A C Breakdown Voltage and Thermal Conductivity Behaviour of Mineral Oil based $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ (BCZT) Nanofluids

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***Abstract*: The Mineral oil based nanofluids are prepared by employing nanoparticles of $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ (BCZT) synthesized via soft chemistry precursor route at various weight percent. The desired phase of $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ is confirmed by x-ray diffraction analysis (XRD) and the particle size by transmission electron microscopy (TEM). The nanofluids are characterised ac breakdown voltage (BDV), loss tangent, dielectric permittivity, resistivity, acidity and also for the effective thermal conductivity using hot disk method as per ISO:22007. The nanofluids are having better thermal stability as revealed by the thermogravimetric analysis (TGA) and base oil with 0.005 wt % of BCZT nanoceramic is showing upto 16% enhancement in the ac breakdown voltage and thermal conductivity by 18 % respectively. The thermal conductivity of the nanofluids are decreased as the temperatures is increased. Nanofluids upto 0.005 wt % of BCZT nanoceramic in base oil does not show any change in the viscosity, flashpoint and acidity values indicating that the nanoceramic not hindering the heat transfer characteristics but improved electric strength and thermal conductivity. These nanofluids are considered as an improved version of mineral oils for insulating purposes.**

1. INTRODUCTION

Mineral oil (transformer oil), a petroleum based bi-product used in power transformers as dielectric liquid till date. This is due to the fact that, mineral oil is low cost, exhibit better dielectric strength, and possess low viscosity. Mineral oil is the by-product obtained from refining crude oil, not having defined chemical structure due to the complex nature of the crude itself. However, it has been broadly classified as paraffin, naphthenic and aromatics based on the carbon composition. Mineral oil is expected to facilitate good heat transfer and also function as good insulating medium. However, the presence of contaminants, and moisture, it undergoes severe ageing and results in deterioration of insulating properties. Other insulating liquids such as Polychlorinated Biphenyls (PCBs), silicone oil, synthetic organic esters and some aromatic hydrocarbons were developed as an alternative to mineral oil, but their use is limited as they are not readily available. Since Polychlorinated Biphenyls (PCBs), are not environmentally friendly, they were banned [1]. Further improvement in the critical parameters and heat transfer characteristics are required to be achieved. To achieve this, more research work is focussed on the development of nanofluids, wherein, small amount of nanoparticles are mixed into mineral oil and improvement in the critical parameters were noticed [2-12]. Researchers have used various types of nanomaterials, such as conducting, magnetic or semiconducting and number of nanofluids were prepared and studied. Mostly, it was targeted to improve the electric strength behaviour of nanofluids. For example, in the recent work done, $CaCu_3Ti_4O_{12}$ (CCTO) ceramic possessing high permittivity was used for the development of nanofluids. The BDV values obtained for synthetic ester nanofluids [10,11] are different from the values obtained for the mineral oil [12] nanofluids, though the nanomaterial used in both the work is same, indicating that the synthetic esters showing better electric strength behaviour. Whereas, $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ (BCZT) ceramic in synthetic ester showed 20 % enhancement in the electric strength value [13]. However, the enhancement in the BDV is less as compared to that of synthetic ester based CCTO nanofluids [12]. Since the particle size of the BCZT is smaller than CCTO, the electric strength behaviour could be related to the particle size. These results indicate that the electric strength of nanofluids are not only depended on the size and type of particles, it is also depended on the type of base oil. The base oil initial BDV value is also important to understand the percent enhancement. For example, barium titanate based nanofluids were prepared using the base oil having 37 kV/2.5mm, and it has exhibited around 38% enhancement. The base oil initial BDV value depends on the type of transformer oil feed stock (TOFS) derived from the crude. Hence, the final enhancement in the electric strength of any nanofluids are depended on the base oil initial BDV value.

