Effect of Iron/Titania Based Nanomaterials on the Dielectric Properties of Mineral oil, Natural and Synthetic Esters as Transformers Insulating Fluid

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ABSTRACT There is a need to develop upgraded insulant which offers superior dielectric properties than mineral oil in terms of cooling, insulation, and efficient functioning under extreme weather conditions. These oils are nanofluids that are synthesized by dispersing nanoparticles in the oil. These fluids are tested with their ability to withstand various faults under operating conditions by determining the dielectric and electrical properties like ac breakdown strength, relative permittivity, and dielectric dissipation factor. A comparison between nanoparticles and their effect on insulating oils has been widely discussed and presented. In this study, three nanoparticles namely Fe₃O₄, TiO₂, and Fe-Cu are dispersed in three different types of oils namely mineral oil (i.e., transformer oil used as insulating liquid in transformers), synthetic ester oil (i.e., pentaerythritol ester), and rapeseed oil (natural-ester transformer fluid). These nanofluids are considered upgraded insulant due to its superior insulation characteristics compared to pure mineral oil. Synthetic ester-based oils and natural rapeseed oil are tested for insulation capabilities for better results, which show they have all the potential to replace transformer oil in high voltage equipments.

INDEX TERMS Upgraded insulant, Ester based oil, Rapeseed oil, Nano-based fluid, electrode configuration, ac breakdown voltages, Relative permittivity, Dielectric dissipation factor, dielectric change.

I. INTRODUCTION

Transformers are the essential component for transmission and distribution of electric power at high voltages in a power system [1]. The operating life of transformer primarily depend on its insulation system, and according to a survey by CIGRE, it was found that insulation breakdown is the key reason for transformers failure [2]. Transformer failure may be disastrous, which may result in the interruption of power flow and financial losses [2]. Therefore, the main aim of the researchers is to develop such an insulant that exhibit upgraded dielectric properties than mineral oil and ester-based oils as outlined in literature [3]-[6].

Mineral oil is primarily used as insulating liquid in transformers due to its excellent dielectric strength and cooling property [7]. The high dielectric strength and better cooling capability of mineral oil makes it highly suitable for performing the dual function of insulation as well as cooling in power transformers. The increase in power demand has put enormous stress on the efficiency of dielectric oils used in electrical equipments such as transformers, circuit breakers and switchgears [7]. Mineral oil is a limited resource that is continuously depleting, and has many disadvantages like lower flash point, non-biodegradable and environmental contamination due to leakage as it is a petroleum derivative. These issues have driven the attention of the researchers to find an alternative for mineral oil. Environment friendly insulating oils are introduced to address these issues [8] that can perform effectively in the overload conditions, and exhibit better insulation and cooling properties than mineral oil for energy applications. Till now many researchers have shown the influence of nanoparticles on oils, and claim the upgraded insulating properties of mineral oil and ester-based nanofluids [9], but they did not compare them all
II. SELECTION AND SYNTHESIS OF NANOFLUIDS

A. Selection of oils and nanoparticles
The selection of nanoparticles is done purely on the basis of their ability to improve dielectric strength. Three nanoparticles are used for the study, namely Iron oxide (Fe$_3$O$_4$), Titanium oxide (TiO$_2$), and Fe-Cu (Iron copper alloy). The TEM images of nanoparticles are shown in figure 1. Three different types of oils used in the experiment are mineral oil, synthetic ester oil, and rapeseed oil (natural ester or vegetable oil-based). The ester oils used for testing were taken from two different gallons provided by the company M&I Materials Ltd. Oils and nanoparticles specifications are shown below in table 1, 2 respectively.

B. Preparation of Nano Fluids
Nanofluids have been prepared by adding nanoparticles in the base oil. There are two methods for the preparation of Nanofluids namely, one-step method and two-step method. The process discussed, and used for the preparation of nanofluids is the two-step method.

TWO STEP METHOD
This is the most commonly used method for nanofluid preparation. It involves low cost and better compatibility. The oil sample consists of 450 ml of pure oil and 50 ml of oleic acid (surfactant). The nanoparticle is dispersed in the oil using magnetic stirrer followed by ultra-sonication. Magnetic stirring is a thermal process that mixes oleic acid in the oil, and then finally nanoparticles are uniformly dispersed in the oil via process called ultra-sonication, which involves generation of high frequency (40 kHz) waves to uniformly mix nanoparticle in the pure oil as shown in figure 2. Because of its low cost and better compatibility widespread with other nanoparticles, this method is widely used [15] [16-18].

