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**ORIGINAL RESEARCH ARTICLE**

# Dielectric Response Analysis of Polyvinylidene Flouride Doped With Alumina, Titanium Oxides, and Calcium Carbonate Nanoparticles

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**ABSTRACT**

Nanoparticle incorporation in polymeric materials alters their inherent properties, such as dielectric constant and loss, hence affecting their application as dielectric materials. In the current study, three different oxide-based nanoparticles (Calcium carbonate, Aluminium and Titanium oxides) were added to polyvinylidene Flouride (PVDF) in the concentration of 0.5, 1.0, and 1.5%w/w for each nanoparticles to develop polymer nanocomposites in order to study the effect of such addition on the dielectric responses of PVDF over wide frequency range (20 Hz-2 MHz) using LCR digital frequency meter. The polymer nanocomposite samples were developed using a rapid roll-mix-milling and compression technique at 50°C. The addition of varying nanoparticles to the polymer matrix seems to have the same effects at rising Frequency on the relative permittivity, dielectric loss and conductivity. This suggests that it may be an interesting material for use in high-energy storage devices. A study of the structure and mechanical properties of the developed material is recommended for other potential application areas.

## INTRODUCTION

Nanocomposite dielectrics are engineered by uniformly dispersing nanoparticles into a dielectric matrix, and such structural modification systematically enhances properties such as breakdown strength, thermal stability, and mechanical durability (Zhang *et al.*, 2021). This integration not only endears the material's capacity to endure high electric fields but also improves its overall uniqueness, making these composites attractive for a variety of applications ranging from high-voltage insulation and capacitors to flexible electronics. The performance of these nanocomposites critically depends on the type of nanoparticles used and how uniformly they are dispersed within the host dielectric material (Saleh *et al.*, 2020). Dielectric materials exhibit distinct electrical insulating properties and are critical in a wide range of applications, including capacitors, sensors, and electronic devices. Among these materials, polyvinylidene fluoride (PVDF) stands out due to its excellent thermal stability, high mechanical strength, and notable dielectric properties (Wang *et al.*, 2019). PVDF, a semi-crystalline polymer, possesses unique ferroelectric characteristics, making it suitable for applications in energy harvesting and piezoelectric sensors (Zhang *et al.*, 2020). However, enhancing the dielectric constant and reducing the dissipation factor of PVDF remains a challenge for advancing its functionalities.

Recent advancements in polymer nanocomposites have provided innovative avenues to improve on the dielectric properties of PVDF. By incorporating non-conductive or dielectric nanoparticles, the overall performance of the composite material can be significantly enhanced. The addition of nanoparticles, such as metal oxides (e.g., $TiO_2$, $BaTiO_3$), carbon-based materials (e.g., graphene, CNTs), or other fillers, leads to interfacial polarization and the formation of conductive pathways, thereby amplifying the dielectric constant and lowering energy loss (Kumar *et al.*, 2021; Xu *et al.*, 2022).

The aim of this dielectric analysis is to investigate the dielectric properties of PVDF doped with various nanoparticles, utilizing LCR (inductance, capacitance, resistance) measurements as a primary analytical technique. LCR technique is a common method that allows for the accurate assessment of the dielectric behavior and frequency response of materials, providing critical insights into the capacitance and resistive

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components that defines the dielectric performance (Moussa *et al*., 2021). This research work seeks to explore how the incorporation of different nanoparticles affects the dielectric response of PVDF, shedding light on mechanisms that underpin enhanced performance while discussing the implications of such addition from the results obtained.

## MATERIALS AND METHODS

### Materials

PVDF with a high molecular weight was acquired from Sigma-Aldrich. The polymer was selected due to its excellent piezoelectric properties and thermal stability. Nanoparticles (Alumina, $TiO_2$, and $CaCO_3$) of high grade were also obtained from BS-Partickel GmbH, Germany.

### Sample Preparation

The PVDF pellets and nanoparticles were separately dried at 80°C for 4 and 6 hours respectively under vacuum in an oven to remove moisture content. This is important because moisture removal is critical to avoid defects like voids or poor dispersion. The PVDF was pre-mixed with the nanoparticles physically using a high-speed mixer. The polymer samples were thereafter measured each of 50g with an addition of 0.5%w/w, 1%w/w and 1.5%w/w of nanoparticles respectively. The premixed samples were processed using a two-roll mill machine operating at 54 rpm. The roller temperature was set and maintained at 50±1℃ due to the material's hardness. Premixed samples with varying nanoparticle contents and control were placed between the rollers, where repeated thermal contact transformed them into a molten state. The molten polymer samples were molded into 80×60 mm² sheets using a molder and then compressed in a compressing machine to achieve the desired shape. This process helped to disperse the nanoparticles more evenly throughout the polymer matrices. The samples are summarized and described in Table 1.

