Characterization of Mineral oil Based Fe_3O_4 Nanofluid for Application in Oil Filled Transformers

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Abstract: In this paper, magnetite (Fe_3O_4) nanoparticles based nanofluid has been synthesized and characterized for application in oil filled transformers. The intent of this work is to check the feasibility of magnetite based nanofluid through appropriate characterizations. Magnetite nanoparticles have been synthesized by chemical co-precipitation method and the corresponding nanoparticles have been characterized by Raman spectroscopy, X-Ray diffraction, UV-Vis spectroscopy and Scanning Electron Microscopy. Further, ferrofluid has been characterized at different concentrations of nanoparticles those including 10%, 20%, 30%, and 40% w/v % to report optimal concentration for improved dielectric and thermal performance of the nanofluid. Electrical measurements such as resistivity, permittivity and loss tangent at different concentrations of magnetite are reported. It is inferred that 20% concentration of magnetite is an optimal concentration with an improvement of 34% of BDV. Importantly, with the application of magnetite nanoparticles overall thermal and dielectric behavior of the insulation fluid will be improved while reducing the size and weight of the transformers.

Keywords: Magnetite, Nanoparticles, Ferrofluid, Mineral oil, Transformer.

1. Introduction

Transformers are one of the significant parts of the electrical power system. As the power transmission system is opting for high voltage high power transmission, high performance insulating materials are drawing attention of the electrical power industry [1]. With increase in performance of the insulating materials, the size of the electrical equipment and cost of the machine is to be protected within the permissible values. In this point of line, critical research has been reported by numerous researchers across the globe through different approaches. Some of those include alternative insulating materials [2-3], performance improvement of existing insulating materials through appropriate additives [4], making use of nanodielectric materials [1] and improvements in diagnostics and prognostic studies [5]. In the recent past, nano dielectrics have drawn the attention of the industry and researchers with their technical advantages.

Transformer's insulation breakdown strength improvement leads to higher voltage level operation with less insulation requirements thus enabling reduction in size and weight of the transformer. Along with breakdown strength, improvement in thermal conductivity is helpful for achieving higher ratings at smaller size due to better heat dissipation [6]. Thus, improvement of thermal and electrical characteristics of oil/paper insulation is a major interest in the present scenario. Conventionally, mineral oil and cellulose insulation paper/pressboard are used for composite transformer insulation. Despite several critiques, mineral oil is still the most popular transformer insulating fluid due to its ease of availability, price and sound dielectric properties. But it has certain drawbacks from the perspective of biodegradability, low fire point, moisture absorption and sludge formation. In an attempt to overcome these drawbacks natural and synthetic ester oils were found as alternatives and are explicitly reported in [3, 7-8]. Ester oils provide better breakdown strength, fire point and bio-degradability thus making themselves a potential candidate for green technology. However, ester oils have some limitations like oxidation stability and cost [9]. Due cost and availability, ester oil still lags the mineral oil in

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practical applicability, making mineral oil as standing insulating material for the transformer in upcoming years. Such a standing of mineral insulating oils in the industry will significantly be facilitated through making use of the functional behaviour of the nanomaterials [1].

Thus improvement in dielectric and thermal properties of mineral oils through the application of nanomaterials is essential for future high voltage operations. In this situation, nanotechnology has opened a new pathway to meet this challenge as it has been found that the addition of several material nanoparticles in insulating fluid improves dielectric and thermal properties [10]. Nanoparticle based fluids were first introduced by Choi *et al*. in 1995 and the term 'nanofluid' was coined [11]. However, in 1998 Segal *et al*. were first to test transformer oil based ferrofluids and reported improvement in dielectric strength and thermal conductivity [12-13]. Initially, numerous researchers emphasized on thermal properties of nanofluids, especially thermal conductivity improvement. Electrical properties and significant diagnostic characterizations were least emphasized. Later on, exploration of dielectric properties was augmented. Mineral oil based nanofluids with various nanoparticles were tested. Silicon dioxide (0.02 wt %), titanium dioxide (0.005 wt %), zinc oxide (0.0005 % vol) and CCTO (0.01 w/v%) showed Breakdown Voltage (BDV) improvement of 27.4% , 31%, 8.3% and 36% respectively [14-16]. However, Fe_3O_4 nanoparticle based mineral oil has been one of the most promising nanofluids in terms of breakdown strength with a reported maximum BDV improvement of 40% with improved thermal conductivity[14-15]. The intent of this study is to report the properties of transformer oil based ferrofluid including electrical parameters like resistivity, permittivity and loss tangent at different concentrations. Significant diagnostic tools including, Raman spectroscopy, X-Ray diffraction, UV-Vis spectroscopy and Scanning Electron Microscope have been also reported. This paper also reports the nanofluid preparation procedure from the initial stage of nanoparticle synthesis using co-precipitation method and their characterization to the preparation of nanofluid at various concentrations.

