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Synthesis of ZrO₂ based Nanofluids for cooling and Insulation of Transformers

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ABSTRACT

Nanofluids have recently emerged as an important new technology for heat transfer in various engineering applications. In this work, zirconia nanoparticles (ZrO_2) were synthesized using a microwave-assisted sol-gel method for the preparation of nanofluids through a two-step synthesis process. The electrical, thermal and rheological properties of the nanofluids produced were studied as a function of the ZrO_2 concentration (g/L). These fluids showed potential as an alternative to the traditionally available ester-based transformer oil. The effect of the ZrO_2 nanoparticle additions on the nanofluid was evaluated by measurements of thermal conductivity, breakdown voltage, and viscosity. The results show an improvement in cooling, insulation, and breakdown voltage due to ZrO_2 , which is explained in terms of a nanoparticle-assisted charge capture mechanism. The optimal performance of the nanofluid was obtained for a concentration of 0.2 g/L of ZrO_2 nanoparticles in the transformer oil.

Index Terms — Transformer oil, Nanofluid, Zirconia, Thermal conductivity, Viscosity.

1 INTRODUCTION

TRANSFORMERS are a static electrical machine that converts the alternating signal from one voltage to another voltage (step up and step down). Transformer oil is an integral part of the transformer that provides cooling and insulation enabling better performance. Important properties include high dielectric strength, thermal conductivity, chemical stability and high temperature stability (140°C) [1-3]. In many cases, the failure of the transformer operation is recorded due to the failure of insulating oil [4]. A range of mineral oils have been used previously for use in transformers, but they have serious disadvantages such as their low dielectric values, sensitivity towards moisture and require proper disposal at the end of their useful life to avoid environmental contamination. In the early 1930s, the use of a new type of oil synthesized from "halogenated hydrocarbons" became widespread in place of mineral oil, but it has since been banned due to its dangerous nature and the dangers regarding handling issues [5]. Synthetic oils (Polychlorinated Biphenyls), ester-based oils, siliconebased oils, vegetable oils, and mixed type oils were also used for the proper operation of transformers [6-9]. In addition, intensive research is underway to develop a new type of transfer

oil that can provide better dielectric strength, cooling, and high operating temperature.

Nanofluids are an emerging field of research and prepared by colloidal suspension of nanoparticles in a liquid medium (base fluid) [10]. Due to the presence of nanoparticles in a base fluid, its thermal, dielectric and rheological properties are significantly improved. Nanofluid-based transformer oil is also an interesting research area to replace conventional oil that can cause transformer failure due to its lower efficiency at high temperatures, cooling, and dielectric characteristics. Segal et al. have reported that the addition of magnetic nanoparticles in a conventional transformer oil will enhance its breakdown strength along with the thermal properties [11]. The effect of different nanoparticles such as TiO₂, ZrO₂, Fe₃O₄, ZrO₂, and Al₂O₃ at different concentrations have been studied for tuning the electrical and thermal properties of the transformer oil [12-17]. The addition of nanoparticles results in the improvement of the breakdown strength of the transformer oil. But, the size and morphology of the nanoparticles, the role of dispersant, the influence of temperature, presence of moisture, poor synthesis and characterization techniques and lack of understanding of theoretical concepts are still a challenge in the application of nanofluid-based transformer oil [18-20].

The preparation of nanoparticles with smaller particle sizes with uniform size distribution is the first step for the preparation of a nanofluid. There are several techniques available to synthesize nanoparticles such as coprecipitation, hydrothermal method, wet chemical synthesis and sol-gel synthesis [18, 21]. Among these chemical routes, sol-gel is commonly used for the synthesis of oxide nanoparticles with smaller size, uniform particle size distribution, and better control of stoichiometry.