Apart from these, researchers had proposed many theories to support the electric strength behaviour of the nanofluids. The formation of conductive structures called streamers [15], electrons traps and potential well formed at the interface between nanoparticle and base oil [16] responsible for enhancing the breakdown performance of the oil. According to the electron trap theory [15], the conductive particles acts as electron scavengers if the time constant of charge relaxation τ_r is shorter than the timescales of streamer growth. The relaxation time is given by :

$$\tau_r = \frac{2\varepsilon_1+\varepsilon_2}{2\sigma_1+\sigma_2} \tag{1}$$

where ε_1, ε_2, and σ_1, σ_2 are permittivities and the conductivities of matrix and nanoparticles, respectively [15].

Sima et.al proposed that, the potential well produced by the surface charge on a semiconductive or dielectric nanoparticle is given by [16]:

$$\varphi = \frac{\varepsilon_2-\varepsilon_1}{2\varepsilon_1+\varepsilon_2} R^3 E_0 \frac{1}{r^2}\cos\theta, r \geq R \tag{2}$$

where E_o is the value of the electric field; R is the nanoparticle radius; r is the distance from the nanoparticle's surface and θ is the angle that is occupied by negative charges on nanoparticle's positive hemisphere.

Since the transformer oil is complex in nature, apart from the electric strength, its thermodynamic properties such as thermal conductivity, and coefficient of thermal expansion are important parameters that needs to be addressed. More importantly, the thermal conductivity highly dependent on the type of carbon compositions such as paraffin, naphthenic and aromatic present in the mineral oil [17]. The addition of nanoparticles apart from improving the electric strength, improvement in thermal conductivity also has been reported [18-27]. The ceramics have high thermal conductivity compared to oils, and hence, addition of such particles in the base fluid give rise to improved thermal conductivity even at very low concentrations. The type of nanomaterials, its size and shape, the concentration level and the basic fluid characteristics will ultimately decide the overall thermal conductivity of the nanofluids. There are reports that, the multiple nanoparticles such as $BaTiO_3$, TiO_2, SiO_2 and Al_2O_3 in transformer oil showed enhanced heat transfer characteristics [18].

The thermal conductivity behaviour of nanofluids were studied by many researchers by employing nano materials such as Al_2O_3, SiO_2 and SiC [19], Silica and Fullerene [20], Carbon Nanotubes and Diamond [21], Functionalized nanodiamond [22], AIN [23], Hexagonal boronnitride (h-BN) [24], Boron Nitride [25], Titanium oxide / exfoliated boron nitride (Eh-BN) [26] and Alumina / Aluminum Nitride [27]. The thermal conductivity behaviour in nanofluids are mainly dependent on the interaction of nanoparticle with liquid at the interface. Formation of nano layer at the solid-liquid interface acts as a thermal bridge between a solid nanoparticle and the base liquid and thereby enhancing the thermal conductivity [28]. The above investigations prompted us to study the thermal conductivity behaviour apart from the other parameters by employing barium calcium zirconium titanate nanoceramic.

In this work, barium calcium zirconium titanate $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ (BCZT), known to be a promising piezo-ceramic possessing a piezoelectric coefficient (d33) in the range of 550–620 pC N was employed in this work for the development of mineral oil nanofluids [13]. The nano fluids are characterised for thermal analysis and thermal conductivity apart from the other critical parameters such as BDV, flash point, tan delta, resistivity, and viscosity.

2. MATERIALS AND METHODS

2.1 Preparation of nanoceramics

The $Ba_{0.85}Ca_{0.15}Zr_{0.1}Ti_{0.9}O_3$ (BCZT) nanoceramics were prepared via oxalate precursor synthesis method as reported in the literature [13,29]. The barium calcium zirconyl titanyl oxalate precursor was isothermally heated at 850°C /5h to obtain the desired monophasic ceramic.