2. Experimental Investigation

A. Synthesis of Nanoparticles

Co-precipitation method was used to synthesize Fe_3O_4 nanoparticles [17]. Analytical grade MERCK's chemical reagents were taken and used without further filtration. First, aqueous solutions of ferrous chloride ($FeCl_2.XH_2O$) and ferric chloride ($FeCl_3.XH_2O$) salt having a molar concentration in ratio 1:2 were prepared with deionized water. Then these two solutions were stirred on a magnetic stirrer for 10-15 minutes for proper mixing. Further, these two solutions were mixed together and stirred again for 10 minutes with the temperature maintained below 40^0C. The pH of the mixed solution was 0.4. Then ammonium hydroxide (NH_4OH) was gradually added to the solution with continuous stirring which resulted in black precipitate inside the solution. After complete precipitation, final pH of the solution is observed to be 9. Later, the solution was left overnight at room temperature so that the particles could be properly digested. Then the particles were washed several times with deionized water until its pH reached 7 where ammonia got completely washed off. Lastly, the particles were heated in an oven for 24 hours at 100^0C temperature for moisture removal and then collected and stored for further characterizations.

B. Synthesis of Ferrofluid

B.1. Functionalization of the nanoparticles

To improve the miscibility of magnetite nanoparticles with transformer oil, oleic acid was used as the surfactant. For capping of nanoparticles with oleic acid first 2.5 grams of nanoparticles were added in 60 ml of demineralized water below 60^0C under constant and gentle stirring. Then with the proportion of 1ml of oleic acid per gram of nanoparticles was added to the solution [18]. Resulted nanoparticles were capped with oleic acid as they attained hydrophobic nature and collected at solution's surface. Later, coated nanoparticles were washed with demineralized water and filtered out.

B.2. Preparation of Ferrofluid

The functionalized Fe_3O_4 nanoparticles were mixed in mineral oil under continuous stirring with constant speed. Four different concentration samples of ferrofluid as 10%, 20%, 30% and 40% (i.e. 0.1gm/l, 0.2gm/l, 0.3gm/l and 0.4gm/l) were prepared [19]. For the homogenous dispersion of nanoparticles, ferrofluid was ultra-sonicated for 30 minutes. Finally, to remove moisture, ferrofluid was kept in air circulated oven for 24 hours at 120^0C.Figure 1represents the schematic diagram of steps involved in the preparation of ferrofluid andFigure 2 shows the photograph of the ferrofluids prepared at various concentration.

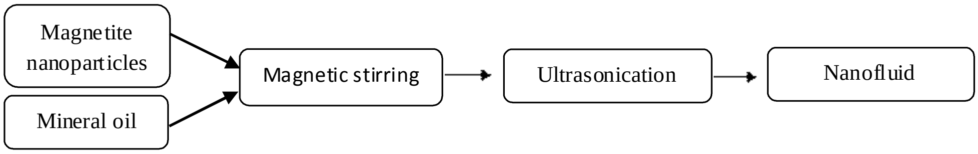

Figure 1. Schematic diagram of preparation of nanofluid

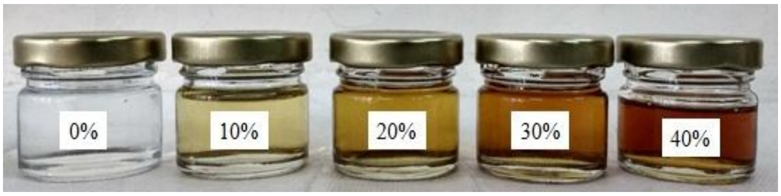

Figure 2. Nanofluids at various concentration (0%=virgin mineral oil, 10%=0.1 gm/l, 20%=0.2 gm/l, 30%=0.3 gm/l, 40% =0.4 gm/l).