### Sample Characterization Using LCR Bridge

The LCR analyzer (shown in Figure 1a) displays measured values, including capacitance and loss tangent, on its screen. These recorded values are then subjected to further analysis.

**Table 1: Sample Description**

| S/n | Samples | Composition |
|---|---|---|
| 1 | Al-1 | PVDF+0.5%w/w Alumina |
| 2 | Al-2 | PVDF+1%w/w Alumina |
| 3 | Al-3 | PVDF+1.5%w/w Alumina |
| 4 | C-1 | PVDF+0.5%w/w Calcium carbonate |
| 5 | C-2 | PVDF+1%w/w Calcium carbonate |
| 6 | C-3 | PVDF+1.5%w/w Calcium carbonate |
| 7 | T-1 | PVDF+0.5%w/w Titanium Oxide |
| 8 | T-2 | PVDF+1%w/w Titanium Oxide |
| 9 | T-3 | PVDF+1.5%w/w Titanium Oxide |
| 10 | Control | Neat PVDF sample |

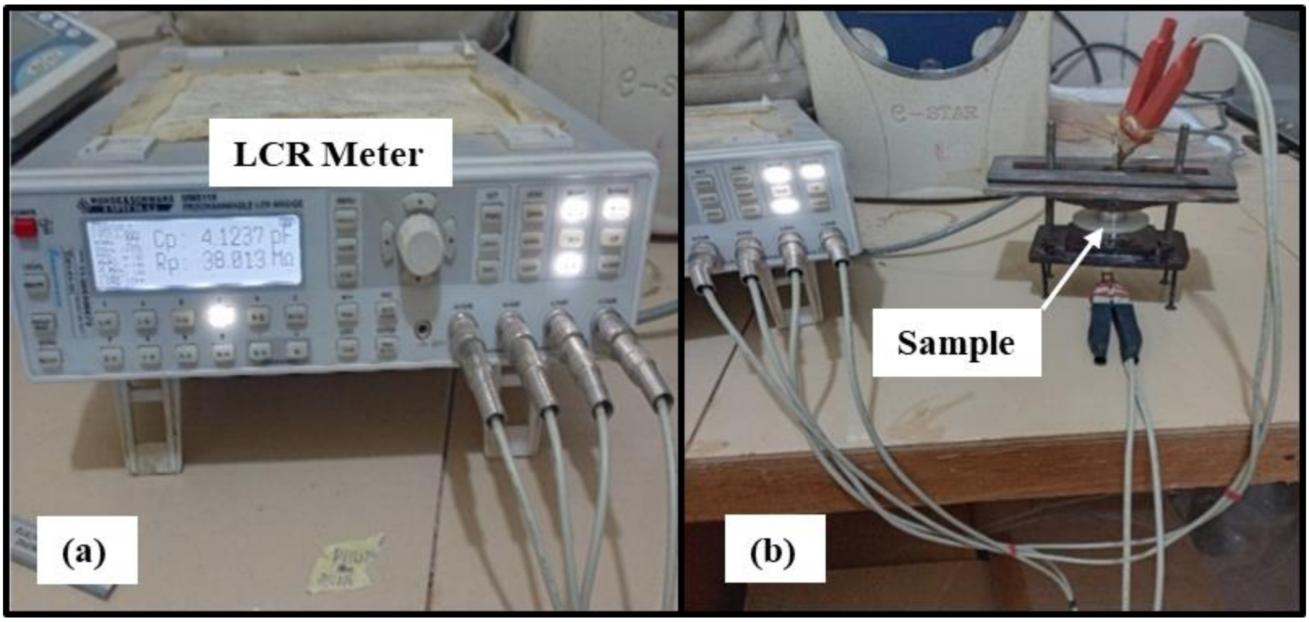


**Figure 1: Experimental set up**

The experimental setup consisted of the sample placed in the sample holder (test cell), with the high and low terminals of the LCR analyzer connected to either side of the sample holder, as shown in Figure 1b. Several capacitance and resistance values of the polymer samples were recorded.

## RESULTS AND DISCUSSIONS

This section presents and discusses the dielectric response test results obtained from the samples.