Nanofluids can be prepared by directly mixing a nanoparticle in the base fluid. But, the type and nature of the base fluid, surfactant, chemical and thermal stability of the nanoparticle, agglomeration of the nanoparticles, chemical complexity on using a stabilizer, and surfactant is also a critical issue for the synthesis of nanofluids. Das et al. suggested a "Single-step method" for nanofluid synthesis by directly mixing the nanoparticles in a base fluid [22]. But, enhanced agglomeration and the chance of settling down of the nanoparticles make it a less preferable method [23, 24]. All the major drawbacks of one step or single-step process are overcome by using a 'Two-step process'. In this method, a surfactant is mixed with the base fluid and then nanoparticles are dispersed in it. The role of surfactant is to stabilize the nanoparticles and keep them suspended in the base fluid to prevent settling. The surfactant increases the particle-particle interaction resulting in the stable dispersion and longer lifespan of nanofluids [24].

In this work, the ZrO_2 nanoparticles based nanofluid was synthesized by using a two-step method for application as transformer oil. The dispersion, electrical, thermal, and rheological properties of the nanofluid were studied for different concentrations of ZrO_2 nanoparticles (0.1-0.5 g/L) and compared with the base fluid. The breakdown strength and failure mechanism are explained using the Weibull probability distribution method.

2 EXPERIMENTAL

2.1 SYNTHESIS OF ZrO₂ NANOPARTICLES

Zirconia nanoparticles were synthesized using the microwave-assisted sol-gel method with zirconium oxychloride octahydrate (ZrOCl₂·8H₂O, 9.78g), and citric acid (C₂O₄H₂,7.68g) as a precursor [16]. The precursors were mixed with deionized water for 1 hour maintaining the pH of the solution in the acidic region (pH~2). The solution obtained was then put into a domestic microwave oven (frequency 2.45 GHz and maximum power of 900 W) for two minutes, which resulted in the formation of a solid white powder. The powder obtained was then ground thoroughly and calcined at 450°C for 4 hours to obtain zirconia nanoparticles.

2.2 SYNTHESIS OF NANOFLUID

The nanofluids were synthesized using different concentrations of the ZrO_2 nanoparticles with Transformer oil as a base fluid (supplied by H.P. Lubricants), and CTAB (Cetyltrimethylammonium bromide, $C_{19}H_{42}BrN$) as a dispersant for the nanoparticles. The optimum quantity for mixing the dispersant was chosen as 0.3% of the weight of the

nanoparticles [12]. The nanofluid was synthesized using the two-step method (Fig.1). In the first step, the nanoparticles were mixed in transformer oil using a magnetic stirrer. ZrO_2 particle additions of 0.1g, 0.2g, 0.3g, 0.4g, and 0.5g in 1 liter of transformer oil were used. The mixture was thoroughly stirred for approximately 30 minutes. In the second step, the dispersant was added to the oil and ultrasonicated for 30 minutes, to ensure the particles we uniformly mixed.



Figure.1. Two-step Process used for the Synthesis of ZrO₂ based nanofluids.

2.3 CHARACTERIZATION

The structural analysis of the zirconia nanoparticles was carried out using X-ray diffraction (Bruker D-8 Advanced, Cu- K_{α}), transmission electron microscopy (FEI, Talos F200X G2), and Fourier transform infrared spectroscopy (Shimadzu). Zeta potential measurement (Beckman Coulter Delso Nano C) was performed to obtain information about the role of surfactant on the stability of nanofluids. To understand the electrical, thermal, and rheological properties of the nanofluids breakdown voltage tests (BDV), thermal conductivity, and viscosity measurements were performed. In this study, the AC breakdown voltage test (BDV) was performed using the BDV oil tester kit (BHEL, Bhopal). The test was carried out in a standard electrode system (mushroom-mushroom type electrode configuration). The separation between the electrodes was set at 2.5 mm with a 2 kV/s rate of increase in the applied voltage. The standing time between each successive test was set to be around 15 minutes. The effective thermal conductivity (keff) was measured using a thermal conductivity measurement kit (KCHT-143, KC Engineers Bhopal). The rheological properties were investigated through viscosity measurements using an Orifice Type Viscometer - Redwood Viscometer type I. The apparatus comprises a cylindrical cup with an orifice at the center. A 50 ml sample was taken for each batch and then poured into the cup. The time taken for the sample to empty through the orifice of the cylindrical cup was recorded. Kinematic Viscosity was then calculated using Equation 1:

$$\gamma = at - \left(\frac{b}{t}\right) \tag{1}$$

where, γ , t, a, and b are kinematic viscosity (cSt), time (sec), viscometer constant, and coefficient of kinetic energy, respectively.