B.3 Nanoparticles characterization

Synthesized nanoparticles were characterized by Zeta Sizer Test using Malvern’s Zeta Sizer Nano instrument, Powder X-ray diffraction (PXRD) with Rigaku Smart Lab, diffractometer using Cu-Kα radiation (λ = 0.154nm) at 45 kV and 100 mA, Raman spectroscopy with RENISHAW Raman microscope with laser wavelength 514 nm, UV-Vis spectroscopy with Perkin Elmer’s UV-Vis NIR spectrometer and finally by scanning electron microscopy (SEM). Raman spectroscopy was done to characterize the synthesized material that whether Fe_3O_4 has formed or not or is it formed with any other iron oxide forms like Fe_2O_3, FeO. The Raman peaks were in accordance with magnetite reference peaks which ensured that Magnetite nanoparticles were formed and no other iron oxide form is present. XRD and SEM were done to identify the crystalline structure and morphology respectively of prepared nanoparticles for proper characterization. Authors didn’t found any data relating BDV of oil with the crystalline structure of nanoparticles. UV-Vis was done to derive the Tauc plot so that conducting nature of particles can be known. Tauc plot analysis characterized the particles as semi conductive which was useful information to avoid the ambiguity in the literature whether magnetite nanoparticles are conducting or semi conductive.

B.4 Electrical parameters of nanofluid

Transformer oil is characterized by its physical, chemical, electrical and thermal properties. This paper focuses on the electrical properties evaluation of the mineral oil based ferrofluid like breakdown voltage (BDV), dissipation factor (loss tangent), resistivity and permittivity with respect to pure mineral oil. BDV test was done in automatic oil test set of ‘Rectifiers and Electronics’ in reference to ASTM standards D877. Spherical electrodes were used with 2.5 mm distance in between them. Test frequency was 50 Hz and a ramp voltage increasing with 2 kV/s was applied to electrodes. Resistivity, permittivity and loss tangent test were performed according to ASTM D924 standard by Eltel's ADTR-2K test set. It had a three-terminal guard cell, operating voltage was 500 V dc and the measurements were taken at 90°C.

3. Result and Discussion

A. Nanoparticle characterization results

XRD: XRD results are used to define the crystalline structure of the synthesized particles. Scanning steps of diffractometer were set at 0.02. Diffraction peaks with respect to six faces (111), (220), (311), (400), (422), (511), (440) and (622)are in accordance with JCPDS: 19-0629 confirming spinel structure of nanoparticles crystal [20]. Figure 3 represents the powder XRD test results of the synthesized Fe_3O_4nanoparticles

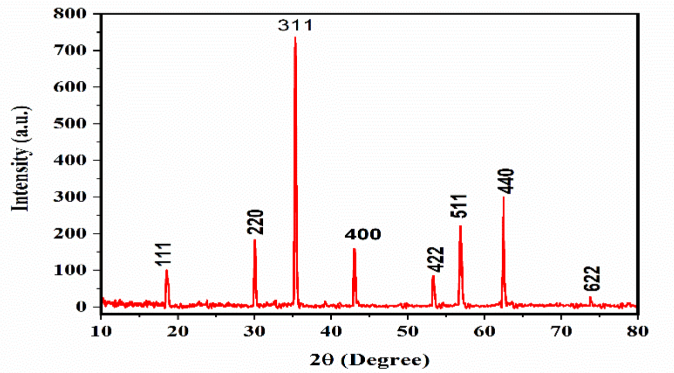

Figure 3. XRD results of magnetite nanoparticles.

SEM: Figure 4 represents the SEM image of synthesized Fe_3O_4 nanoparticles to study the morphological structure. It clearly shows particles are spherical in shape and size is 30-40 nm range.

