielectric properties. To date, only a small fraction of research groups has focused on the destructive effect of moisture on the dielectric properties of modification transformer oil. Jin *et al.* [17] found that the AC breakdown voltage of mineral oil is obviously enhanced with high water content. They indicated that the breakdown voltage is strongly related to the hydrophilicity of NPs. Meanwhile, Atiya *et al.* [18] have found that the breakdown strength of NFs with different moisture content was alleviated due to the effect of moisture on the electrostatic stability in the electrical double layer. Du *et al.* [19] measured the dissipation of the space charge of oil-based TiO_2 NF with high relative humidity. They pointed that the strong affinity of a Gouy–Chapman–Stern layer for water molecules gives rise to a change in the NP–oil interface structure. According to what is known in solid physics and colloid chemistry, NPs in liquids will form a double electrical layer on the surface. However, we noticed that the simple double electrical layer model has some limitations and cannot fully explain the results found in different published reports. Furthermore, the traditional double-layer model deals only with molecular-scale ions. Transformer oils are complex systems that contain various ion impurities as well as cluster particles, such as that of water.

In this paper, mineral oil-based Al_2O_3 NF is prepared and measured with different NP concentrations and relative humidity. The diagnostic techniques, including the AC breakdown voltage, thermally stimulated current, and dielectric spectroscopy, are carried out to study the dielectric properties of transformer oil with different NP concentrations and relative humidity, and the introduction of Al_2O_3 NPs for alleviating the moisture in transformer oil is investigated. The effect of the NP concentration on the electrical properties and the combination of different forms of water clusters with NPs in the NF are discussed under different relative humidity conditions. A large unit model formed by water and NPs based on different water content is proposed to explain the effect of their coexistence on electric properties of transformer oil. Water is inevitable for long-term operation of transformers; research on the introduction of Al_2O_3 NPs into transformer oil to reduce the destructive effect of water on the dielectric properties can provide a reference for the design of the transformer.

II. METHOD

The mineral oil used for this study is called kakamayi #25. This type of mineral oil is widely used in high-voltage transformers and high-voltage DC (HVDC) converter stations. The relative permittivity of the oil is $2.2\varepsilon_0$, where ε_0 (8.85×10^{-12} F/m) is the vacuum permittivity. The Al_2O_3 NPs used for the experiment are purchased from DK Nanotechnology. The morphology of the as-prepared Al_2O_3 NP is characterized by a JEOL JEM-2100F high-resolution transmission electron microscope (TEM). The average diameter of the Al_2O_3 NPs is less than 20 nm, as shown in Fig. 1. Some of the properties of Al_2O_3 NP (including the charging relaxation time [8]) used to prepared NF and base oil are described in Table 1.

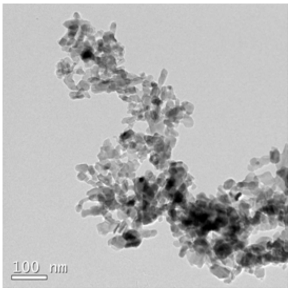

FIGURE 1. TEM image of Al_2O_3 NP.

TABLE 1. Main property of the pure oil and Al_2O_3 NP.

	ε (F/m)	σ (S/m)	ρ (g/cm^3)	τ(s)
Oil	$2.2\varepsilon_0$	10^{-12}	0.895	-
Al_2O_3	$9.9\varepsilon_0$	10^{-15}	-	42.5

