Thermal and Sporadic Investigation of Silicon based Rubber Nano Composites

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Abstract. This paper comprises the details about the synthesis, thermal and sporadic investigations on the nano composites. For thermal analysis, the synthesized nano composite samples are subjected to Thermo gravitometric Analysis and Differential Scanning Calorimetry tests. Whereas breakdown tests, tracking test and dielectric breakdown tests are done through galvanic investigation. The effects of fillers of various concentrations in the range of 1%, 2% and 3% of their total volume subject to different frequency in particular from 1 kHz and 10 kHz for various components. The result shows the propitious behavior of polymer nano composites. The relative permittivity and loss tangent values are reduced radically in relation with frequency. The galvanic investigation of samples indicated the promising characteristics of tracking erosion test and electrical tests for various nano composites.

1 Introduction

For the past few decades, nano technology plays a predominant role in methodological mutiny. To add feathers to the cap, high voltage aspects are inevitable from early days to till now. There are several discussions have been made on the glass transition changes for various nano fillers incorporated in polymer composites. The result shows the clear-cut view of the electrical applications [1]. When nano silica is added to epoxy resin, modulus losses can be partially or entirely offset [2]. Strong mechanical properties and morphological structure can be observed by the dispersion of silica nano particle at 1.0 weight% concentration in unsaturated polyester resin matrix [3]. When tested in the frequency range of 40Hz–10 MHz, epoxy resin using calcium and zirconium doped barium titanate ceramics as filler

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a dielectric constant of 51, which was eight times higher than that of pure epoxy resin[4]. The electrical conductivities of poly phenyl acetylene and its compounds with Emeraldine Base were investigated [5]. About 50 nm-sized Bifeo3 nanoparticles show low leakage current and a high resistance of ρ = 3*109 Ω-cm [6]. For carbon nanotube-filled elastomers with substantial mechanical actuation, the photomechanical response strength is in the range of tens of kilopascals [7]. The two main advantages of carbon fibre reinforced epoxy-based polymer composites over metallic materials are their increased stiffness and better strength to weight ratio [8]. The ageing factors like temperature and UV were investigated for silicone composite insulating materials. The dielectric losses increased with respect to time and ageing factors at the vicinity of industrial frequencies [9].
Due to non-uniform contamination, the insulation needs to be resized, or preventive measures for insulator pollution management need to be put in place [10]. In most places, the insulators were made with the compound called silicon-di-oxide, the ceramic material which has the major composition of Silicon. This made the cost to be cheaper than any other composites. This cheapness is due to its abundant presence, but when the size, weight, requirements of the raw materials are considered, it ranks last. In order to decrease the size and weight of the raw material used to make insulators, nano composites are being created [11]. But in the last decade, the authors suggested different types of nano fillers with different composition of matrix material [12-15]. High voltage insulation requires specific ceramic disks, such as 40-disc type insulators for 440kV transmission lines. Nano-composites, which are less weighty and easy to use, can provide perfect insulation with 1 or 2 insulators. However, there is a literature gap on galvanic and alternating investigations for certain nano composites.

2 Material selection

Polymer Nano-Composite are Polymer composites with nano-dimensions that are prepared by mixing polymers with inorganic materials such as reinforcing fibers are assorted in an organic fillers and that particulate solids exhibit physical properties synergistically.

2.1 Matrix Material

Silicone Rubber is a polymer matrix which is a base with large molecule of repeating structural units.

2.1.1 Properties of Nano-Fillers:

Nano-Filler, a non-reactive, stable, and excellent dielectric and electrical conductor, can withstand temperatures ranging from -55°C to 300°C, providing thermal and galvanic properties.
a. Al_2O_3: This composition improves the abrasive, mechanical strength, and thermal stability of polymer, making it appropriate for use in abrasive applications and cutting tools. Good adherence is ensured by its insulating qualities and high dielectric constant.
b. TiO_2: It provides good insulation to the materials to which this material is added at nano size. Its high dielectric constant, which is larger than or equal to 100, is another feature.
c. $BaTiO_3$: Barium titanate, with a high dielectric constant, offers good insulation and can be formed from titanium dioxide and barium carbonate. It undergoes phase changes, increasing permittivity by 40%. Its negative bulk modulus stiffens it, and it is insoluble in water.

