

# Investigation of Dielectric and Thermal Properties of Nano-dielectric Materials in Electrical Applications

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**Abstract**—Insulating materials plays a vital role in the design and performance of electrical power systems for both steady state and transient state conditions. The last decade has witnessed significant developments in the area of nano-dielectric materials and significant effects of nano-scale fillers on electric, thermal and mechanical properties of polymeric materials have been observed. The dielectric and thermal properties are more important for the insulating materials used in high voltage applications. In this paper, the investigation of electrical and thermal properties of polymeric-epoxy based zirconia nano-composite evaluated and determined their ability as insulating materials for heavy electrical applications. The various electrical properties dielectric loss ( $\tan \delta$ ), dielectric constant ( $\epsilon$ ), dielectric strength and partial discharge voltage were investigated. In this study, zirconia has been tested as filler. Experimental results showed that the zirconia nano-composites improved the dielectric and thermal properties compare with the standard polymeric materials, and concluded that, the zirconia nano-composites has the ability to use insulating materials for heavy electrical applications.

**Index Terms**—Dielectrics, Dielectric Strength, Nano-Fillers, Nano-Composites, Partial Discharge, SEM Analysis, TGA, Zirconia.

## I. INTRODUCTION

Nano-composites offer exciting improvements for dielectric and insulating materials and practical application has been a primary concern in recent research [1]. Many studies shown that, the addition of few weight percentages of nano-fillers improves the dielectric and insulation properties of nano-composites. The developments of new and advanced materials to be used in power systems require extensive studies on electrical insulation characteristics of these materials before they can be used in commercial systems [14]. Surface flashover across the solid insulating materials plays another role in power systems. The nano-composites improve the surface flashovers [4]. A primary concern in recent nano-composite research is application. Solid insulation systems using nano-composite materials are an effective approach to reducing the use of SF<sub>6</sub> gas [8]. The nano-filler dispersion is effective in improving insulation properties of epoxy-based insulating materials, also the nano-filler combination systems not only verify the nano-composite materials but also provide synergy effects on improvement of insulation properties [8]. The addition of nano-fillers to the conventional filled epoxy will give epoxy casting both low thermal expansion and excellent

electrical insulation properties. Also the nano-composites have excellent insulation breakdown time in comparison with the conventional filled epoxy [5]. Nano-particle doped composites show a positive effect on the long-term failure properties such as ageing resistance and partial discharge (PD) properties of nano-composites are superior to the standard polymer materials [6]. Nano-scale filled varnish have shown better behaviour for partial discharge inception voltage (PDIV) and lifetime have been increased and bonding strength has not deteriorated [8]. A lot of research has been carried out recently incorporating various nano-particles into existing dielectric systems in a cost effective manner, resulting in nano-composites with improved benefits over conventional filler systems [5]. The improvements can be a combination of electrical, mechanical and thermal enhancements [7], [11]. Insulation integrity is of great importance for all electrical power applications including energy conversion, power delivery, and energy storage and power consumption. Nano-dielectrics can enhance the reliability of current systems and more importantly can improve by enabling innovative design and the utilization of renewable energy resources [5]. The often very high surface area to volume ratio of nano-particles provides a tremendous driving force for diffusion, especially at elevated temperatures. The nano-composites have one of the special features; the containment of a uniformly dispersed assembly of strongly interacting particles in suspension requires total control over particle-particle interactions. During the switching operation the system voltage would be increases for this period the insulation is important to withstand the induced stress to operate the system at steady state condition. So in order to provide the proper insulation the nano-scale fillers plays a significant role. The high resolution of thermally cured composites showed that the nano dispersed zirconia particles were uniformly distributed within matrix [1]. As a result, the recent improvement has been achieved in many technologies by using nano-fillers.