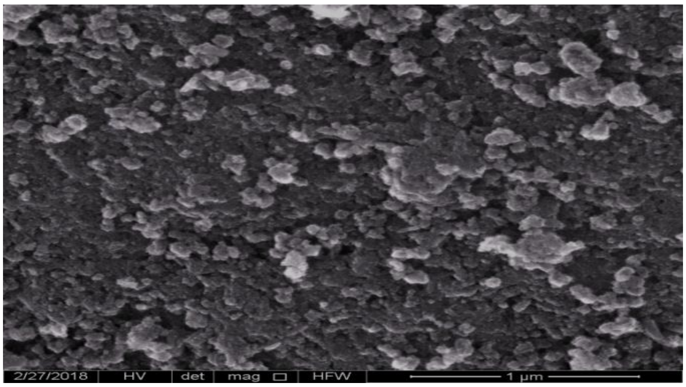

Figure 4. SEM image of Fe_3O_4 nanoparticles

UV-Vis: Though quantitative analysis of material is the most common application of UV-Vis spectroscopy, however, here it is used to determine the conductive nature of synthesized nanoparticles, whether it is conducting, semiconducting or insulating. It was determined with the help of Tauc plot derived from the wavelength-absorbance graph of UV-Vis Spectroscopy. Figure 5 shows UV-Vis spectroscopy absorbance curve whose peaks are in accordance with the Fe_3O_4 characteristic absorption wavelength of 492 nm [21]. Broad peaks have been observed because a number of various vibrational energy levels accompany different electronic level and transition takes place to and from various vibrational levels [22].

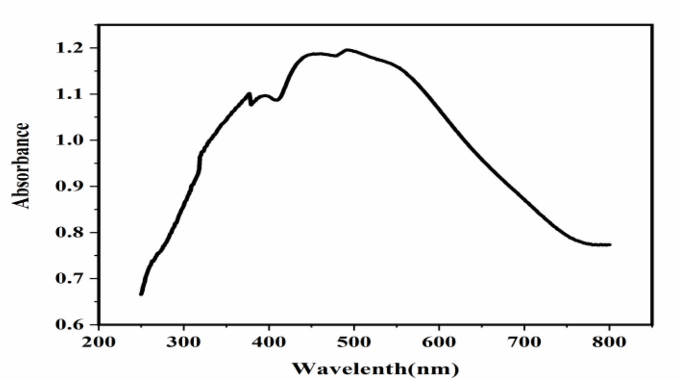

Figure 5. Fe_3O_4 UV-Vis absorbance plot

The graph is plotted between band gap energy (hν) on the x-axis and $(\alpha h\nu)^2$ on y-axis where ‘α’ is the absorption coefficient, ‘h’ is plank constant and ‘ν’ is frequency. From the graph, the band gap energy of the nanoparticles was found to be 1.65 eV which shows that the Fe3O4 nanoparticles are semiconductive in nature as reported energy gap for semiconductors is in between 0-3 eV [21]. Figure6 shows the direct band gap Tauc plot of Fe_3O_4.

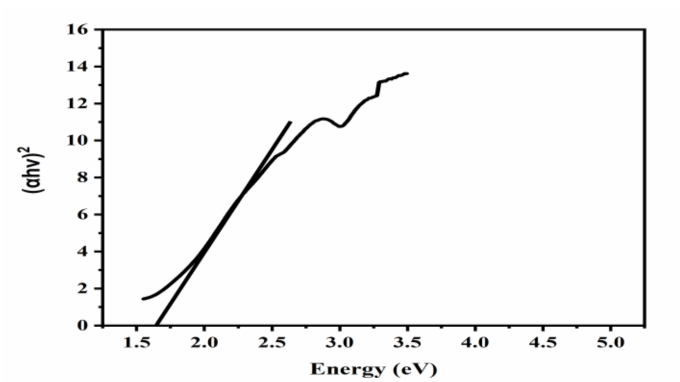

Figure 6. Direct bandgap Tauc plot for Fe_3O_4nanoparticle

Raman Spectroscopy: Raman spectroscopy is used to characterize prepared nanoparticles. The band obtained for Fe_3O_4 nanoparticles was at 675 cm^{-1} Raman shift. This band gap represents modes corresponding to Fe-O stretching and in accordance with the reference Raman spectrum [23-24]. Figure 7 shows the Raman graph plotted for magnetite nanoparticles with 514 nm laser at room temperature.

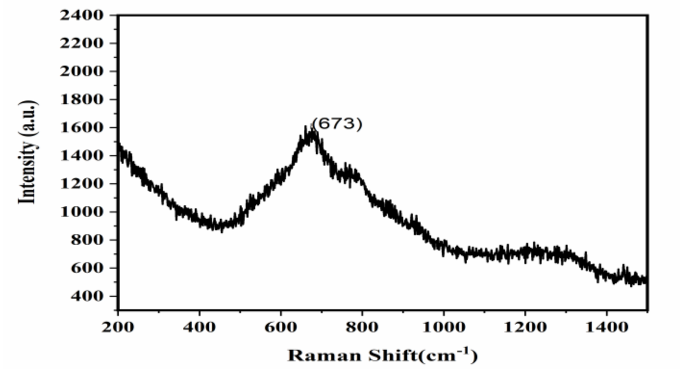

Figure 7. Raman Spectrum for magnetite nanoparticles

Zeta Sizer test result is used to know the size of the prepared nanoparticles. Methanol was used as the dispersant for particles in the test. Particle size determined by zeta potential test was 31.4 nm.