3 Synthesis and testing samples

3.1 Synthesis of Polymer Nano-Composites

In its natural state, silicone rubber is a mixture of lactic acid and water, or latex. The two roll mill and nano are used to feed silicone rubber through, distributing fillers equally throughout the sample and rolling it on top of one another. Once more, the treated samples were rolled ten to fifteen times to ensure that the nano-fillers were evenly distributed. It is crushed to take on the required shape after being run through the dye at a temperature of 200°C.

3.1.1 Sample dimension

The samples are made in Madurai at M/S Acme Industries. Samples with a 90 mm diameter and thickness ranges of 2 mm, 4 mm, 6 mm, and 8 mm are ready.

3.1.2 Sample details

Nano Fillers are combined with silicone at weight percentages of 1%, 2%, and 3%, and 40 samples are chosen for examination. In a similar manner to pure silicone rubber, alumina, titanium dioxide (rutile), and barium titanate nanofillers are combined at weight percentages of 1%, 2%, and 3%.

3.2 Thermal Analysis

A collection of techniques centered on figuring out how a material's chemical or physical characteristics vary with temperature is known as thermal analysis. The three main thermo analytic techniques are differential scanning calorimetry, differential thermal analysis, and thermogravimetry. At Karaikudi's Central Electro Chemical Research Institute (CECRI), materials are evaluated here.

3.3 Tracking and erosion test

The samples are evaluated at Sona Engineering College in Salem for tracking and erosion tests.

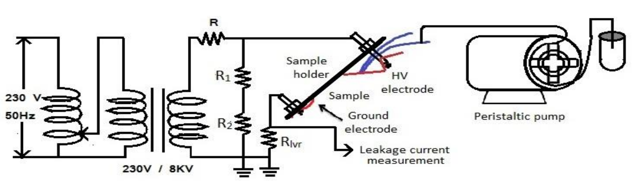

Fig. 1. Inclined Plane Tracking and Erosion Test Setu.

3.3.1 Tracking Index

An electrical breakdown on the surface of an insulating substance is known as tracking. A material's electrical breakdown characteristics are measured using the Tracking Index. The Sample is positioned between two electrodes and voltage is applied in order to measure the tracking. Reduce the distance as much as you can. 50 drops of a 0.1% ammonium chloride solution should be added per minute. Watch to see when a voltage arc appears. By generating a carbonized track, a significant voltage differential progressively forms a conductive leakage channel over the material's surface. IEC standard 60112 specifies the testing procedure.

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3.4 Breakdown Tests

Samples undergo breakdown testing at National Engineering College, Kovilpatti, between two Rogowski electrodes. High voltage is maintained, and the electrode is grounded. The setup is immersed in insulating oil, and the arcing occurs when the sample gets punctured. The breakdown voltage is measured at this point.

3.5 Dielectric Studies

Samples are evaluated at the Central Electronics Engineers Research Institute (CEERI), Pilani, for dielectric research. The AGILENT 4284A LCR metre is used to test the samples. Tan delta and permittivity, two dielectric characteristics of rubber nanocomposite materials, are the subjects of the current study.

3.5.1 Relative Permittivity Test

The Central Electronics Engineers Research Institute (CEERI), in Pilani, is where the samples are evaluated. The AGILENT 4284A LCR metre is used to test the samples. In a composite insulation system, permittivity controls the electric field distribution and a dielectric material's ability to store charge. According to the graph's outcome, relative permittivity is rising.

3.5.2 Dielectric Loss Test

The Central Electronics Engineers Research Institute (CEERI) evaluates samples using the AGILENT 4284A LCR metre, utilizing Tan delta values to measure dielectric losses in insulating materials.The result of the graph indicates there is a decrease in dielectric loss.

4 Characterization results and discussions

4.1 Thermal Analysis

Thermogravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC) determination are used to explain the thermal analysis findings.The graph shows that the blue indicates the characteristic curve for Differential Scanning Calorimeter (DSC) and the green indicates the characteristic curve for Thermo Gravimetric Analysis (TGA).