During magnetic stirring process the oil samples were heated at a temperature of 50°C for about 30 minutes using a hot plate magnetic stirrer, which removes the humidity and unwanted moisture present in the oil. Furthermore, the oils that were used for testing, have very low moisture content as reported in table 2 [10-11]. The oils were filtered using a fine paper filter. The headspace of the oil container was properly sealed and kept air tight which help keep the
liquid dry, as the dielectric properties of an insulating liquid are affected by humidity and contaminants.

![Figure 1: TEM representation of conductive (Fe$_3$O$_4$ & Fe-Cu) and semiconductive (TiO$_2$) nanoparticles.](image)

**Figure 1:** TEM representation of conductive (Fe$_3$O$_4$ & Fe-Cu) and semiconductive (TiO$_2$) nanoparticles.

**Stability of Nano Fluids**

The oleic acid is used as a surfactant to modify the nanoparticles surface and, to improve the stability of nanofluid samples. The role of surfactant is, to act as a bridge between nanoparticle surface and insulating liquid so that nanoparticles do not form aggregate with time. Long term stability of nanofluids is the major concern for their application as insulating oil, because nanoparticles when dispersed in the base oil are subjected to various stresses like van der Waals forces and electrostatic forces etc. And, therefore nanoparticles may agglomerate under these forces, and eventually lose their size and functionalities. The DVLO theory [19] is used to determine the stability of nanofluids. According to the theory, if the electrostatic repulsive forces are higher than the van der Waals attractive forces, then the nanofluid dispersion is stable. However, further addition of surfactant will develop additional repulsive force between nanoparticles due to formation of double chain on the nanoparticle surface, shown in figure 3. Hence agglomeration of nanoparticles is restricted with oleic acid to obtain stable dispersions.

![Figure 2: Schematic diagram showing the preparation of nanofluid](image)

**Figure 2:** Schematic diagram showing the preparation of nanofluid

**III. POSSIBLE MECHANISM OF DIELECTRIC FLUID ALTERATION**

The process of dielectric modification due to conductive, semiconducting, and insulating nanoparticles is explained. The most important reason for the improved breakdown strength is the electron trapping by the nanoparticles. Basically, nanoparticles perform as electron scavengers in the base oil. Due to the applied electric field, nanoparticles are rapidly polarized. Therefore, fast-moving electrons that were created at high field were captured, and then rapidly released, hence converting themselves into slow in motion negatively charged particles. Therefore, the rate for charge decay increases and mobility of charged electrons is reduced effectively. Hence propagation of streamer is hampered as result breakdown strength is improved.

A model [20] is proposed which plays a leading role in dielectric modification. In this proposed model relaxation time constant ($\tau_r$) of the system (nanoparticle/oil) is determined using the formula given below in equation (1):

$$\tau_r = \frac{(2\epsilon_1 + \epsilon_2)}{(2\sigma_1 + \sigma_2)}$$  \hspace{1cm} (1)

Where $\epsilon$ is the oil permittivity and $\sigma$ is the dc conductivity of the mineral oil and nanoparticle respectively.

If system time constant value is shorter than microseconds, that is the time involved in the propagation of streamer then nanoparticle will capture the fast-moving electrons present in the oil and convert these electrons into low mobility negatively charged particles [20].
IV. EXPERIMENTAL TECHNIQUE

As explained previously, due to applied electric field fast electrons are produced, and nanoparticles get polarized rapidly. So, conductive nanoparticles (Fe$_3$O$_4$) with a low value of the time constant would capture the fast electrons in transformer oil, and reduces their mobility [20]. The potential drop increases along the streamer length, and hence breakdown strength is increased. So, by adding conductive nanoparticle dielectric performance is enhanced.

The theory of time constant cannot explain the change in breakdown strength due to addition of semi-conductive nanoparticles (TiO$_2$). These nanoparticles act as electron scavengers and produce shallow traps in the oil. The fast electrons produced due to applied electric field would be captured in these shallow traps and then released from it. This process of charge trapping and de-trapping would eventually reduce the speed of electrons in the oil, thereby suppressing the streamer growth by TiO$_2$ nanoparticles. Hence, results in improved breakdown strength of oils. This concept is well explained with the help of TSC method [14]. The measurement of thermally stimulated current (TSC) by spectroscopy reveals that the formation of shallow traps centre for nano-oil (0.418 eV) is higher than pure mineral oil (0.416 eV), which leads to more charge trapped hence more charge density in nano-oil than pure mineral oil. This shows that the TiO$_2$ nanoparticles will improve the breakdown strength of mineral oil.