## II. EXPERIMENTAL

### A. Synthesis of Nano-materials

Bulk materials are broken into nano sized particle (for processing solid-state materials) by using Ball milling method. Small hard balls are allowed to rotate inside a container and then it is made to fall on a solid with high force to crush the solid into nano crystal is shown in Fig. 1. The hardened steel or tungsten carbide balls are put in

a container along with powder of particles (50mm) of a desired material. The container is closed with tight lids. When the container is rotating around the central axis, the material is forced to press against the walls. The milling balls impart energy on collision and produce smaller grain size of nano particle. Nano-filler were dispersed in the standard polymer (polyamide).

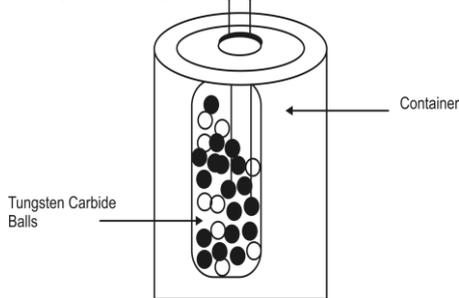


Fig. 1 Ball Milling Process of nano-composites

**B. Sample Preparation**

The nano-composites were prepared by radical initiator curing method. In this method, the proportionality of 80% of enamel and 20% of epoxy resin was taken, then adding a curing agent act as DDM (Diamino Diphenyl Methane) in proportion to epoxy resin (For 1g of resin 0.27g of DDM). To melt the DDM for 10 minutes at 60-80°C using magnetic mantle equipment. To mix the both solution ( melted DDM (hardener), enamel and resin), then the mixture was pouring in to the mold (die), and it has been placed in an oven at a temperature of 80°C for one hour or based on the curing time. After the time period, the mixture is pouring in to the mold which was coated by a Teflon sheet. For proper epoxy curing the die has heated at 80°C for 2 hours and 120° C for three hours. After the specific time period cool the mold for one hour and get the solid nano-composite sample. Four series of specimens were produced, each one with different filler content, starting from 0% (pure polymer), 1, 3 and 5% weight. The process involved for preparation of nano-composites is revealed in Fig. 2. The nano-composite specimens were produced by a curing reaction with a hardener.

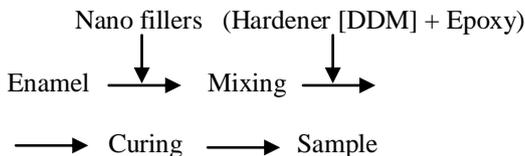


Fig. 2 Preparation of nanocomposites

**C. Analysis of Nano-scale Structure**

The nano particles were subjected to scanning electron microscopy (SEM) to study and analyze the surface and structure of the nano particles. SEM is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the

sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. All samples must also be of an appropriate size (extends from less than 100 nm to around 5 μm) to fit in the specimen chamber and are generally mounted rigidly on a specimen holder. This technique is used to determine the size of the samples.

**D. Dielectric Spectroscopy Analysis**

The inductance, capacitance and impedance of the nano-composites have been measured by using impedance spectroscopy instrument. It also measures the dielectric properties of a medium as a function of frequency. It is based on the interaction of an external field with the electric dipole moment of the sample, often expressed by permittivity. The prepared nano-composite samples were sliced, whose area is 0.5cm × 0.5cm then it is placed between the electrodes of dielectric spectroscopy at different temperatures (60°C, 155°C and 300°C) for the frequency range of 0–500 MHz. From these properties, the permittivity and quality factor of the samples were found.

The measurement of permittivity is calculated by using contacting electrode method. This method derives permittivity by measuring the capacitance of the electrodes contacting the specimen directly. Permittivity and loss tangent are calculated using the equations below: [15].

$$\epsilon_r = \frac{t_m \times C_p}{A \times \epsilon_0} = \frac{t_m \times C_p}{\pi \left(\frac{d}{2}\right)^2 \times \epsilon_0}$$

$$\tan \delta = D$$

Where  $C_p$ : Equivalent parallel capacitance of specimen [F],  $t_m$ : Average thickness of specimen [m], A: Guarded electrode's surface area [m<sup>2</sup>], d: Guarded electrode's diameter[m],  $\epsilon_0$ : Permittivity of free space.