2.2 Preparation of nanofluids.

Mineral oil was procured from local supplier and filtered to remove any solid impurities if present. The BCZT nanoceramics in various concentration by weight percent of 0, 0.001, 0.0025, and 0.005 was added to the mineral oil. The samples were subjected to sonication using probe ultrasonicator, at a frequency of 60 Hz (Make:Sonics Materials, Vibracell-750W). The sonication was done for 30 min to obtain the homogeneous samples with less agglomeration. After that, whole sample is subjected to vacuum degassing at 80ºC for 5hr to remove any moisture that would have trapped during ultrasonication.

Table 1. Samples prepared from Nanofluids

Samples	Description of oil samples
MNF-0	Mineral oil with 0.00 wt % BCZT ceramic
MNF-1	Mineral oil with 0.0010 wt % BCZT ceramic
MNF-2	Mineral oil with 0.0025 wt % BCZT ceramic
MNF-3	Mineral oil with 0.0050 wt % BCZT ceramic

These samples were kept in desiccator and cooled and used for the characterization. The nanofluids prepared are designated as MNF-0, MNF-1, MNF-2 and MNF-3 respectively and details are given in the Table.1. The critical parameters evaluated are given in the Table.2

3. EXPERIMENTAL DETAILS

3.1 X-Ray diffraction (XRD)

The Pananalytical, X-pert Pro XRD equipment was used in the 2θ range of 10-60 degrees for was used to confirm the phas purity of the ceramic.

3.2 Transmission Electron Microscopy (TEM)

Transmission (JEOL JEM-2001F) electron microscope used for finding out morphological features and grain size determination of BCZT ceramic.

3.3 Thermogravimetric Analysis (TGA)

Thermo gravimetric analyses (Model:TGA:Q500, TA Instruments) were carried out in nitrogen atmosphere with flow rate of 60 ml/min, and 10ºC/min heating rate.

3.4 AC Breakdown Voltage (BDV)

Electric strength measurements were conducted using breakdown voltage tester (make: b2 electronics; model: BA100). The tests were conducted in duplicate. Mean value of 12 individual tests along with standard deviation were taken as BDV value in terms of kV/2.5 mm.

3.5 Dissipation factor (loss tangent) and Volume Resistivity

Both dielectric dissipation factor (DDF) and volume resistivity was measured using an automated bridge at 50 Hz, in the three-electrode test cell as per the ASTM D 924 and ASTM D 1169 respectively [12]. Dissipation factor reveals presence of contaminants in oil derived due to poorly manufactured oxidation products and water soluble.

Table 2. Parameters of nanofluids developed

Samples	AC BDV [kV/2.5mm]	Moisture Content, ppm	Loss Tangent (tan δ) at 90°C	Dielectric Constant 90ºC.	Resistivity @90ºC	Viscosity [c-St] @40ºC	@90ºC	Flash Point [°C]	Acidity [mg KOH/g]
MNF-0	58.78	7.6	0.000367	2.171	7.21 E13	9.72	3.84	152	0.00688
MNF-1	65.8	7.3	0.000438	2.159	8.00 E13	9.67	4.23	152	0.00710
MNF-2	67.99	7.1	0.000558	2.162	8.90 E13	9.62	4.19	153	0.00695
MNF-3	70.35	6.6	0.000752	2.166	1.19 E14	9.69	4.21	154	0.00698

3.6 Viscosity

Calibrated U-tube glass capillary viscometer was used for measuring Viscosity of the nanofluids at different temperature like 27, 45 and 90°C. The flow time of the liquid under gravity is noted and the viscosity was calculated from the product of flow time and viscometer constant.

3.7. Flash Point

Flash point tests was carried out using an Pensky-Martens closed cup apparatus, which indicates the qualitative indication of flammable materials. Helps in assessing the flammability hazard of a material.

3.8. Acid Number

The measure of organic or inorganic acidity is expressed in milligrams of potassium hydroxide required to neutralize total acidity in one gram of the sample (mg of KOH/gm oil oil). Acidic by-products produce would increase dielectric loss, low specific resistance and increased corrosion.