As shown in figure 4, nanoparticles act as a perfect dielectric in the oil, because under the applied electric field nanoparticle will get charged (polarized), and becomes an electric dipole. This transformation of a large number of nanoparticles into electric dipoles improves the breakdown strength of the oil. There is a hindrance in the development of net space charge at the streamer tip due to the low mobility of particles which reduces the streamer growth in the oil and results in higher breakdown strength [21].

A. AC Breakdown voltage

Dielectric breakdown test serves as an important part in acceptance of insulating oil in transformer. The experiment to determine AC breakdown voltage is carried out on a Fully Automatic Oil Breakdown Voltage Tester (i.e. PE-AOBDV.M100 model), which contains a chamber that holds the oil sample and electrodes inside. The experimental standard used to determine ac breakdown voltage is ASTM D877. Electrodes are made up of brass material having a specific gap length. The diameter of the electrode is 10mm. The rate of rising of voltage applied is 2kv/sec. The experiment is carried out at room temperature. Nanofluid is filled inside the chamber containing an electrode configuration up to the oil level marking. This chamber is then kept inside the Fully Automatic oil Breakdown Voltage Tester by opening the closure of the instrument. The standing time is fixed to 4 minutes, stirring time to 2 minutes and intermediate time to 3 minutes while performing the experiment for each nanofluid as per standard. Experiment is conducted on two different electrode configurations (i.e. sphere-sphere and Mushroom-Mushroom) as shown in figure 5. ASTM D-1816 standard-based electrodes are used to analyze breakdown characteristics under different electrode configurations. Nanofluid breakdown results in the formation of an arc between electrodes. Twelve breakdown readings of each sample are calculated and by averaging all the readings, the final value is reported. Two batches for each individual oils were used for testing. The AC breakdown tester to measure dielectric breakdown of nanofluids is shown in Figure 6 (a). AOBDV tester gives the breakdown voltage with 1% accuracy. [6], [15].

B. Relative Permittivity and Dissipation Factor (DDF)

Relative permittivity defines the intensity of dielectric polarization in insulating oils. Oils having high relative permittivity can contribute in forming more uniform electric field in the insulation system. Dissipation factor is the vital parameter that measures dielectric losses in the oil and its higher value indicate the presence of contaminants or impurity inside the oil [21]. The experimental standard used to determine relative permittivity and dissipation factor of oils is ASTM-D-924, IEC 60247. The PE-ORDF-2 apparatus, (which consists of oil cell and dissipation factor meter) is used to determine the relative permittivity and dielectric dissipation factor of oils, and corresponding nanofluids based on the ratio of capacitance of oils with that of air using the dissipation Factor Bridge. The oil cell maintains uniform air gap between the electrodes. The two piece instrument containing dissipation factor meter, and oil cell with its connection to measure relative permittivity and dissipation factor of oils is shown in figure 6 (b). The set of experiments were performed at room temperature. Five set of readings were performed for each nanoparticle concentration, and average value is reported.
V. ANALYSIS ON RESULTS AND DISCUSSION

A. AC Breakdown Voltage

The experimental results for AC Breakdown voltage of pure oils, and its corresponding nanofluids at four different concentrations of nanoparticle i.e., 5mg, 10mg, 15mg and 20mg is graphically represented with error bars as shown in Figure 10-11. The maximum enhancement of ac breakdown voltage has been observed, and calculated using the graphs plotted, and formula given below. It was observed that for a particular concentration of nanoparticle there is a maximum increase in breakdown voltage of particular nanofluid. The decrease in breakdown voltage at higher concentrations can be attributed to agglomeration of nanoparticles that form conductive chains, and bridge the electrode gap [21].

\[
\%\text{enhancement} = \frac{\text{Maximum Breakdown Voltage of nanofluid}}{\text{Breakdown voltage of insulating oil}} \times 100
\]

Comparison between nanoparticles on the basis of three different oils and between oils on the basis of the nanoparticle is being done, and consequently graphs are plotted between average breakdown voltage and nanoparticle concentration, which is shown in graphs from figures 9 to 10. [22-27]

B. RELATIVE PERMITTIVITY

The result for relative permittivity is formulated. It is observed that the relative permittivity of nano-modified oils is higher than pure oils. The results show that magnetic nanoparticles gives maximum increment in relative permittivity of pure oils. Its value is calculated using the capacitance of nanofluid and pure oil, and comparing it with the capacitance of air. Values at four different (5, 10, 15 and 20mg) nanoparticle concentrations is calculated, and compared as shown in tables 3-5. The enhanced relative permittivity of nanofluids is credited to high permittivity of magnetic nanoparticles that acts as perfect dielectric in the base oil [28].