B. Ferrofluid Electrical Parameter Results

Breakdown Voltage (BDV): It is the voltage at which the oil breaks down when subjected to an AC electric field with a continuously increasing voltage [25]. BDV provides the dielectric strength of insulation.

Table 1 represents the BDV test data for mineral oil and various concentration ferrofluids. It also shows the percentage BDV improvement in different concentration ferrofluids with respect to pure mineral oil. For every sample, an average of six repeated breakdown voltage value was taken as final breakdown voltage. Table 1 clearly represents that 34% maximum BDV improvement was obtained with 20% concentration. For 10% and 30% concentration, the slight increment was observed than virgin MO BDV but less than BDV of 20% concentration and for 40% concentration, BDV value was even lower than that of the virgin oil. Figure 8 shows the variation pattern of BDV with increasing concentration. These BDV results are in agreement with the fact that BDV of virgin transformer oil improves with the addition of nanoparticles but only up to a certain concentration and after that particular concentration, its value starts decreasing [1]. Thus the optimum concentration of ferrofluid was taken as 20% and kept constant for further tests.

Table 1. BDV Test Results

S. No.	Sample Type	BDV	% Improvement
1	Virgin mineral oil (MO)	42.6	--
2	MO+ n (10%)	46.7	+9.1%
3	MO+ n (20%)	57.3	+34%
4	MO+ n (30%)	45.2	+6.35%
5	MO+ n (40%)	38.6	-9.8%

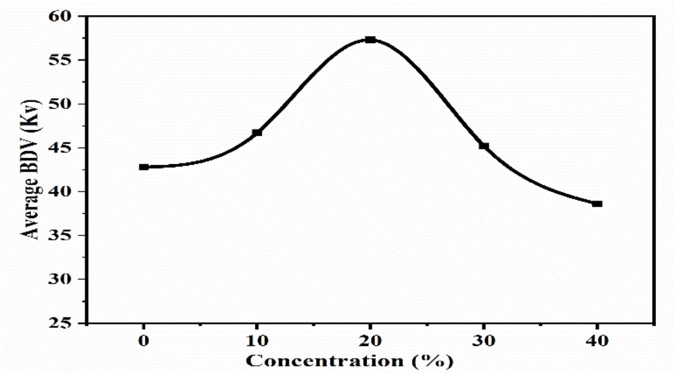

Figure 8. Breakdown voltage of different concentration ferrofluids.

The explanation of the improved breakdown strength of mineral oil on the inclusion of nanoparticles to certain concentration rests on the electron scavenging property of nanoparticles [19]. Due to this property nanoparticles can check the breakdown of insulation by reducing the streamer propagation as they can trap the generated electrons from the applied electric field. The explanation further elaborated by the space charge effect in mineral oil as breakdown strength is closely related to the electric field and internal space charge [26]. On application of electric field, space charge affects the inception and origination of streamer discharge [27]. With the introduction of Fe_3O_4 nanoparticles in mineral oil, its interface area act as electronic traps that can capture electrons and thus convert themselves into slow-moving charged particles in a nanofluid. This reduces the space charge density in the mineral oil and results in the more uniform distribution of electric field. Thus trapping of electrons by nanoparticles inhibits streamer progression in nanofluids resulting in breakdown improvement. Inhibition of streamer propagation also depends upon the charge relaxation time constant of nanoparticle material. If charge relaxation time constant of particles is shorter compared to time scales of streamer development then electrodynamics of streamer progression will be much affected i.e. shorter time constant will corresponds faster absorption of electron whereas if relaxation time constant of nanoparticles is comparable to the time scale of interest of streamer progression then they will have less effect on insulation electrodynamics [28]. The mineral oil/ nanoparticles charge relaxation time constant (τ_r) is given by-

$$\tau_r = \frac{2\varepsilon_1 + \varepsilon_2}{2\sigma_1 + \sigma_2}$$

where $\sigma_1, \varepsilon_{1,}\sigma_2\varepsilon_2$ are electrical conductivity and permittivity of the mineral oil and nanoparticles respectively. The charge relaxation time constant with magnetite nanoparticles was found to be 7.47×10^{-14}s which is much shorter than the microsecond streamer propagation time. Thus magnetite nanoparticles can capture space free electrons which lead to the reduced space charge density checking streamer propagation velocity and finally improving the breakdown strength.