3.9. Interfacial Tension (IFT)

Interfacial tension (IFT) was measured using Kruss K9 digital interfacial tensiometer. Interfacial tension is the molecular attractive force between unlike molecules at the interface of water and oil sample. Interfacial tension is determined by measuring the force necessary to detach a planar ring of platinum wire from the surface of the liquid of higher surface tension, that is, upward from the water-oil interface.

4.0 Thermal Conductivity

The thermal conductivity by transient Plane Source (TPS) method was measured using Hot Disk, (Sweden, Model: TPS 2200) as per ISO:22007. For the solid samples, the diameter of the sample should not be less than twice that of sensor and thickness of the sample should not be less than the radius of the sensor. The liquid samples are contained in a small cell, maintaining a short distance between sensor and sample boundary to avoid convection during measurements. The sensor size is maximum 3.2 mm in radius, the aluminium cell fixes the sensor in a vertical position and has a small cavity with a narrow inlet and outlet for the liquid sample. This design stabilises temperature, reduce convection and prevent evaporation.

4. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction Studies

The Fig.1 shows the X-ray diffraction pattern recorded in the 2θ range between 10-60 degrees. The oxalate precursor that was prepared was heat treated at various temperature to obtain the phase pure ceramic. As reported [25], the phase pure could be obtained when the precursor is heated above 750ºC and hence, the powder was calcined at 850ºC. The powder heat treated at 850ºC (Fig.1a) has confirmed the single phase nature of the BCZT and also matches with the reported results and ICSD-data 187673 (Fig.1b) [29].

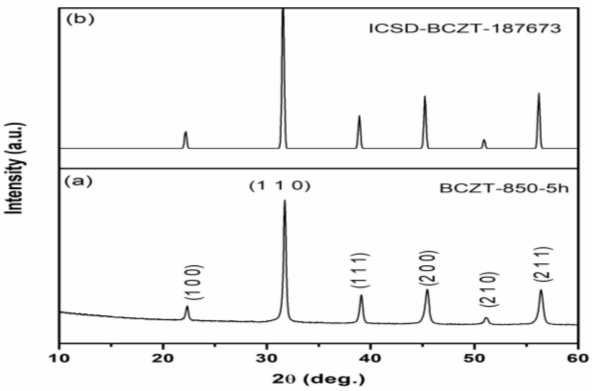

Figure.1. XRD of (a) BCZT nanoceramics after calcination at 850ºC, (b) BCZT ICSD-data file no.187673.

4.2 Microstructure by TEM

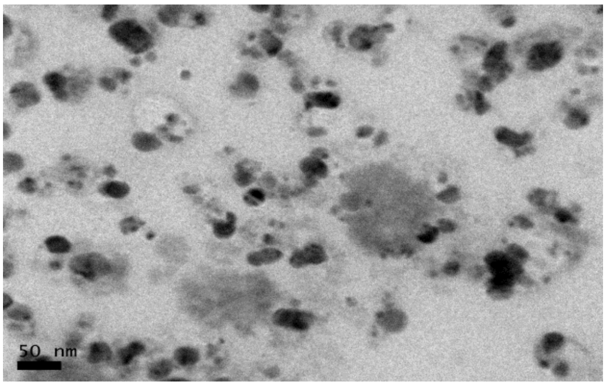

Figure.2. Bright field TEM images of phase-pure BCZT obtained at 850ºC showing the particle dimensions ranging from 10 to 50nm.

Figure.2 represent the bright field TEM image of the phase pure BCZT nanocrystalline powder obtained from the thermal decomposition of the oxalate precursor at 850°C/5 h. The particles are less agglomerated and the crystallite size are in the range of 10-50 nm.