Figure 2 shows the variation of the relative permittivity for a polymer composites/samples as a function of Frequency. The relative permittivity, defined as the ability of a material to store electrical potential energy under the influence of an electric field, serves as a key indicator of how effectively the material stores versus dissipates electrical energy. Sample C-2 had the highest relative permittivity. Adding 1w% of calcium carbonate to PVDF caused its dielectric constant to increase. As frequency increases, the polymer's molecular chains gain energy, intensifying molecular agitation. This heightened mobility enhances dipolar friction, leading to an initial increase in relative permittivity. However, the molecular agitation exhibits distinct patterns, with all samples displaying peaks except the undoped sample. Meanwhile, at higher frequencies, the rapid oscillation of the electric field hinders dipole alignment, resulting in a consistently stretched response

The graph exhibits an initial peak at characteristic molecular vibration frequencies, followed by a plateau or slight decrease in permittivity at 60 Hz and 2000 Hz. This suggests that at higher frequencies, the samples' dipolar alignment with the electric field becomes less efficient, potentially due to increased disorder, limiting further energy dissipation (Pinto & Martins 2020).

The trend in Figure 2 underscores the pivotal role of Frequency in shaping the dielectric performance of filled-PVDF. This insight is crucial for optimizing polymer-based dielectrics in applications demanding stable performance across varying operating conditions, especially in high-frequency and high-temperature settings (Kremer & Schönhals, 2003).

Figure 3 shows the relationship between dielectric loss and Frequency for PVDF doped with CaCO3, alumina, and TiO2. The graph indicates that dielectric loss, which represents energy dissipation due to dipolar alignment lag, remains relatively constant with increasing Frequency.

At low frequencies, the molecular chains in the polymer are relatively relaxed, increasing dipole mobility. This enhanced freedom enables dipoles to reorient more dynamically, resulting in higher energy dissipation under an electric field. Conversely, as frequency increases, the polymer chains become more rigid, restricting dipole movement and potentially affecting the doping efficiency.

At the onset of the electric field, rapid oscillations may cause frequent field direction changes, contributing to increased dielectric loss. In contrast, at lower frequencies, the slowly oscillating electric field allows dipoles sufficient time to align, resulting in comparatively lower dielectric losses (Niu *et al*, 2017).

Furthermore, the sharp increase in dielectric loss at lower frequencies suggests the onset of a relaxation process. This process occurs when dipoles reach a critical point where their response to the electric field becomes significantly delayed, leading to a marked increase in energy dissipation.