Resistivity: It is defined as the resistance between opposite faces of a centimeter cube of liquid, expressed in ohm-cm [6]. It also signifies the dielectric strength of the insulation in direct proportion thus higher value is desirable. Table 2 represents the experimental resistivity values for pure mineral oil and various concentration ferrofluids which show that resistivity improves with the inclusion of nanoparticles but only up to a certain concentration and it is in complete accordance with BDV test result. The maximum increment was recorded for 20% concentration. For 10% and 30% concentration ferrofluid, resistivity was more than virgin mineral oil but less than that of 20% concentration and for 40% concentration, resistivity value was poor from pure mineral oil. Figure 9 shows this resistivity variation pattern.

Table 2. Resistivity test result at 90^0C and 500V DC

S. No.	Sample Type	Resistivity (Tohm-cm)	% Increment
1	Virgin mineral oil (MO)	498.8	--
2	MO+ n (10%)	998	+100.08%
3	MO+ n (20%)	3000	+501.44%
4	MO+ n (30%)	850	+70.40%
5	MO+ n (40%)	200.28	-59.9%

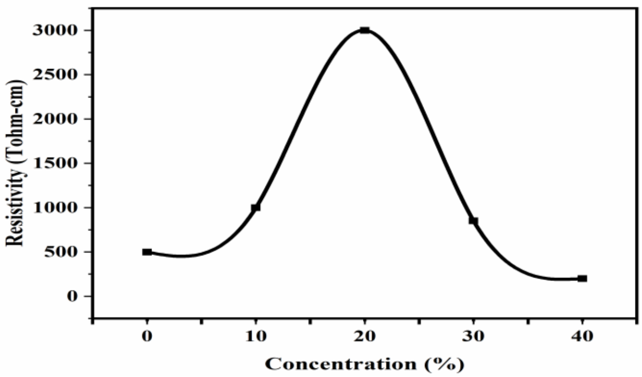

Figure 9. Resistivity of different concentration ferrofluids.

Loss tangent: For dielectric, it is the tangent of the angle (delta) by which the phase difference between applied voltage and resulting current deviates from π/2 radian, when the dielectric of the capacitor consists exclusively of the insulating oil [25]. It is the measure of loss of energy in form of heat inside the dielectric when subjected to electrical stress. Its minimum value is required for transformer application. Table 3 represents the loss tangent test data for various concentration samples. From the test result, loss tangent value for virgin mineral oil is 0.001 and it is less than any concentration ferrofluid. It was observed that with an increase in concentration there was a decrease in loss tangent. Loss tangent value variation with respect to different concentration is shown in Figure 10.

Table 3. Dissipation factor of ferrofluids at 90^0C

S.No.	Sample Type	Loss Tangent
1	Virgin mineral oil (MO)	0.001
2	MO+n (10%)	0.0206
3	MO+n (20%)	0.01718
4	MO+n (30%)	0.015
5	MO+n (40%)	0.00637

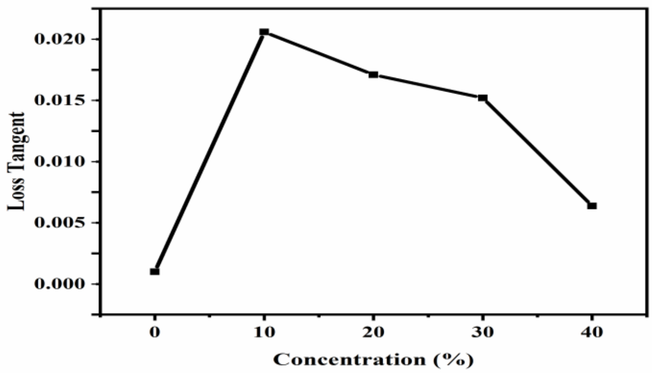

Figure 10. Dissipation factor result for various concentration ferrofluid

Permittivity: Permittivity can be seen in terms of relative permittivity. It is the ratio of the capacitance of the dielectric with respect to the capacitance of the vacuum [29]. Table 4 represents the permittivity test values and percentage decrement in permittivity value for different concentration ferrofluids with respect to pure mineral oil. Figure 11 shows the permittivity value variation pattern. It clearly illustrates that the due to the magnetite nanoparticles addition permittivity value of ferrofluids has decreased and maximum decrement was observed for 20% concentration ferrofluid.