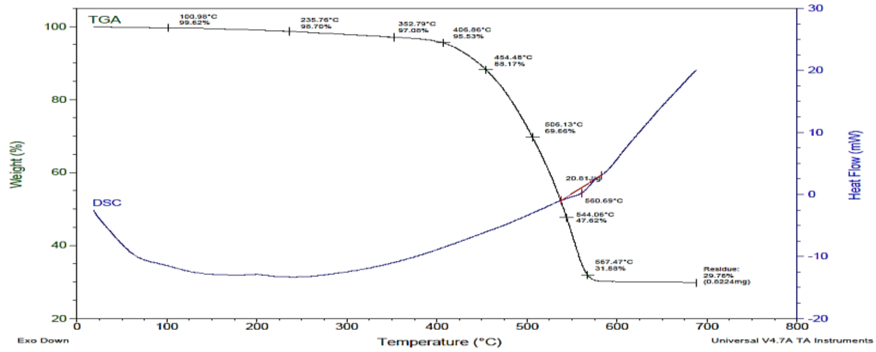

Fig. 2. DSC and TGA characteristic curve for synthetic rubber

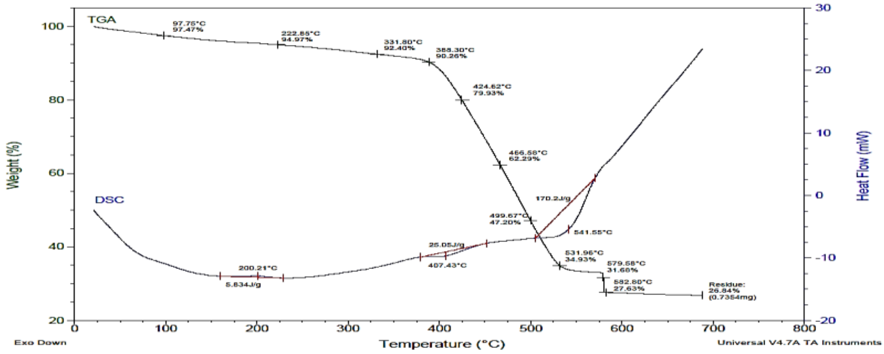

Fig. 3. DSC and TGA characteristic curve for synthetic rubber with 3% Alumina

From the Figure 2, it is clear that the initial decomposition temperature is slightly higher that is 406.86 ˚C for TGA analysis. From the above figures (2 to 7), it is clear that the initial decomposition temperature will be decreased for most of the nano - composites with the fillers.