3. RESULTS AND DISCUSSION

3.1 STRUCTURAL AND ZETA POTENTIAL ANALYSIS

Fig.2 (a) shows the XRD pattern of the ZrO₂ nanoparticles synthesized using the sol-gel method. The XRD peaks are indexed using the JCPDS files (Joint Committee on Powder Diffraction Standards) for the tetragonal phase (No.79-1771) and monoclinic phase (No. 31-1484) of the ZrO₂. It clearly shows that the ZrO₂ nanoparticles are crystallized into a tetragonal structure with a minor trace of the monoclinic phase. The average crystallite size of the ZrO₂ nanoparticle is calculated as 3.7 nm by using the Debye Scherrer formula. The transmission electron microscope image of the ZrO₂ nanoparticles is shown in Fig.2 (b). The image reveals that very fine ZrO₂ nanoparticles are formed having a particle size in the range of 5-10 nm which is in agreement with the XRD result. The average particle size of the ZrO₂ nanoparticles is calculated as 5.3 nm. The ZrO₂ nanoparticles are separated from each other showing less agglomeration. The smaller particle size is attributed to the microwave-assisted sol-gel method which produces smaller size nanoparticles with a uniform size distribution. Fig.2 (c) shows the FTIR spectrum of ZrO₂ nanoparticles. The absorption peak in the range of 950-1650 cm⁻¹ appears due to the bending vibration modes of the Zr-OH, and 450-650 cm⁻¹ is due to the Zr-O vibrational mode [25]. The broad peak around 3000-3600 cm⁻¹ can be assigned to vibrations due to the -OH bond in water and in the sample. The results indicated the absence of any organic and inorganic impurities.

Nanoparticle agglomeration is a great challenge for the application of nanofluids. A surfactant (CTAB) is used to improve the stabilization of the nanoparticles in order to obtain a better dispersion of the nanoparticles in a base liquid. Fig. 3 (a) shows the measurement of the zeta potential of nanofluids with different concentrations of surfactant, which is an estimate of the surface charge created around the nanoparticles due to the electrostatic repulsive force between them[26]. A higher value of zeta potential indicates more stable nanofluids due to the lower tendency of the nanoparticles to come together. The zeta potential value initially increases with the CTAB concentration and then decreases for a higher concentration. The optimum value was obtained for 0.3% which is used for the analysis of the performance of the nanofluids. Fig.3 (b) and 3 (c) shows the TEM image of the nanofluid with and without surfactant for the comparison. The TEM image shows that the larger agglomeration of the without surfactant.

3.2 BREAKDOWN VOLTAGE (BDV) TEST

The dielectric breakdown strength of the nanofluid was measured by the breakdown voltage test (BDV). Fig.4 shows the Breakdown voltage vs. Concentration level (g/L) for pure oil as well as for the nanofluid with different ZrO_2 nanoparticle additions. For pure oil, the mean BDV value is 16.82 kV, and it increases due to the addition of ZrO_2 nanoparticles. It is noted that the BDV value increases significantly up to 0.2 g/L before it starts decreasing. The highest increment was noted to be 25.62% for the 0.2g/L sample and the corresponding Mean Breakdown voltage was 21.13 kV which reduced to a value of 16.19 kV at 0.5g/L. The value of BDV falls above the 0.2g/L

concentration due to agglomeration and electric field distortion by the larger particles [27, 28].



Figure.2 (a) XRD pattern of ZrO_2 nanoparticles including JCPDS files for the tetragonal (middle) and monoclinic (bottom) forms, (b) Transmission electron microscope image, and (c) FTIR spectrum of the ZrO_2 nanoparticles.