Table: 3. Relative Permittivity of Mineral oil and similar nanofluid after adding different Nanoparticles

| Concentration of NP | FeO₄ | TiO₂ | Fe-Cu |
|---------------------|------|------|-------|
| 0 mg                | 2.20 | 2.20 | 2.20  |
| 5 mg                | 2.12 | 2.20 | 2.21  |
| 10 mg               | 2.26 | 2.25 | 2.24  |
| 15 mg               | 2.24 | 2.23 | 2.23  |
| 20 mg               | 2.25 | 2.24 | 2.23  |
Table: 4. Relative Permittivity of synthetic ester oil and similar nanofluids after adding different Nanoparticles

| Concentration of NP | Fe$_3$O$_4$ | TiO$_2$ | Fe-Cu |
|---------------------|------------|---------|-------|
| 0 mg                | 3.20       | 3.20    | 3.20  |
| 5 mg                | 3.20       | 3.21    | 3.18  |
| 10 mg               | 3.21       | 3.24    | 3.23  |
| 15 mg               | 3.23       | 3.22    | 3.21  |
| 20 mg               | 3.24       | 3.22    | 3.21  |

Table: 5. Relative Permittivity of Rapeseed Oil and similar nanofluids after adding different Nanoparticle

| Concentration of NP | Fe$_3$O$_4$ | TiO$_2$ | Fe-Cu |
|---------------------|------------|---------|-------|
| 0 mg                | 3.10       | 3.10    | 3.10  |
| 5 mg                | 3.08       | 3.02    | 3.08  |
| 10 mg               | 3.18       | 3.14    | 3.16  |
| 15 mg               | 3.20       | 3.12    | 3.15  |
| 20 mg               | 3.22       | 3.13    | 3.16  |

C. Dielectric dissipation factor

The dielectric dissipation factor measures the dielectric losses in insulating oils. Results for dielectric dissipation factor for mineral oil, synthetic ester oil, and rapeseed oil along its corresponding nanofluids is shown in Figure 7-9. From the graphs, it can be seen that the dissipation factor of pure mineral oil is lower than Rapeseed oil, and higher than synthetic ester oil. It was observed that as the nanoparticle concentration increases the dissipation factor of oils and nanofluids increases except for TiO$_2$ nanofluid, which indicates improved dissipation factor due to addition of TiO$_2$ nanoparticles. It was also observed that magnetic nanoparticles (Fe$_3$O$_4$ and Fe-Cu) exhibit higher dissipation factor due to different dielectric property of the nanoparticles because these nanoparticles may form conductive chains in the oil, which starts conduction and reduces dissipation factor of oil [28].
Figure 10. Comparison between nanoparticles based on oils; based on mineral oil under S-S (a) and M-M (b), based on synthetic ester oil under S-S (c) and M-M (d), based on Rapeseed oil under S-S (e) and M-M (f).
Figure 11. Comparison between oils based on nanoparticles; 
Based on Fe₃O₄ nanoparticle under S-S (a) and M-M (b), 
Based on TiO₂ nanoparticle under S-S (c) and M-M (d), 
Based on Fe-Cu nanoparticle under S-S (e) and M-M (f).
C. Discussion
It can be seen from the graphs that; the voltage reaches its maximum value for the specific (optimum) concentration of nanoparticle. The reason, for the increase in ac breakdown voltage with the concentration of nanoparticles is attributed to the concept of interfacial layer, and high permittivity of nanoparticles might be the reason for enhanced relative permittivity of nanofluids. The interfacial layer between nanoparticles and the oil plays a vital role in enhancing the dielectric strength of nanofluids [29]. At lower nanoparticle concentration more traps are introduced in the oil due to large interfacial volume, as nanoparticles are uniformly distributed, which results in higher breakdown voltage. But, at higher concentration, nanoparticles form aggregate matter as a result losing their size properties and become micrometre size nanoparticles [30], thus the interfacial volume decrease, which reduces the number of traps. Hence, breakdown voltage starts reducing and phenomenon is well explained above in section III. Furthermore, at higher nanoparticle concentrations there is a decrease in the inter-particle distance of the added nanoparticles, which results in the formation of conductive paths or chain like structure above a particular threshold value [1]. Some amount of leakage currents may pass through the conducting paths in the oil, and eventually results in the reduction of ac breakdown voltage at higher concentrations of nanoparticles [31-32]. Other reasons that contribute for dropping the breakdown voltage may be the moisture present in the oil samples [33-35].