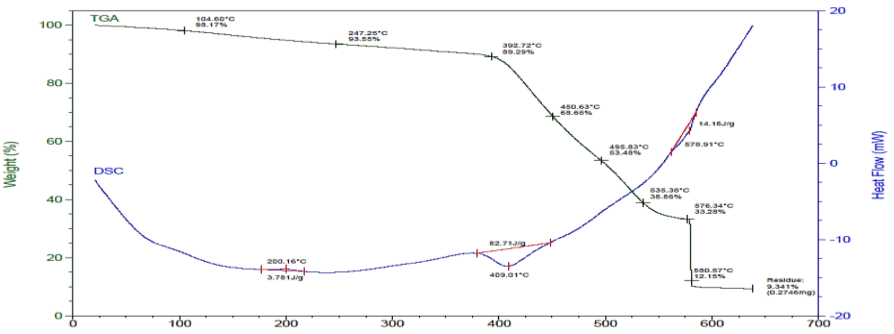

Fig. 4. DSC and TGA characteristic curve for rubber with 3% Titanium di Oxide

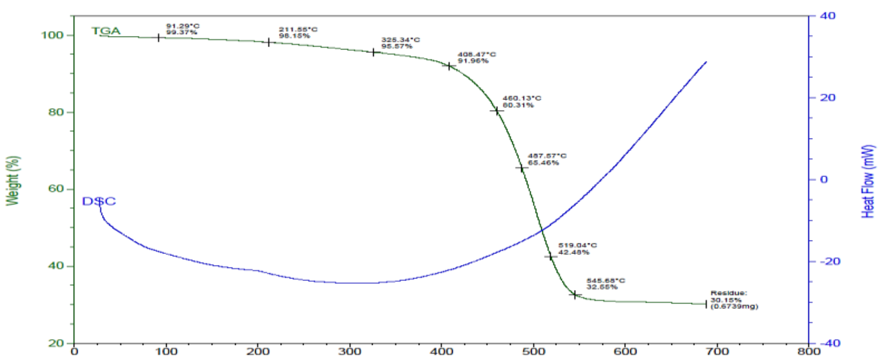

Fig. 5. DSC and TGA characteristic curve for synthetic rubber with 2% Alumina

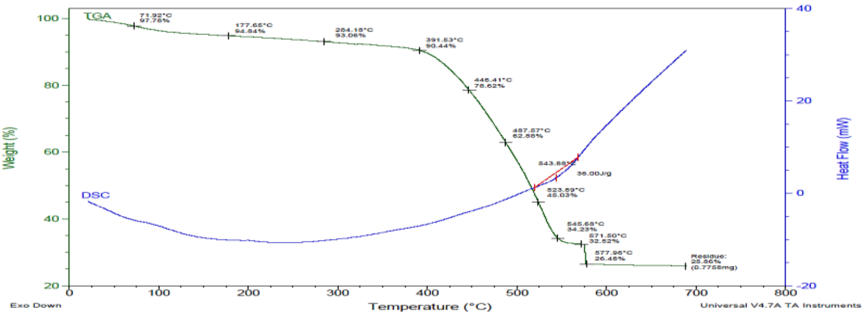

Fig 6. DSC and TGA characteristic curve for rubber with 3% Barium Titanate

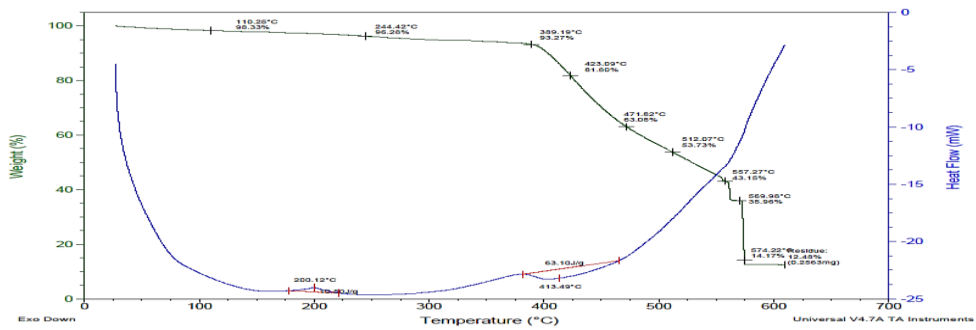

Fig. 7. DSC and TGA characteristic curve for rubber with 2% Titanium di Oxide

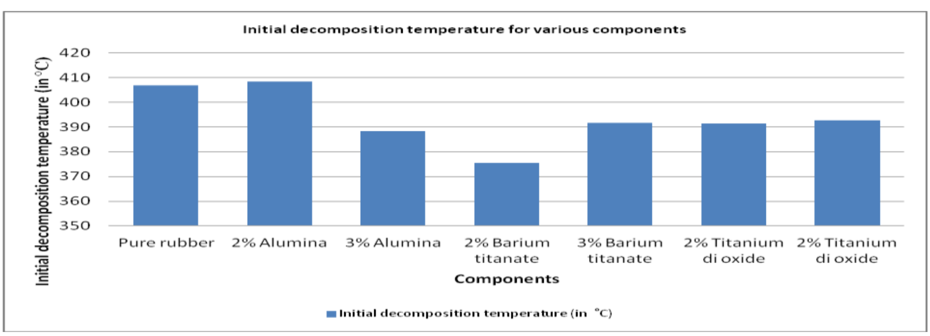

Fig. 8. Initial decomposition temperature for various components

Fig.8 shows that there is a decrease in initial decomposition temperature when adding nano - fillers to the pure rubber with respect to changes in weight of samples. As a whole the sample of rubber with 2% Barium titanate holds good for this test with the initial decomposition temperature of 375.38˚C.

4.2 Tracking and Erosion Test

For the tracking samples are tested at Sona Engineering College, Salem. The outcome of the study shows that there is tracking index for the tested samples, and the material is certified as passed and the sample can withstand upto 600V. These are some of the tracking images of the samples shown in figure 9.

Fig. 9. Tracking images for various components & Breakdown images for various components

4.3 Breakdown Tests

For Breakdown tests the samples are tested at National Engineering College, Kovilpatti. In this, the samples are tested under high voltage and check how much voltage it can withstand.

Table 1. Withstand Voltage And % Rise for Various Components

Component	Breakdown voltage	Percentage rise
Pure rubber	22.75 kV	-
3% $BaTiO_3$	25.3 kV	11.21 %
3% TiO_2	27.04 kV	18.86 %
3% Alumina	29.18 kV	28.27 %

The table 1 shows the withstand voltage and percentage rise of various components of pure rubber, rubber+3%$BaTio_3$, rubber +3% TiO_2 (Rutile) and rubber +3% Alumina.