A. PREPARATION OF NF

The mineral oil-based Al_2O_3 NF is prepared by dispersing Al_2O_3 NPs into the mineral oil. The Al_2O_3 NPs used in this experiment are purchased from Beijing DK Nanotechnology Company. The surfaces of the received alumina NP are left untreated. The material is placed in the vacuum drying oven at 525 K for 24 h. Batches of 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L, and 50 mg/L volume concentration of Al_2O_3 NF are prepared by mechanical stirring for 20 min. To avoid particle agglomeration, ultrasonic dispersing equipment is used to uniformly disperse NPs into transformer oil. The prepared mineral oil-based Al_2O_3 NF is colorless and transparent. Then, to avoid the influence of microbubbles, which could be created during the sonication treatment and mechanical stirring, all the samples are degassed in a vacuum oven for approximately 24 h. Due to the high ambient humidity level in the laboratory during the test (relative humidity of 25–65%), it is not possible to decrease the moisture content of the samples to less than 0.003 mL/L. To obtain the samples with suitable relative humidity, a constant-temperature and -humidity chamber is adopted. The samples are placed in the container for 15 days so that the moisture is distributed evenly in the samples to reach a stable state. Take a sample (1200 mL) with 40% relative humidity as an example at room temperature (298 K). The water in the sample is 0.011 g. The preparation procedure of the Al_2O_3 NF is shown in Fig. 2. The relative humidity in the samples varies from 10% to 80%. In this paper, the water content in all the samples is measured

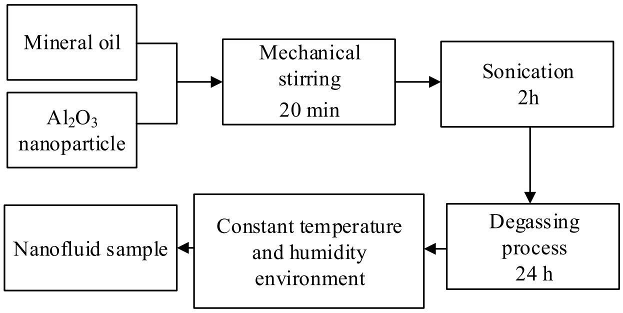

FIGURE 2. Flowchart of a preparation produces for mineral oil-based Al_2O_3 NF.

using a JF-5 Rishang Coulometric Karl Fischer moisture meter (made by Daqing Rishang Instrument Manufacturing Co., Ltd., in China). The Al_2O_3 NF samples are transparent.

B. AC BREAKDOWN

The AC breakdown strength is the maximum voltage level, at which the transformer oil loses insulation and allows current to pass through it. It is one of the most important factors in transformer design. The AC breakdown voltages of the prepared samples are measured according to the ASTMD1816 standard using aJKJQ-3BaoDing Automatic 50 Hz Electrical Breakdown equipment. The gap of brass-sphere-capped electrodes is 2.5 mm. The increase rate of the voltage is 2 kV/s. The time interval after each breakdown is set as 3 min. The water content in all samples is tested before and after each voltage breakdown test. The breakdown voltage of each sample is tested 5 times, and 6 groups of data are measured each time. The test temperature is 25 centigrade. The change range of water content of NF and base oil is 0.001-0.005 ml/L.

C. DIELECTRIC SPECTRA

For electrical property measurement, a Concept 80 Broadband Dielectric Spectrometer is employed. This device can measure the dielectric properties (complex permittivity, dielectric loss factor, complex capacitance, complex conductivity, etc.) of dielectrics in an extremely wide bandwidth and under variable temperature conditions. The frequency ranges of this testing equipment are from 3×10^{-6} Hz to 3 GHz, the testing temperature is from 113 K to 723 K, the resolution can reach 10^{-5}, and the WinDE-TA software can be configured to compare and analyze different dielectric spectrum curves. In this paper, the test frequency range is between 10^{-1} Hz and 10^5 Hz. The temperature is from 260 K to 363 K.

D. TRAP CHARACTERIZATION

From [20] and [21], it is known that thermally stimulated current (TSC) measurements can be used to investigate the nature and origin of charge carriers in dielectrics, including the change in the number and energy of trap sites. TSC is the current released from a pre-electrostressed dielectric as a result of heating. First, a negative DC field of 2.5 kV/mm is applied to the tested sample for 20 min at 323 K. Next, the temperature is quickly lowered down to 260 K. After that, the equivalent capacitor of electrodes is discharged for 1 min. Finally, the equipment begins measuring the TSC by raising the temperature at a rate of 2 K/min. To ensure the accuracy of the test, each sample is tested three times.