The moisture tolerance is very high in synthetic ester oil which means it can absorb far greater amount of water than mineral oil without affecting its breakdown strength. Therefore, experimental results show maximum breakdown voltage for synthetic ester oil than mineral oil and rapeseed oil. The consideration of nanoparticle concentration that maximum enhances the breakdown value is also very important for the purpose of commercialization [36-37].

For the purpose of statistical analysis, the standard deviation of the data is calculated and correspondingly error bars are plotted for all combinations of oils, nanoparticles and electrode configuration as shown in Figures 10-11. The graphs representation with error bars is important to analyse our results and its implementation.

Maximum increment in Breakdown Voltage of Transformer oil-based Nano oil, rapeseed oil-based nanofluid and synthetic ester oil-based nanofluid is as follows:

For Mineral Oil
The maximum increment is 32.20% for Fe-cu at 10 mg using the S-S configuration.
The maximum enhancement is 36.66% for Fe-Cu at 10 mg using M-M Configuration.

For Rapeseed Oil
The maximum enhancement is 33.19% for TiO$_2$ at 15 mg using S-S Configuration.

The maximum enhancement is 31.53% for TiO$_2$ at 15 mg using M-M Configuration.

For Synthetic Ester Oil
The maximum enhancement is 16.56% for TiO$_2$ at 15 mg using S-S Configuration.
The maximum enhancement is 9.91% for Fe-Cu at 15 mg using M-M Configuration.

After noticing considering change in breakdown strength, and based on comparison between different oils and nanoparticles, it is concluded that AC breakdown strength maximum increases by 32.20% under sphere-sphere (S-S) and 36.66% under mushroom-mushroom (M-M) electrode configuration for Fe-Cu Nanoparticles at 10 mg concentration as compared to unmixed i.e. pure mineral oil. Similarly, AC breakdown strength maximum increases by 16.56% for TiO$_2$ at 15mg under sphere-sphere configuration and 9.91% for Fe-Cu at 15mg under Mushroom-Mushroom configuration compared with base synthetic ester oil. And, AC breakdown strength maximum increases by 33.19% under sphere-sphere configuration and 31.53% under Mushroom-Mushroom configuration for TiO$_2$ at 15mg compared with pure Rapeseed oil.

Moreover, the results for relative permittivity of nano-modified oils show that there is a considerable increase in the value specifically at higher concentrations of nanoparticles. It is observed that magnetic nanoparticles (Fe$_3$O$_4$) shows maximum increment in relative permittivity of pure oils. The high permittivity of magnetic nanoparticles might be the reason for enhanced relative permittivity of nanofluids.

The minimum value of dissipation factor is obtained for synthetic ester oil based TiO$_2$ nanofluid among all the oils. The dielectric dissipation factor of oils reduces with increase in nanoparticle concentration, which might be due to formation of conductive chain, which may initiate conduction or the flow of leakage current in the dielectric [28]. Hence, losses in the oil results in the deterioration of dissipation factor of nano-oils.

VI. CONCLUSION
From the results, it can be concluded that breakdown voltage for M-M configuration is higher than S-S configuration for different combinations of nanoparticles and oils, because the mushroom electrodes have larger gap length between them than the sphere electrodes. It is also observed that the synthetic ester oil gives maximum breakdown voltage followed by rapeseed oil, then followed by mineral oil among all the tested oils. In general, synthetic ester oil exhibit excellent insulating characteristics, and could be a better alternative for transformers oil among all the tested oils. Moreover, dissipation factor of oils is worse than pure oils. The results have verified that nanoparticles have adverse effect on dissipation factor of oils. Considerable improvement in the relative permittivity value from 5 to 20 mg range of nanoparticle concentration is observed. The
reason for improved relative permittivity of nanofluids might be the higher permittivity of added nanoparticles. However, remarkable change can be seen on increasing concentration levels up to two hundred times in comparison to concentration levels that have been taken. Such high concentrations would lower the breakdown voltage of the oils. Therefore, there is a need to research further for the optimum nanoparticle concentration to be added in the oils, which not only form stable dispersions but exhibit excellent dielectric property to make it an alternative to mineral oils.

VII. REFERENCES

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