**E. Partial Discharge Measurements**

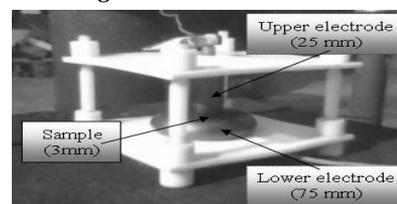


Fig. 3 Electrode Setup for BD and PD Measurement

The partial discharge experiment was carried out inside the shielded room because to avoid the external noises. Because the noise are produces the discharges. A standardized testing arrangement with electrode setup for the determination of the breakdown voltage test (BD test) and partial discharge measurement (PD test) of film or foils as per standard (IEC 60243 – 1) is as shown in Fig.

3. The nano-composites were placed between the electrodes and the electrode setup was kept inside the oil. To applying the voltages gradually and capturing the initial discharges occur in the composites by using high quality oscilloscope. Also the inception and extinction voltages were noted. The same process would be repeated four to five times and calculate the average value. The experimental set up used for partial discharge measurement is shown in Fig. 4.



Fig. 4 Set Up For Partial Discharge Measurement

The partial discharge inception and extinction voltages for uniform field and non uniform field configurations were conducted.

#### F. Dielectric Strength Measurements

The dielectric strength i.e. breakdown strength test was conducted with alternating voltage, which should be gradually increased from zero to the breakdown value. The voltage would be applied to the sample by using high voltage generator with high voltage transformer and note down were the breakdown was occurred. For the breakdown test, at particular composition 10 to 15 samples were taken and calculate the average value for the dielectric strength. The arrangement of electrode setup for dielectric strength measurements is revealed in Fig. 5. The sample thickness was 3mm and the size of the electrode configuration is shown in Fig 3. The entire arrangement is immersed in an insulating liquid with higher dielectric constant (Ex: Insulating oil).

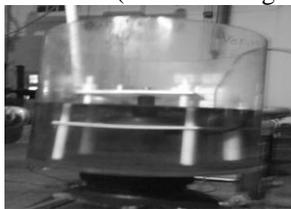


Fig. 5 Arrangement for Dielectric Strength Measurement

#### G. Thermo Gravimetric Analysis (TGA)

Thermo gravimetric analysis is a simple analytical technique that measures the weight loss (or weight gain) of a material as a function of temperature. This test is used to analyse the thermal properties of the nano-composite samples. As materials are heated, they can lose weight from a simple process such as drying, or from chemical reactions that liberate gases. Some materials can gain weight by reacting with the atmosphere in the testing environment. The TGA results had been obtained from

diamond TG/DTA 6000 instrument system. The sampling size can be analyzed from 0.1mg to 10g and the heating rate of 0.1-50°C/min for the temperature range from 50°C to 900°C and it maintain consistent heating rate and gas flow. The instrument measures sampling purity, reaction rate, identification, activation energy and heat of reactions.

### III. RESULTS

#### A. Analysis of Nano-filler Structure

From SEM image results, the particles are in the form of nano metric range and the sizes of the particles were found in the range from 50 to 120 nm size and varying at different areas. The analysed result is shown in Fig. 6.

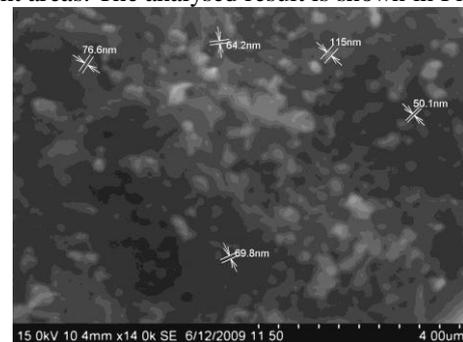


Fig. 6 SEM Analysis of Zirconia

#### B. Dielectric Spectroscopy Analysis

The dielectric characteristics of the sample 0 (pure), 1, 3 and 5wt% were analyzed by dielectric spectroscopy instruments from 0 to 5MHz range. For every sample was analyzed by three different temperatures are at 60°C, 155°C and 300°C.