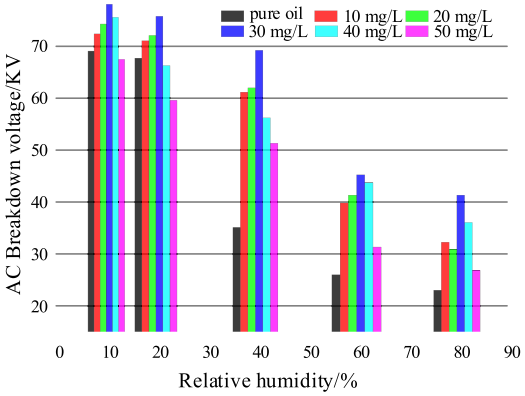

FIGURE 3. Breakdown voltage of samples as a function of relative humidity and NP concentration.

III. RESULTS

A. RESULTS OF AC BREAKDOWN VOLTAGE

The results of the breakdown voltage of the NF and pure oil as a function of relative humidity and NP concentration are shown in Fig. 3. It is clearly seen that the AC breakdown voltages of all samples quickly decrease with increasing relative humidity. The breakdown voltages are only slightly different between the NF and pure oil at low relative humidity ($\leq$ 20%) and are only related to the concentration of NPs. It is worth noting that the breakdown voltages of NFs are significantly increased from those of pure oil with increasing relative humidity and low decrease rate of electrical strength in the NF. This shows that the damage of water to the breakdown strength of NF is alleviated. The breakdown strengths of NF reach the maximum when the concentration of NPs is 30 mg/L, as shown in Fig. 3. This shows that there is a certain matching relationship between the concentration of NPs and the relative humidity, such that the breakdown voltage of mineral oil-based Al_2O_3 NF reaches the maximum. The maximum breakdown voltage of NF can be significantly improved to twice that of pure oil at a relative humidity of 40%. The maximum breakdown voltage is also increased to 1.6 times under a relative humidity of 80%. Because multi-molecular water clusters reduce the breakdown strength of oil more than single water molecules [22], based on the breakdown mechanisms of dielectric liquids [23], we think that the improved breakdown performance of the NF is largely attributed to the water-confinement effect of the NPs or the NP–oil interface zone. Due to the diversity of water types with different water content, it is necessary to discuss the effects of different types of water-association structures on breakdown strength. The possible mechanism of the formation of NPs and water under an applied field is discussed in Section 4.

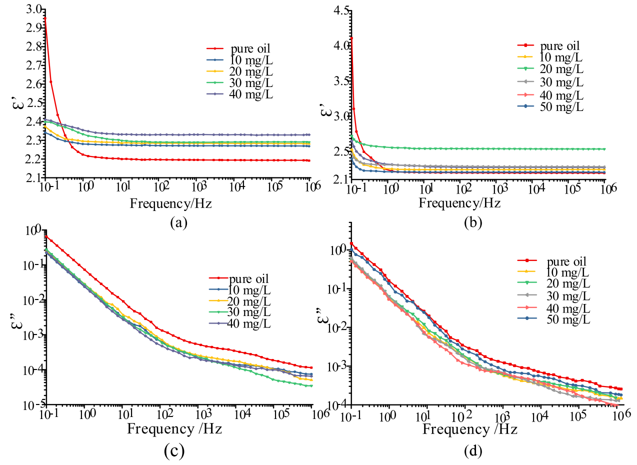

FIGURE 4. **Real parts of the permittivity of pure oil and NFs with relative humidity of (a) 20% and (c) 40%, imaginary parts of permittivity of pure oil and NFs with relative humidity of (b) 20% and (d) 40%. The insets are the NP concentration.**