Results show that there is an increase in breakdown voltage when adding nano - fillers to the pure rubber. From this test we can infer that it can withstand 29.18KV for 3% Alumina with the rise of 28.27%. The breakdown images of the samples which is shown in figure 9.

4.4 Relative Permittivity Results

The AGILENT 4284A LCR metre is used in the study at CEERI, Pilani, to test samples for permittivity, a measure of dielectric characteristics in rubber nano-composites that influences charge storage capacity and electric field distribution in composite insulation systems.
The study reports on the dielectric permittivities and tan delta of rubber nano-composite materials, including single Alumina, BaTiO3, and TiO2 nano-fillers, at low filler concentrations(1%, 2%, and 3%).

4.4.1 Effect of Filler Material

Through orientation polarization effects, nano-filler, a polymer or primary particle compound, affects the dielectric permittivity in rubber nano-composites including Al_2O_3, $BaTiO_3$, and TiO_2 nano-fillers.

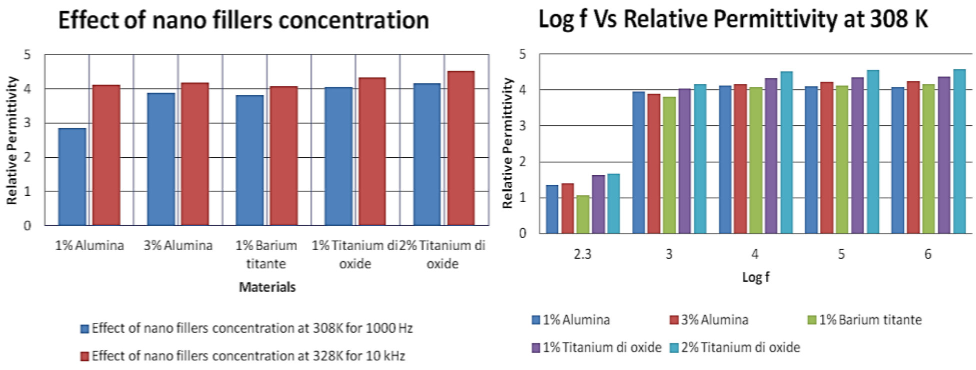

Fig. 10. Effect of nano filler concentration at 308K for 1000HZ and 328K for 10kHZ **&** Log F Vs Relative Permittivity for Various Nano –Composites

The permittivity of the system may be influenced by unreacted dipolar groups that contribute to the polarization processes in epoxy nano-composite materials and may have defined dipole moments. In comparison to an unfilled rubber system, a filled rubber system will have a substantially lower number of these dipolar groups.

Rubber permittivity, filler permittivity, and composite filler concentration all have an impact on effective permittivity. The permittivity in the polymer is usually small and changes will also be marginal because of frequency effect. Despite the immobilization of the polymer chain, the permittivity associated with the rubber component remains because of the unreacted dipolar group in the nano - composite materials, which is free to orient with the applied electric field. Applying the Lichteneker-Rother mixing rule,

$$Log\ \varepsilon c = x\ Log\ \varepsilon_1 + y\ Log\ \varepsilon_2 \qquad (1)$$

The effective permittivity (εc) is determined by dividing the filler and polymer permittivity (ε_1 and ε_2) by the concentration of the filler and polymer.

The minimum effect of nano-filler permittivity and strong polymer-nano-particle interactions define the effective permittivity of nano-composite, resulting in the lowest permittivity seen in Al_2O_3, $BaTiO_3$, and TiO_2 filled rubber nano-composites.

By increasing filler concentration, the effective permittivity of nano – composites can be changed and the following graph shows the resultant of adding nano filler concentration at 308K for 1000Hz.

4.4.2 Effect of Filler Concentration

Rubber nano-composite permittivities are lower than empty rubber due to dielectric polarization mechanisms caused by space charge accumulation. High filler concentrations increase nanoparticle quantity and cost but reduce effective permittivity and immobility. Interfacial phenomena, polarization mechanisms, and interfacial effects are more prominent at lower concentrations, weakening surfaces.