4.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) carried out on the BCZT ceramic as well as on the nanofluids are given in the figure. 3. The BCZT ceramic was already heat treated upto 850^{o}C and the thermal analysis (straight line) showed that the ceramic is stable and not containing any volatile or unreacted components. Interestingly, the nanofluids with 0.005 weight percent of BCZT showed improved stability. The inset of the Figure.3 indicating the difference clearly.

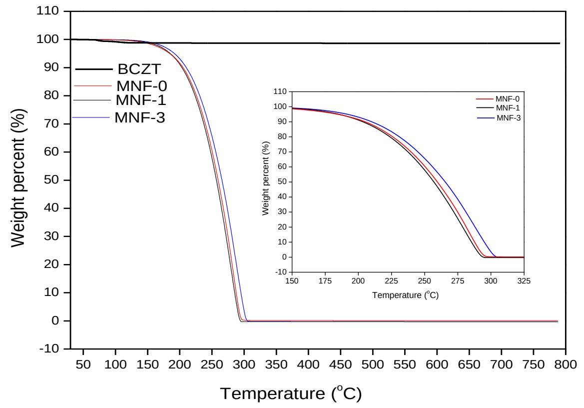

Figure.3. Thermogravimetric Analyses of BCZT ceramics and the nanofluids

4.3 Breakdown Voltage

AC breakdown voltage measured on BCZT nanofluids are shown in the figure.4. While preparing nanofluids under ultrasonication, it was observed that samples picks up moisture. Hence, these samples one again subjected to vacuum heating at 100^{o}C for 8 hr and then cooled in the desiccator. The moisture content measured on these samples are less than 10 ppm. It was observed that, as the concentration of nanomaterials increased in the base oil, the BDV values also increased. The base oil has 58.78 kV/mm BDV values and it has increase to 70.35 kV/mm for the 0.0050 wt % BCZT content in the base oil. Around 19.7% enhancement in electric strength was observed. In the previous work done on the synthetic ester based BCZT nanofluids, it has shown improvement upto 20% [13]. Similarly, CCTO nanofluids developed using synthetic esters [11] showed around 41 % enhancement as against 21% for mineral oil based nanofluids [12]. For the barium titanate based nanofluids [14], around 30 % enhancement was observed for the base oil having electric strength value of 37 kV/2.5 mm. These results indicate that, synthetic ester based nanofluids are showing better electric strength behaviour than the mineral oil. This is because synthetic esters has better moisture tolerance and hence they exhibit improved electric strength behaviour.

The dielectric TiO_2 based nanofluids showed 29% enhancement, whereas ferromagnetic Fe_3O_4 based nanofluids showed 16% enhancement in the breakdown voltage. Also, ZnO based nanofluids exhibited electric strength as high as 45%. [7]. In this work, BCZT ceramic which is ferroelectric and also shows piezoelectric effect has been used. The BCZT nano ceramic has smaller particle compared to CCTO nanoceramic. Still, the difference in the BDV values obtained for the BCZT nanofluids in this work exhibiting similar trend to that of mineral oil based CCTO nanofluids 12]. Though many theories proposed for the electric strength behaviour in nanofluids, it is evident that the BDV is not only depends on the size of the particles, and the type of base oil used, but also related to the permittivity of the ceramics [16]. The BCZT nanofluids have exhibited improved electric strength behaviour.

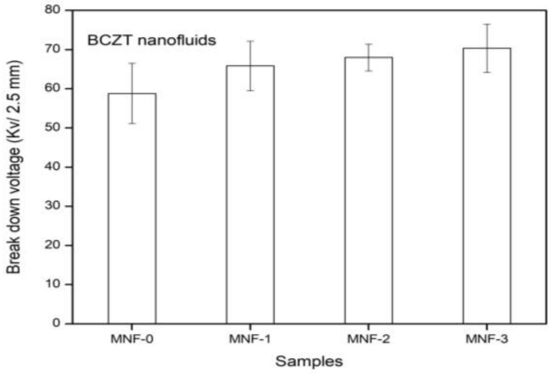

Figure.4. Electric Strength behaviour of nanofluids as function of wt percent of BCZT nanoparticles..