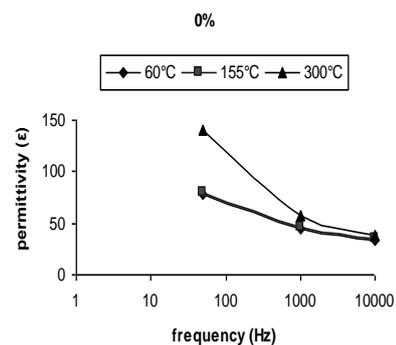
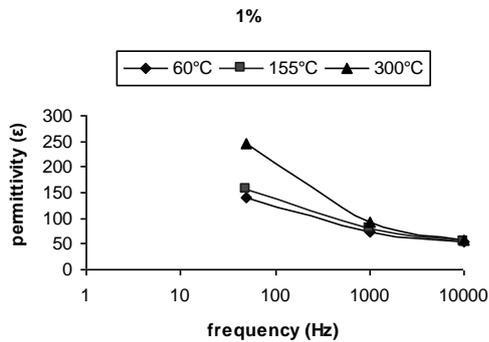


Fig. 7 Permittivity versus Frequency at Various Temperatures of Standard Polymer

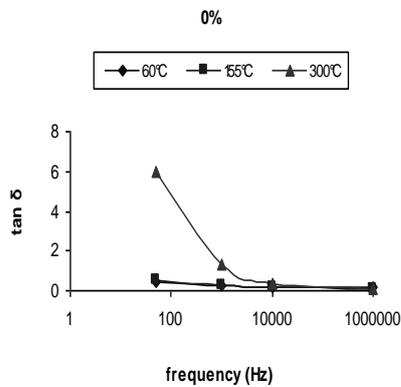
From the Fig.7 results the sample temperature increases with increase its permittivity. Similarly when the frequency was increases the permittivity of the sample was decreases. Because at lower frequencies the permittivity is higher because of settling time is higher in polarization, so the permittivity is higher value. At higher frequencies settling time is lower and the polarization fails to settle itself, so the permittivity is lower value. At

10 KHz above frequency range there is minute variation in permittivity as on study.

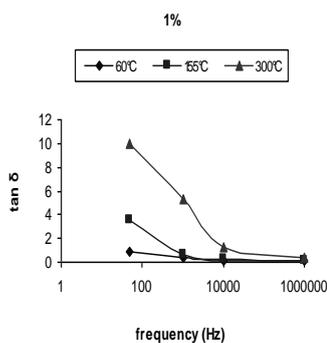


**Fig. 8 Permittivity versus Frequency at Various Temperatures of 1wt% Zirconia Nano-Composite**

From the Fig.8 1wt% of the zirconia nano-composite was analysed with various temperature ranges. From the results, it is similar to the standard polymer sample temperature increases with increase its permittivity. Similarly when the frequency was increases the permittivity of the sample was decreases. For the samples pure, 1wt%, 3wt% and 5wt% were analysed for the performance of permittivity of the samples the temperature with frequency. For all the cases the same results were found.



**Fig. 9 Dielectric Loss versus Frequency at Various Temperatures of Standard Polymer**



**Fig. 10 Dielectric Loss versus Frequency at Various Temperatures of 1wt% Zirconia Nano-Composite**

Fig. 9 and Fig. 10 are represents the dielectric loss versus various frequencies at various temperatures for standard polymer (pure) and 1wt% zirconia nano-composites. From these results, when the frequency increases with decrease in dielectric loss. Because at lower frequencies at low frequencies dipoles are able to keep in phase with changes in electric field and power losses are low. As frequency is increased the point is reached when dipole orientation cannot be completed in time available and the dipole becomes out of phase. This internal friction leads to generation of heat. When frequency is increased further there is no time for substantial dipole movement so the power losses are reduced. The dielectric loss becomes higher at 300°C for power frequency. At lower temperature in power frequency the dielectric loss is not much variation among the nano-composites. The dielectric losses become higher at 1wt% sample from 50Hz to 1 KHz.