Figure.3 (a) Zeta potential measurement of the nanofluids with different surfactant concentration, Transmission electron microscope images of the nanofluid with (b) no surfactant, and (c) 0.3 % surfactant concentration.

The Weibull probability was used for the analysis of survival and failure probabilities of the nanofluids [29]. It is a useful statistical tool to investigate the effects of insulation failure under high electrical stress and sustaining life of liquid insulation [30, 31]. Mathematically, the breakdown probabilities can be calculated using Equation 2 [32]:

$$F(x) = 1 - \exp\left[-(x/\eta)^{\beta}\right]$$
(2)

Where, F(x), x, η , and β represent Breakdown voltage probability, Breakdown voltage, scale parameter, and shape parameter, respectively. Since two parameters are involved,

such a probability model is also known as a 'Two-parameter Weibull plot'. In this study, the experimental data have been fitted to the closest Weibull probability plot and respective breakdown or failure probabilities along with corresponding breakdown voltage values have been depicted. Fig.5 shows the Weibull probability plots for all samples in which the vertical axis shows failure probabilities (%) and the horizontal axis depicts corresponding Breakdown Voltage (kV). A comparison of Breakdown Voltage based on failure probabilities at 1%, 5% and 63.2% has been shown in Table-1. The data indicate that the BDV value for 0.2g/L nanofluid is 55.68%, 39.15% and 20.15% higher than the pure oil sample having the risk of failure at 1%, 5% and 63.2%, respectively. However, incremental increases in BDV occurred up to a nanoparticle loading of 0.2g/L, after which it started to reduce. Hence it can be concluded that 0.2g/L is the optimum concentration for ZrO_2 nanofluids to be used as transformer oil. The comparison for shape and scale parameters for all samples is shown in Table-2.

The enhancement in the breakdown voltage of the transformer oil due to the addition of the ZrO_2 nanoparticles is explained using the 'charge trapping mechanism' proposed by Hwang et al. [28]. The polarized nanoparticle inside the fluid will tend to capture or trap the electron which subsequently reduces the mobility of charge carriers. Hence, the average kinetic energy of the particles is reduced in the fluid that increases breakdown voltage. The charge trapping depends on the relaxation time constant (τ) which is defined as the time required to capture (trap) and relax (release) the charge by the nanoparticle. The relaxation time (τ) can be expressions by Equation 3 [28]:

$$\tau = \frac{2\varepsilon_1 + \varepsilon_2}{2\sigma_1 + \sigma_2} \tag{3}$$

where σ_1 and σ_2 are electrical conductivities of base fluid and nanoparticles, respectively and ε_1 and ε_2 are relative

permittivity's of the base fluid and nanoparticles, respectively. Hence, the relaxation time or time to capture and release charges depend upon relative permittivity and electrical conductivity of both the nanoparticles as well as base fluid. The addition of ZrO_2 nanoparticles increases the relaxation time that results in the enhancement of the dielectric strength of the fluid.

However, the breakdown voltage is a maximum for 0.2 g/L of the ZrO_2 nanoparticles before it decreases at higher concentrations. The decrease in breakdown voltage after a certain concentration of the nanoparticles is explained in terms of agglomeration or formation of clusters of the nanoparticles [27]. As a result, the accumulation of charge over clusters of nanoparticles increases distortion of the electric field between the electrodes. The charge accumulation increases with nanoparticle concentration as a result the breakdown voltage decreasing. Hence, the charge trapping is dominant at lower ZrO_2 nanoparticle concentrations ($\leq 0.2g/L$) while agglomeration and electric field distortion are dominant at higher nanoparticle concentrations.



Figure.4. The breakdown voltage of the oil based nanofluid containing different concentrations of ZrO_2 nanoparticles.



Figure.5. Weibull distribution of the AC breakdown voltage of the transformer oil and nanofluid for a different concentration of ZrO_2 nanoparticles.