4.4 Thermal Conductivity

The thermal conductivity by Transient Plane Source (TPS) method is considered to be most precise and convenient technique for studying thermal transport properties. It is an absolute technique, yielding information on thermal conductivity, thermal diffusivity as well as specific heat of the material under study, in accordance with ISO 22007-2.

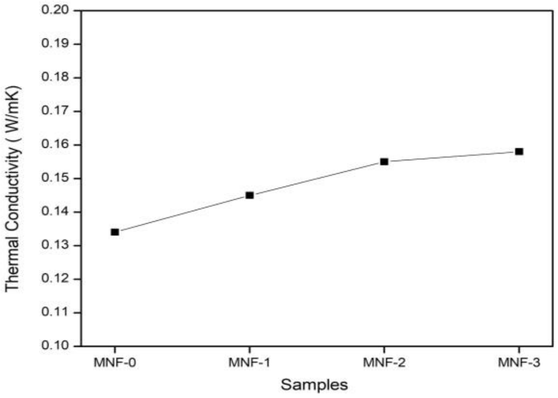

Figure.5. Room temperature thermal conductivity of BCZT based nanofluids.

Figure.5 shows the thermal conductivity measured at room temperature for the nanofluids. H J. Wang et.al [17] had clearly investigated that thermal conductivity is directly related to the carbon content of the mineral oil. The thermal conductivity values varies between 0.1031 W/mK and 0.1086 W/mK depending on the type of base. In this work, the base oil used is of naphthenic type with less than 5 % of aromatic content. Thermal conductivity measured at room temperature for base oil used is around 0.134 W/mK. There is gradual increase in the thermal conductivity as the weight percent of BCZT increased in the base oil. The nanoceramics upto 0.005 wt % has exhibited improved thermal conductivity and the value obtained is around 0.158 W/mK, which is 18% enhancement compared to the base oil. Chiesa et al have reported improved thermal conductivity behaviour for mineral oil based Al_2O_3, SiO_2 and SiC nanofluids measured by transient hot wire method [19]. There are some nanofluids prepared using silica and fullerene in mineral oil exhibited negligible effect [20]. Improvement in the thermal conductivity as high as 27% has been achieved for carbon nanotube based nanofluids [21]. Newtonian nanofluids using functionalized nanodiamonds in naphthenic transformer oil exhibited around 14.5% enhancement [22]. AlN nanofluids showed only 3-7% improvement in the thermal conductivity [23]. Thermal conductivity around 76% enhancement has been reported in the case of h-boron nitride based nanofluids [24]. Whereas, TiO_2 nanofluids showed only 5.4 % improvement [23]. Al_2O_3/AlN–mineral oil nanofluids showed 18% improvement for the 1% volume fraction of Al_2O_3 and increased by 7% with a 0.5% volume fraction of AlN [27]. Thermal conductivity as high as 18% with very low level of nanoparticles addition is achieved in this work, which is very significant. It is also to be noted that with the 0.005 wt % addition of BCZT nanoparticle in mineral oil, the value obtained is much higher as compared to the reported value [14]. The thermal conductivity of the oils are less compared to metals and metal oxides. Hence, the effective thermal conductivity of the nanofluids are related to the thermal conductivity and particle size of the ceramics and more importantly, particles-liquid interface influencing the thermal conductivity [28].

4.5 Thermal Conductivity as a function of temperature

Figure.6 gives the thermal conductivity measured as a function of temperature for the nanofluids. Thermal conductivity decreased as the temperature increased. However, the decrease is not very drastic due to the high molecular mass hindered by the molecular motion. The mineral oil is complex in nature with different components and mass fractions of different components in mineral oil has great influence on the thermal conductivity [16].