B. RESULT OF DIELECTRIC SPECTRUM

The dielectric properties may be obtained by examining the variation of ε' and ε'' (the real and imaginary parts of the permittivity) as a function of the frequency. Fig. 4 shows the results of the NFs and pure oil of ε' and ε'' with different relative humidity; the insets are the NP concentration. Fig. 4(a) and 4(c) have a relative humidity of 20%, and Fig. 4(b) and 4(d) are at 40%. From Fig. 4(a) and 4(b), it is clearly seen that ε' is sensitive to frequency at low frequency ($f < 10$ Hz), mainly as ε' decreases rapidly with increasing frequency. It is interesting to note that the permittivity of the NF is different from that of pure oil with different relative humidity, indicating the smaller value and lower rate of descent of the permittivity of NF. As above mentioned, the probability of water molecules in transformer oil forming water clusters increase significantly under high water content. The clusters may contain some population of water oligomers $(H_2O)n$ with different bonding schemes among the water molecules, which can result in different polarization degrees of the liquid. This phenomenon becomes more obvious at lower frequencies, where the molecules have more time to orient their polarization vectors of different origin with the externally applied field [24]. The phenomenon is attributed to the introduction of NPs, which form dipole moments to restrict the response of water to the external applied field.

The value of ε' remains unchanged at high frequency ($f > 10$ Hz) with different relative humidity, and it is only related to NP concentration due to the different water forms has little time to respond to the applied field. It is consistent with the Maxwell–Garneet formula, as shown in equation (1); as the volume fraction of the particles increases, the effective dielectric constant of the NF will increase, and it is only related to the concentration of NPs.

$$\frac{\varepsilon_{eff}-\varepsilon_m}{\varepsilon_{eff}+\varepsilon_m}=\psi\frac{\varepsilon-\varepsilon_m}{\varepsilon+2\varepsilon_m} \tag{1}$$

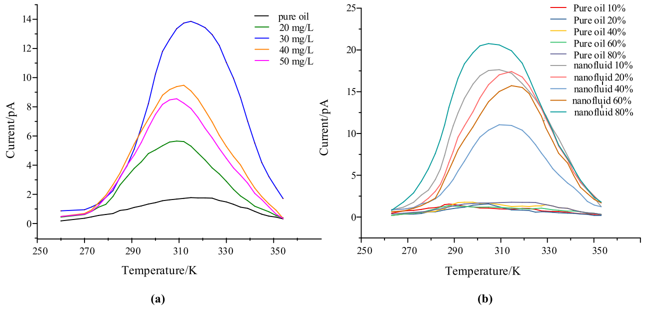

FIGURE 5. **(a) TSC test results of thermally stimulated current in the pure oil and NF at a relative humidity of 20%; the inset is the NP concentration. (b) TSC results of pure oil and NF with different relative humidity stressed at 3 kV/mm; the concentration of NPs is 30 mg/L.**

where ε_{eff} represents the effective dielectric constant of the system, and ε_m and ε denote the dielectric constant of oil and NP, respectively. Ψ is the volume fraction of NP.

From Fig. 4(c) and 4(d), it is clearly seen that ε'' of all sample decreases rapidly when the test frequency is between 10^{-1} Hz and 10^3 Hz. ε'' of the NF is smaller than that of pure oil under the test frequency with different relative humidity; the concentration of NPs in the sample has little effect on ε''. The imaginary permittivity of the pure oil compared with the low (20%) and high (40%) relative humidity, from the red curves in Fig. 4(c) and 4(d), it can be seen that the change without NP doping is similar, where the ε'' of the dielectric constant decreases monotonically with increasing frequency. The different forms of water have enough time to respond to the applied field, we believe that the main contribution of loss is the polarization response of different water forms with different relative humidity. Based on the results of the test, as shown in Fig. 4(a) and 4(b), ε' of the samples remain essentially unchanged when the frequency is greater than 10 Hz. It is well known that in the dissipation factor $\tan\delta = \varepsilon''/\varepsilon'$, $\tan\delta$ is proportional to ε''. The difference between the samples of pure oil and NF is only the different content of NPs. The dielectric losses of NF are smaller than that of pure oil at the power frequency ($f = 50$ Hz) due to the limitation of the water clusters by the NPs.