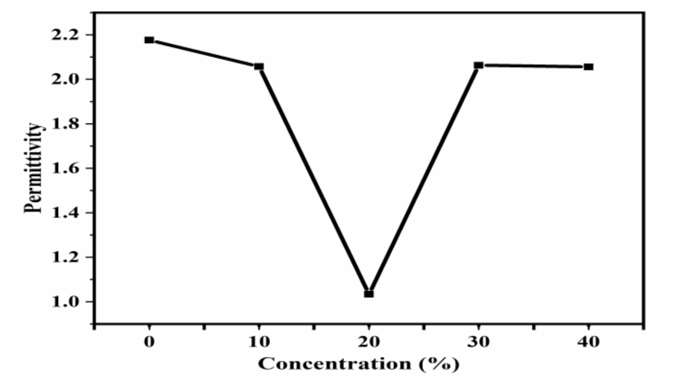

Figure 11. Permittivity value variation for different concentration

The significance of permittivity in transformer application is that it has an inverse relation with the electric field intensity so if two dielectrics are acting in series as in transformers then dielectric having less permittivity value will experience more electrical stress.

Generally, the permittivity value of mineral oil is considered to be around 2.2 and solid dielectric permittivity considered in the range of 3.6-4.5 [1]. Therefore the liquid insulation experiences more stress than that of solid insulation which is a favorable condition because liquid insulation is replaceable. Further decrease in permittivity value due to magnetite nanoparticles inclusion will increase stress on the liquid insulation but decrease the stress of solid insulation which will prevent aging of insulating paper and aid to improve transformer's life. Increased stress on liquid insulation is acceptable as its electric stress withstand capability has also improved with nanoparticles inclusion.

Table 4. Permittivity of different concentration ferrofluids at 90^0C

S.No.	Sample Type	Permittivity	% Decrease
1	Virgin mineral oil (MO)	2.176	--
2	MO+ n (10%)	2.057	5.46%
3	MO + n (20%)	1.034	52.4%
4	MO+ n (30%)	2.055	5.56%
5	MO + n (40%)	2.056	5.55%

For a mixture of m component the dielectric permittivity ϵ^* is connected by a relation, where Log ϵ^* is given by

$$\mathrm{Log}\ \epsilon^* = \sum_{i=1}^{m} y_i\ \mathrm{Log}\ \epsilon_i$$

Where ϵ^* is the dielectric permittivity of the mixture and y is the weight fraction of the component. For a two-component system the relationship can be written as

Log ϵ^*= y_1 Log ϵ_1+ y_2 Log ϵ_2

Where ϵ^* is the dielectric constant of the composite, ϵ_1,y_1 and ϵ_2 ,y_2 are the dielectric constant and weight fractions of the matrix and the filler component respectively. Applying this relation,

For 10% concentration, ϵ_1=2.2, y_1=0.1, ϵ_2= 15, y_1=0.9, Log ϵ^*=1.56.

For 20% concentration, ϵ_1=2.2, y_1=0.2, ϵ_2= 15, y_1=0.8, Log ϵ^*= 1.009.

However in highly concentrated ferrofluid system, there is a large deviation from the expected behaviour because of the strong dipole-dipole interaction.

4. Conclusion

In this study, experimental studies on magnetite (Fe_3O_4) nanoparticles based nanofluid has been synthesized and characterized for use in oil filled transformers. Different concentrations of ferrofluid have been investigated to report optimal concentration in terms of maximum BDV improvement. It is found that 20% concentration reported a maximum BDV improvement of 34 %. It is also observed that resistivity is in accordance with BDV and reported a maximum improvement of 501.44% value at 20% concentration. Dielectric dissipation factor of ferrofluids is noted to be more than mineral oil but it is found that with an increase in concentration dielectric dissipation factor is decreasing. Permittivity value of various ferrofluids is less than that of mineral oil and maximum decrement of 52.4% for 20% concentration ferrofluid is noticed. Hence, it is inferred that 20% concentration of magnetite is an optimal concentration for use in oil filled transformer. Long term aging analysis is to be carried out to further recommend this fluid to the industry.

5. References

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