Breakdown probabilities	Pure oil	0.1g/L		0.2g/L		0.3g/L		0.4g/L		0.5g/L	
(70)	BDV (kV)	BDV (kV)	Increase (%)								
1	11.60	13.45	15.94	18.06	55.68	12.33	6.29	11.82	1.89	9.97	-14.05
5	13.97	15.59	11.59	19.44	39.15	15.04	7.65	14.47	3.57	12.83	-8.16
63.2	18.01	18.91	4.99	21.64	20.15	19.21	6.66	18.59	3.22	17.62	-2.16

Table-1. AC breakdown voltage of the transformer oil and ZrO2 nanoparticle-based nanofluid at breakdown probabilities of 1, 5 and 63.2%

Table-2. Fitting parameters obtained from the Two Parameter Weibull plot of the AC breakdown voltage of the transformer oil with different ZrO₂ nanoparticle concentrations.

Parameters	Pure Oil	0.1 g/L	0.2 g/L	0.3 g/L	0.4 g/L	0.5 g/L
Shape Parameters (β)	11.37	15.448	27.945	12.132	11.767	9.260
Scale Parameters (η)	17.643	18.702	21.563	18.893	18.236	17.099

3.3 THERMAL CONDUCTIVITY

Thermal conductivity is a measure of the ability of a material to conduct heat. It is used to evaluate the heat transfer capability of the nanofluid and its higher value indicates better performance of the transformer oil. As nanofluids are a mixture of two phases, the effective thermal conductivity (keff) is the combination of thermal conductivities of both nanoparticles and base fluid. Fig.6 shows the variation of the thermal conductivity of the nanofluid with ZrO₂ nanoparticle concentration (% vol) and the results obtained are summarized in Table-3. The value of thermal conductivity for pure oil is found to be about 0.167 W/m.K. The thermal conductivity of the nanofluid increased with ZrO_2 concentration. The increments (%) in thermal conductivities were 7.18%, 16.64%, 27.24%, 40% and 55.5% for samples with ZrO₂ additions of 0.1 g/L, 0.2 g/L, 0.3 g/L, 0.4 g/L and 0.5 g/L respectively. The increment in nanoparticle loading led to an incremental increase in the Brownian motion of the nanoparticles. This gain in random motion increases the average Kinetic energy of the nanoparticles resulting in an incremental increase in thermal conductivity.

The thermal conductivity of nanofluids can be compared using different models and theories that have been previously proposed, provided some constraints are considered. There have been many predictive theories to explain the thermal conductivity of nanofluids [33]. The experimental thermal conductivity measured from the apparatus was compared with theoretical values calculated using the Hamilton and Crosser theory and the Maxwell theory. The theory proposed by Maxwell can be expressed using Equation 4 [34]:

$$\frac{k_{eff}}{k_f} = 1 + \frac{3\alpha(k_p - k_f)}{2k_{f+}k_p - \alpha(k_p - k_f)}$$
(4)

where, k_p = thermal conductivity of nanoparticles, k_f = thermal conductivity of the base fluid, α = volume fraction of particles.

An additional theory proposed by Hamilton and Crosser for a mixture of two phases can be expressed using Equation 5 [35]:

$$\frac{k_{eff}}{k_f} = \frac{k_{p+}(n-1)\,k_f + (n-1)\alpha(k_p - k_f)}{k_{p+}(n-1)\,k_f - \alpha(k_p - k_f)} \tag{5}$$

where, k_p = thermal conductivity of nanoparticles, k_f = thermal conductivity of the base fluid, α = volume fraction of particles, n = empirical shape factor. Mathematically it is given by:

$$n = \frac{3}{\psi} \tag{6}$$

where, ψ is the sphericity which is the ratio of the surface area of a spherical particle and surface area of the actual particle taken in the same volume. The value n can range from 0.5 to 6.0. This theory suggests that the thermal conductivity of nanofluids is dependent on the concentration of nanoparticles in addition to the surface area of nanoparticles. The value of k_p and k_f is taken as 2.7 W/m.K and 0.1461 W/m.K respectively. The empirical shape factor 'n' was calculated to be around 5.3 nm from an average size of particles measured directly from TEM observations.