The various Nano-fillers of Titanium di Oxide – TiO_2, Barium Titananate – $BaTiO_3$, Alumina - Al_2O_3 are selected to enhance the relative permittivity. The various filler weight percentages of 1%, 2%, 3% are selected. With the various weight percentages of pure rubber the material's relative permittivity can be increased and moreover the percentage rise of dielectric constant also increases with different filler concentration. The above-mentioned graph shows the influence of nanofiller concentration on vibration at 10 kHz at 328K is investigated in this study.

4.4.3 Effect of Frequency

The effective permittivity of nano-composite materials decreases with increasing frequency due to the influence of individual permittivity of rubber, fillers, and filler loadings. As frequency increases, the permittivity of pure rubber and Al2O3, BaTiO3, and TiO2 also decreases. This results in a gradual decline in permittivity in the combined system, with measurement at various frequencies with different fillers.

For different nano-composite materials, the relative permittivity is measured along the nano-fillers when the frequency varies and the temperature stays constant. For example, the relative permittivity of rubber with 1% $BaTiO_3$ increases gradually from 1.055820 to 4.156022 as the frequency increases by keeping the temperature remains constant at 308K.

4.5 Dielectric Loss Test Results

The samples are analysed for dielectric loss at CEERI, Pilani, using an AGILENT 4284A LCR metre. Polymer nano-composites with enhanced dielectric and electrical insulation qualities are becoming increasingly popular. The phrase "nano-dielectrics" is becoming more popular. Tan delta is another dielectric characteristic that indicates the dielectric losses in an insulating material. Electrical insulation systems require a low tan delta value, whereas high permittivity is preferred depending on the application.

4.5.1 Effect of Filler Material.

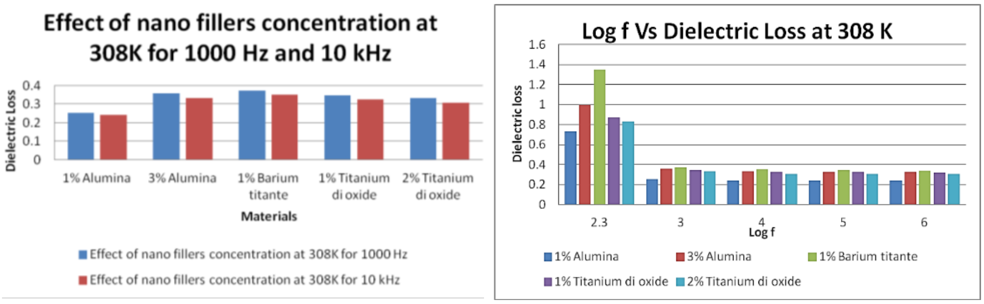

Fig. 11. Effect of Nano Fillers At 308K for 1000Hz and 10kHz **&** Log f Vs Dielectric loss at 308 K

Nano-fillers, like alumina, titanium di oxide, and barium titanate, are used in rubber matrix to reduce dielectric loss, affecting tan delta values and reducing frequency fluctuations as shown in Figure 11.

4.5.2 Effect of Filler Concentration

With the various weight percentages of pure rubber the material's dielectric loss can be decreased and percentage rise of dielectric constant also decreases with different filler concentration.

Electrical conductivity and excitation frequency impact the loss tangent in polymers and composites. For all filler concentrations, nano fillers in filled rubber nano-composites exhibit a minor drop in tan delta values with increasing frequency. Tan delta values in filled rubber decrease as frequency increases as compared to tan delta values in empty rubber.

4.5.3 Effect of Frequency

Charge carriers in nano-composite systems, particularly along polymer chains, contribute to high-frequency conductivity, but polymer chain entanglements limit charge mobility, resulting in lower electrical conductivity.
When the frequency varies and the temperature remains constant, the dielectric loss of different nano composites along their Nano fillers is calculated.
For example, consider the relative permeability of rubber with 1% Al_2O_3 decreases gradually from 0.733482 to 0.244792 as the frequency increases by keeping the temperature remains constant at 308 K.

5 Conclusion

The experiments show that Alumina samples perform well in thermal analysis, particularly TGA and DSC, and exhibit excellent withstanding capability under polluted conditions. Alumina's electrical properties, including breakdown voltage, relative permittivity, and tan delta, are significantly impacted by its presence. This cost-efficient nano composite material is suitable for electrical insulators.

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