### C. Partial Discharge Measurements

Partial discharges are in general a consequence of local electrical stress concentrations in the insulation or on the surface of the insulation. It is the main important property for any insulation system for a long term process. Generally such discharges appear as pulses of duration of much less than 1s. The partial discharge includes a wide group of discharge phenomena such as internal discharges occurring in voids or cavities within solid or liquid dielectrics, surface discharges appearing at the boundary of different insulation materials, corona discharges occurring in gaseous dielectrics in the presence of inhomogeneous fields and continuous impact of discharges in solid dielectrics forming discharge channels (treeing). The partial discharge measurement has done by both uniform and non uniform filed electrode configurations.

**Table 1: Partial Discharge Inception and Extinction Voltages**

Sample	Inception voltage (kV)	pC	Extinction voltage (kV/cm)	pC
0%	4.8	39	4.1	1.5
1%	5.1	33	4.2	1.1
3%	5.2	35	4.4	1.2
5%	5.6	40	4.6	1.3

The different values of PD inception and extinction voltage for uniform field configurations are shown in table 1. From the results the 5wt% nano-composites sample has higher inception and extinction voltages.

### D. Dielectric Strength Measurements

All electrical equipments are operating at higher voltage levels. In order to determine the electrical strength of the materials by using dielectric strength measurement test. It is also called as breakdown strength test. The breakdown voltage shows an increasing

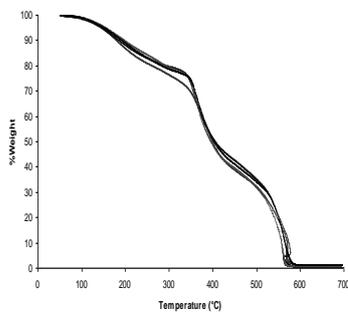
dependence on the nature and smoothness of the electrode material. The breakdown strength reduces considerably due to the presence of impurities. The breakdown field strength is an extraordinary important material property for dimensioning an insulation system. The breakdown strength of the nano-composites at different configurations is done by breakdown voltage measurements. The different values of AC breakdown voltage test for uniform field configuration are shown in table 2. From the results the 1wt% nano-composites has higher dielectric strength.

**Table 2: Breakdown Strength of Various Samples**

Sample	Breakdown Strength (kV/cm)
0%	29.4
1%	30.3
3%	34.8
5%	37.8

**E. Thermo Gravimetric Analysis (TGA)**

The TGA result of the 0% (standard polymer), 1wt%, 3wt% and 5wt% are shown in the Fig. 13. The graphs of the TGA signal (actual weight loss or gain converted to percent weight loss) on the Y-axis plotted versus the sample temperature in °C on the X-axis. The melting point temperatures for various samples are given in the table 3. From the result the 5wt% sample has the higher melting point compare with other samples.



**Fig. 11 TGA Results For 0 (Pure), 1, 3, and 5wt% Nano-Composite Samples**

**Table 3: TGA Result for Melting Temperature of Various Samples**

Sample	Melting Temp (°C)
Pure	564
1wt%	554
3wt%	558
5wt%	575

**IV. CONCLUSION**

From the SEM analysis test results showed that the prepared zirconia particles were appearing in the form of nano metric size. The particle size distribution was found

to be tens of nano-meter and varying at different areas. From the results, the nano-fillers were found in nano-scale size was conformed. From the dielectric results, clearly shows that the 5wt% samples have higher permittivity (dielectric constant) compare with other samples. From the TGA results, the 5wt% sample has high melting point compare with other samples but not varies at higher values. So the 5wt% nano-composite has high thermal strength compare with other samples. But there was no significant variation of glass transition among the nano filled samples. From these test results, the additions of few weight percentages of nano fillers have superior dielectric and thermal properties were improved. From the various properties were investigated among those the 5wt% nano-composite sample performances were improved for using as insulation. For adding weight percentages above 5wt% to the standard polymer the dielectric and thermal properties will improve or not based on the investigation of the future work.

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