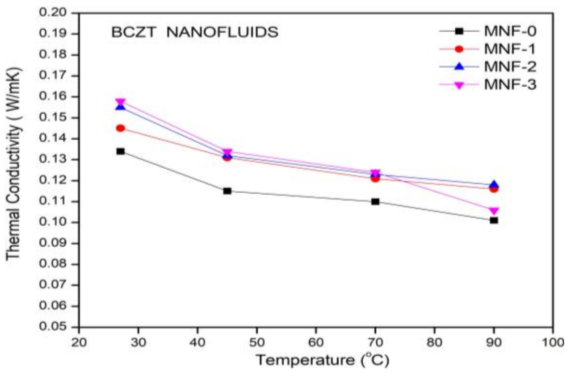

Figure.6. Thermal conductivity as a function of temperature for BCZT nanofluids.

4.6 Loss tangent, Dielectric Constant and Resistivity

The dielectric dissipation factor (tan delta) and dielectric constant measured at 90°C is shown in the Figure 7. As the BCZT ceramic concentration increased in the base oil, the tandelta increased. The base oil tan delta value is 0.000367 and increased to 0.000752 for the nanofluids with 0.005 weight percent. The dielectric constant is showing slight variation but it is insignificant.

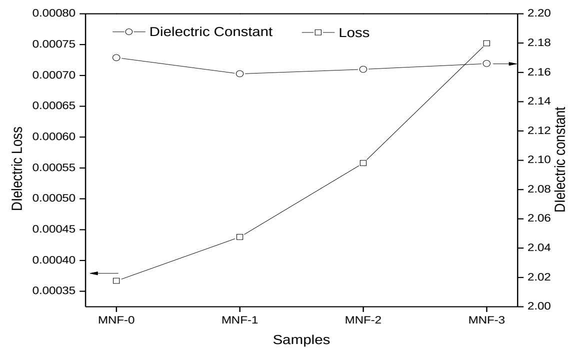

Figure.7. Loss tangent, Dielectric Constant for BCZT nanofluids.

The resistivity measured at 90°C is given in the Figure.8. There is a linear increase in the resistivity values for the nanofluids. The base oil resistivity is 7.21XE13, which increased to 1.19XE14 for the nanofluid with 0.005 weight percent BCZT. The increased specific resistance of the nanofluids means that improvement in insulating property, which could be attributed to the nanoparticles of BCZT. Around 65 % enhancement in the resistivity value is obtained as against 46.6 % enhancement in the case of CCTO nanofluids reported [12]. However, the BCZT in synthetic ester showed decreasing trend [13].

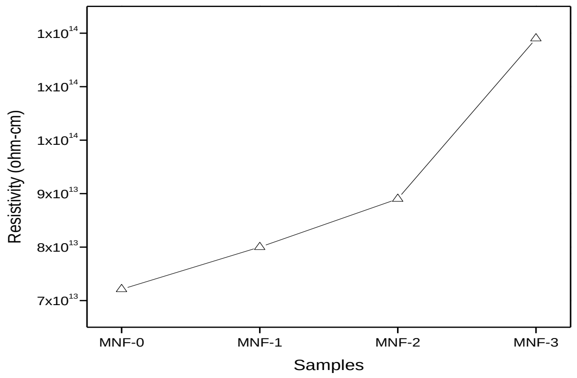

Figure.8. Resistivity measured at 90°C for BCZT nanofluids.

4.7 Viscosity

Viscosity is the product of flow time and the viscometer constant, evaluated as per ASTM D 445. Figure .9 shows the viscosity measured at 40°C and 70°C respectively. For the fixed viscometer constant, only the flow time varies depending on the fluid viscosity. The base oil viscosity around 9.27 c-St at 40°C, is almost maintained constant even after adding nanoceramics upto 0.005 wt percent. It is clear that, upto 0.005 wt percent of BCZT, the time of flow of the fluid under gravity is minimum, does not show any change in the value, implying nanoparticles is not hindering the flow property. If the particles are soluble in the fluid forming a solid solution, there is chances that the viscosity of the final fluid would show some variation. Since the particles are suspended in the liquid, and not affecting the time of flow of the liquid under gravity and hence no change in the viscosity was observed.