The other model used to explain the improved thermal conductivity of nanofluid is based on Maxwell's model in combination with the contribution of Brownian motion and the electrical double layer[36]-

$$k_{eff} = k_{Mw} + k_{Br} \left(1 + \frac{k_{EDL}}{k_{Br}} \right) \tag{7}$$

where, k_{Mw} , k_{Br} , and k_{EDL} is the thermal conductivity calculated by Maxwell model due to particle motion, and thermal electrical double layer, respectively. The theoretical model based on the Maxwell model with k_{Br} , and k_{EDL} provide the closest prediction of the experimental data compared to the Maxwell model and Hamilton and Crosser model. The greater consistency with this model may be due to the inclusion of an interparticle interaction and Brownian motion of the particle along with Maxwell model.



Figure.6. Thermal conductivity of the nanofluid as a function of ZrO_2 volume %.

Table-3. Thermal Conductivity of Nanoparticles at different loadings and their comparison with theoretical values calculated using different methods.

S.	ZrO ₂	ZrO ₂	Thermal Conductivity (W/m.K)						
110.	(g/L)	(%vol)	Maxwell model	Hamilton Crosser Model	Maxwell +k _{Br} +k _{EDL}	Exp.			
1.	0	0	0.167	0.16699	0.1672	0.167			
2.	0.1	0.015	0.17335	0.17694	0.1782	0.1798			
3.	0.2	0.03	0.17987	0.18712	0.1913	0.1948			
4.	0.3	0.045	0.18656	0.19753	0.2055	0.2125			
5.	0.4	0.06	0.19342	0.20818	0.2221	0.2338			
6.	0.5	0.075	0.20047	0.21908	0.2418	0.2597			

3.4 VISCOSITY MEASUREMENT

The kinematic viscosity of the nanofluid synthesized with different amounts of ZrO_2 nanoparticles were measured at temperatures from 20 to 60°C. Fig.7 shows the variation of the kinematic viscosity with temperature. The viscosity of the pure sample was found to be around 18.428 cSt at 20°C increasing with ZrO_2 concentration. The highest viscosity was observed for the 0.5g/L sample i.e., 21.121 cSt. The kinematic viscosity for all the samples decreased with increase in temperature which was attributed to the reduction in fluid layer resistance [37]. Very close values were obtained for viscosity which indicated that nanoparticle loading has less impact on viscosity. The increase in kinetic energy of the nanoparticles with temperature reduced the cohesive forces of molecules within the fluid thereby reducing viscosity with increasing temperature [38].

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Figure.7. Variation of kinematic viscosity with temperature of the nanofluid with different ZrO₂ nanoparticles concentrations.

4. CONCLUSIONS

Transformer oil based nanofluids with ZrO₂ nanoparticles (0.1 to 0.5 g/L) has been synthesized by two-step method with CTAB as a surfactant. A microwave assisted sol-gel method was used for the synthesis of ZrO₂ nanoparticles with a size range of 5 to 10 nm. The dispersion behavior, electrical, thermal, electrical and rheological properties of the nanofluids has been studied to characterize the performance of nanofluid. The addition of surfactant results in the stabilization of nanofluid by steric stabilization mechanism. The breakdown voltage of the nanofluid increased when compared to pure oil, and a maximum breakdown voltage of 21.53 kV for 0.2 g/L ZrO₂ nanoparticle concentration was obtained. The increase in breakdown voltage was explained in terms of the charge trapping mechanism provided by the nanoparticles, which is reduced at higher concentrations due to agglomeration and distortion within the electric field. The thermal conductivity of the samples increased from 0.167 W/m.K to 0.259 W/m.K with increasing ZrO₂ nanoparticle concentration. The viscosity of the nanofluid also increases (14.550 cSt to 17.335 cSt at 30°C) with ZrO₂ nanoparticle concentration. Thus, ZrO₂ nanoparticle based nanofluids showed enhanced electrical, thermal, and rheological properties compared to the pure transformer oil. Adoption of nanofluids will provide greater cooling and insulation performance of transformers.

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