C. RESULT OF TSC

From the TSC results in Fig. 5(a), it is seen that the peak values of TSC curves for NF significantly increase than that of pure oil, and the current value of NF is closely related to the Al_2O_3 NP concentration at low relative humidity ($\leq 20\%$). The peak value of current for NF is almost 7 times than that of pure oil at the NP concentration 30 mg/L. A large number of experimental datum are measured. For the clarity and aesthetics of the graphics, a continuous curve is used as shown in Figure 5. Since the peak value of TSC curve is related to maximum trap density in a dielectric liquid [20]. The shallow trap density significantly increases with the introduction of Al_2O_3 NP, it is consistent with the [25]. It is noteworthy that the trap density decreases on the contrary, when the concentration of NPs is more than 40 mg/L because

NPs are prone to agglomerate into larger particles with high NP concentration. From the Fig. 5(b), the value of the pure oil is reasonably constant with the relative humidity increasing. However, a larger peak value of the shallow trap center is formed with relative humidity increase. The maximum current value of NF increased nearly 10 times than that of pure oil with the relative humidity 80%. It can be assumed that Al_2O_3 NF has a higher trap density than the pure oil with high relative humidity. From the [26] know that the charge carries have higher mobility in a dielectric nanocomposite. The charge carries have high mobility at high relative humidity and the space charge dissipates rapidly in Al_2O_3 NF under external field. The destruction of local electric field and the accumulation of space charge by water clusters can be mitigated. Therefore, it can be judged that compared to pure oil, mineral oil–based Al_2O_3 NFs have higher dielectric strength than that of pure oil with high relative humidity. However, the relative humidity is too high, due to there are not enough NPs to inhibit their destructive effect, the excess water will form charge channels and the breakdown strengths of NFs decrease at high relative humidity, as shown in Fig. 3.

IV. DISCUSSION

It is necessary to discuss the structure of water, namely, different relative humidity and the complexity of the form of water (individual molecules, pairs of molecules, and clusters) in transformer oil with different water content, as shown in Fig. 6. The water in oil exists in the form of a single molecule or a pair of water molecules under low water content ($< 20\%$); in addition to these, water is also present in the oil in the form of clusters at high water content (40%–80%). Due to the polarization of the NPs and different forms of water under the external applied field, these small units or clusters of water are immobilized around the NP, forming a large unit. This large unit structure will affect the electrical performance of transformer oil.

The results in Fig. 3 show that the breakdown voltages of NFs improved from those of pure oil with different relative humidity. There is a small variation in the breakdown voltages of NFs and pure oil at low relative humidity ($< 20\%$). According to [22], we believe that water exists as small units (single water molecules or pairs of water molecules) in transformer oil. All these small water units are immobilized by NPs; some NPs do not even have these small water units on their surfaces when the concentration of the NPs increases. Small amounts water cannot obviously affect the dielectric strength of the transformer oil. Therefore, the breakdown voltages of the NF are only related to the concentration of NPs. Under high relative humidity ($> 40\%$), the water in oil mainly exists in the form of clusters. Because the water clusters reduce the breakdown strength of oil more than single water molecules, the breakdown voltages of all samples are significantly lower than those of low water content. However, the breakdown voltages of NFs are significantly higher than those of pure oil. This indicates that the damage water causes to the breakdown strengths can be effectively alleviated. Based on the above analysis, we believe that the relationship between the breakdown voltage and NP concentration is not obvious as the relative humidity increases further.

A small amount water in transformer oil has a great influence on the electrical performance. Under low humidity (20%), all the small water units in NFs are fixed by the NPs. Because the relative permittivity of Al_2O_3 NPs (9.9) is far less than that of water (79) at room temperature. the immobilization of NPs affects the response of these small units to external field; hence, the permittivity of the NF is smaller and the rate of the NF is lower than that of pure oil at low test frequencies ($f < 10$ Hz), as shown in Fig. 4(a). However, under high frequencies, the clusters and small units do not have enough time to respond to the external field, and the real permittivity of the pure oil and NFs remains constant and is related to NP concentration, as shown in Fig. 4(a) and 4(b). Dielectric losses of NFs are less than that of pure oil under the test frequency, as shown in Fig. 4(c). This phenomenon is also observed in high moisture content (40%), as shown in Fig. 4(d). This is mainly because of the fixed effect of NPs on different forms of water, which reduces their polarization response to external field. It is noteworthy that at the same frequency ($f < 10$ Hz), the dielectric constant of high moisture content is greater than that of low moisture content, the reason for this phenomenon is that the clusters have more time to orient their polarization vectors with the externally applied field.