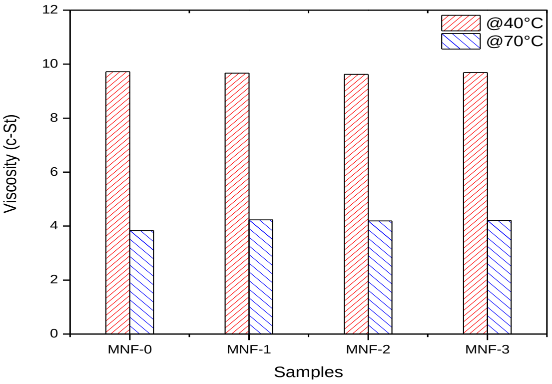

Figure.9.Viscosity as a function of temperature for BCZT nanofluids.

As expected, the viscosity of the nanofluids decreased as the temperature increased. The base oil having the viscosity of 9.27 c-St at 40°C, had decreased to 4.2 c-St @ 70°C. However, increasing concentration of nanomaterial up to 0.005 wt % in base oil, nanofluids viscosity has not changed, indicating that the BCZT nanomaterials not hindering the heat transfer characteristics of the base oil.

4.8 Flash point

Figure.10 showing the flash point characteristics of the nanofluids. Flash point is the qualitative indication of contamination with more flammable materials. The flash point of any material is the lowest temperature at which a liquid in a specified apparatus will give off sufficient vapour to ignite momentarily on application of a flame. The flash point of the base oil is 152°C and the addition of nanoceramic in to the base oil, no appreciable change oserved, indicating that the ceramic is not releasing any flammable substances other than mineral oil, and also not participating in arresting / resisting the release of flammable substances. In the case of CCTO nanofluids [11,12], improvement in the flash point of the nanofluids was reported. In the case of BCZT nanofluids, no improvement observed, however, no adverse effect on the flame resistant characteristics.

4.9 Acidity

The acidity expressed as number of milligrams of potassium hydroxide required to neutralize total acidity in one gram of the sample. The presence of acidity would increase dielectric loss, lower the specific resistance and also increases corrosion. Hence, addition of nanoceramics in to the base oil should not increase the acidity. The base oil acidity was around 0.000678 mg KOH/ gm, had increased to 0.000698 mg KOH/ gm when BCZT concentration increased to 0.005 the weight percent in base oil. The acidity of the nanofluids shows only marginal increase, which is negligible and not significant.

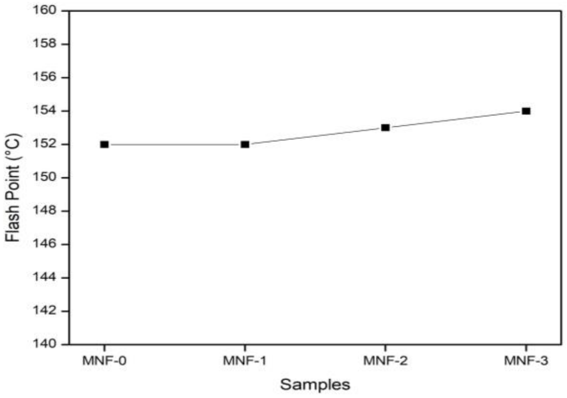

Figure.10. Flash point characteristics of BCZT nanofluids.

5. CONCLUSION

The mineral based nanofluids were successfully prepared by employing the BCZT nanoparticles under ultrasonication. Both electric strength and thermal conductivity had increased as the weight percent of nanoparticles increased in the mineral oil. The base oil viscosity, flash point and acidity remains stable upto 0.005 weight percent addition of nanoparticle.

6. ACKNOWLEDGMENTS

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