The trap characteristics of NFs and pure oil are tested using the TSC method. To evaluate the trap energy depth, it is necessary to calculate the electrical potential distribution produced by the permanent dipole moment of chemical defects. Here, the dipole moment is defined as $m = qL$, where q is the electric charge and L is the distance between two dipole electric charges. The electrical potential distribution, $V(r, \theta, \varphi)$, is given in spherical coordinates [27] as follows.

$$V(r,\theta,\phi) = \frac{m\cos\phi}{\varepsilon_0\varepsilon_r 4\pi r^2}\sin\theta \tag{2}$$

The induced trap depth corresponds to the maximum potential at the surface of the large unit. At this time, the distances of the permanent dipole moment, $L = D + 2r$, where D is the diameter of the NP (in this paper, $D = 20$ nm), r is the distance of the water group to the surface of the NP. Due to the immobilization of NPs on small units, L is much larger than that between chemical defects; hence, the deeper electron trap of the large unit is introduced, although r can be negligible under low relative humidity (20%), as shown in Fig. 5(a). The immobilization of water by NPs reaches saturation as the moisture content increases and the small water unit and clusters independently respond to the field. Based on [22], it was found that the monomer form of water has the highest total dipole moment. Therefore, the trap densities increase significantly with the higher relative humidity, as shown in Fig. 5(b). It is well known that electrical breakdown in liquid is closely related to the electron transport process, which includes electron-hopping transport in the traps and

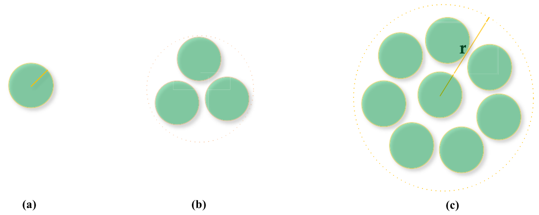

FIGURE 6. **Schematic illustrations of water forms in transformer oil with different water content: (a) and (b) at low water content; (a), (b), and (c) at high water content.**

transport in a delocalized state [23]. Although the trap density increases significantly, the distance between the large units becomes closer and the breakdown process becomes easier.

V. CONCLUSIONS

In this study, mineral oil-based Al_2O_3 NFs were prepared, tested, and compared with the base oil. The breakdown voltage of the NF significantly improved compared to that of pure oil with the introduction of Al_2O_3 NPs at varying relative humidity. The complexity of the form of water is discussed with respect to relative humidity. The real part of the permittivity of all samples (ε') is sensitive to the external field at low frequencies ($f < 10$ Hz). ε' of the Al_2O_3 NF has a smaller value and slower descent rate than that of pure oil. It is mainly the response of different forms of water to the applied field. The value of ε' remains constant under high frequencies ($f > 10$ Hz). The dielectric losses of NF are smaller than that of pure oil at different relative humidity. The shallow trap density in NF significantly increases due to the introduction of NPs with different relative humidity.

the effect of water on the electrical properties of NF has not yet been explained definitively due to many factors, such as the NP concentration and external electric field. The study on the inhibition of Al_2O_3 NPs on water can provide a reference for the design of transformers because water is inevitable in the long-term operation process of transformers. The proposed large-unit model, which is formed by NPs and water under an external field, can explain the effect of their coexistence on the electric properties of NF. All the test results indicate that the introduction of NPs can improve the electrical properties of transformer oil and restrain the damage of water. The possible mechanism of large units is discussed, but we do not think it is the only one that requires more experiments to verify it.

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