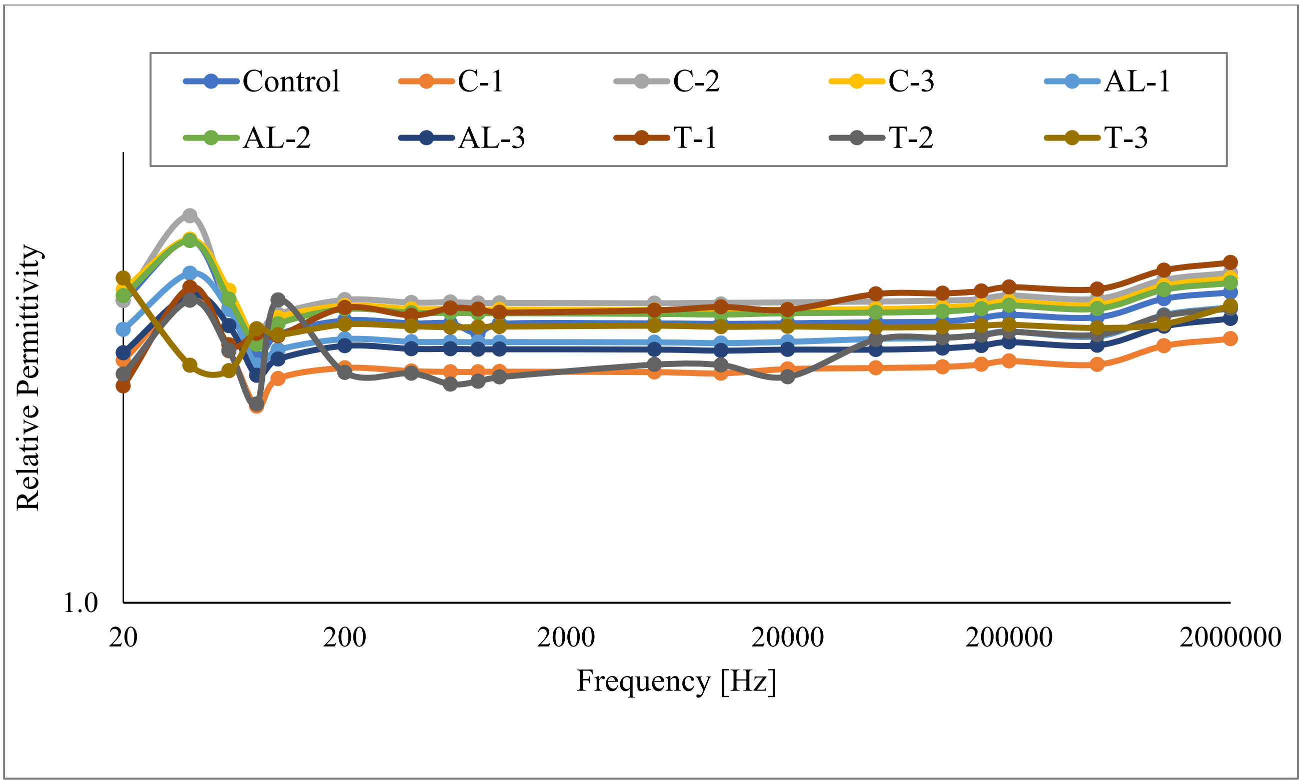


**Figure 2: Dielectric dispersion of the samples developed**

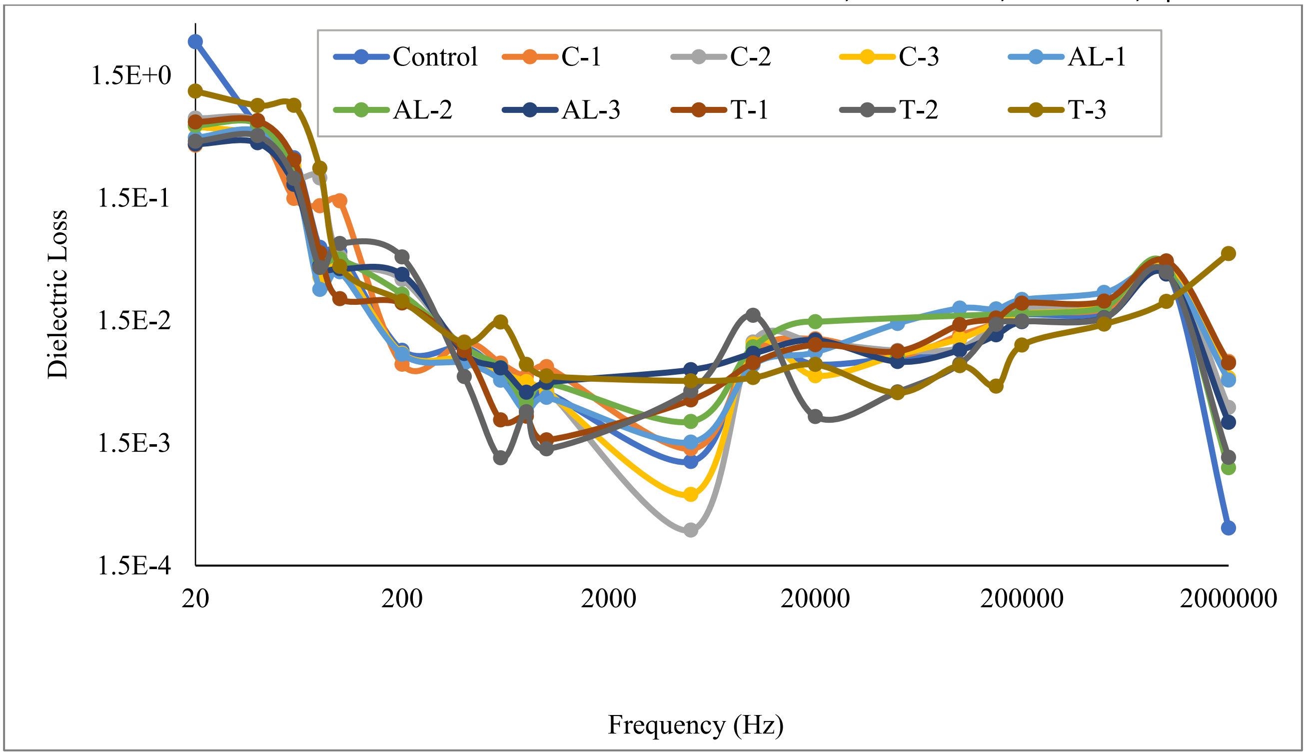


**Figure 3: Frequency dependence of dielectric loss**

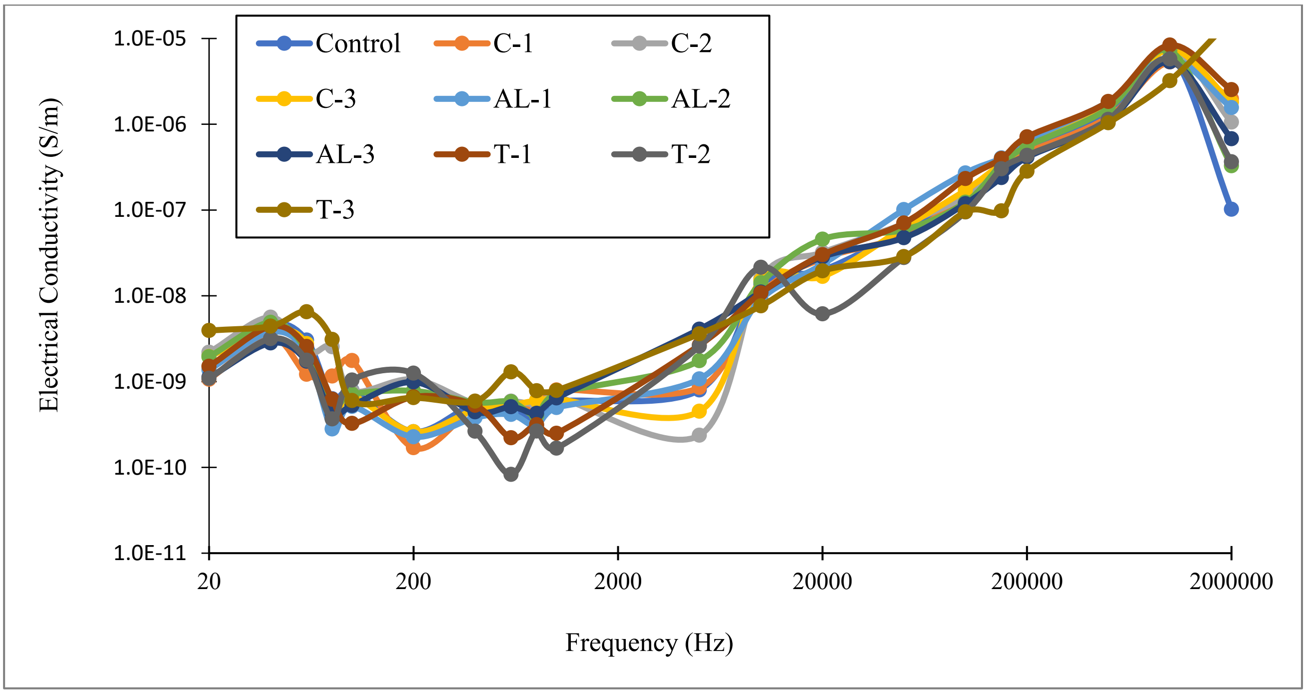


**Figure 4: Frequency dependence of electric conductivity of the polymer samples**

Figure 3 not only highlights the intrinsic sensitivity of PVDF's dielectric loss to Frequency but also underscores the importance of managing these parameters in applications. Understanding these dependencies is crucial for optimizing the performance and reliability of polymer-based dielectrics in high-frequency and high-temperature environments, such as in capacitors, insulating materials, and other electronic components (Kremer & Schönhals, 2003).

At lower frequencies, charge carriers have ample time to respond to the applied electric field, leading to a more pronounced steep rising in conductivity with rising Frequency. However, at higher frequencies, the scenario becomes more complex. The rapid oscillations of the electric field challenge the ability of the polymer chains and dipoles to align promptly. This misalignment can affect the pathways available for charge transport, potentially causing subtle variations in conductivity that deviate from the simple thermal activation model.

Figure 4 underscores that polymer resistivity which is the inverse of conductivity plot in the graph, with a marked drop indicating a decrease in the enhanced charge mobility. At the same time, frequency effects introduce an additional layer of complexity, highlighting how polarization dynamics and the finite response time of polymer chains can modulate conduction. This comprehensive behavior reflects the dual nature of conduction in polymers, where both thermal energy and the dynamic response to alternating fields are critical factors in determining electrical performance (Dyre, 1988).

## CONCLUSION

Polymer nanocomposites were developed using a rapid roll-mix-milling and compression technique. Calcium carbonate-based polymer nanocomposites exhibited an enhanced dielectric constant and reduced dielectric loss, making them suitable for energy storage applications. These nanocomposites have shown promising dielectric response properties, indicating their potential for use in high-voltage capacitors and electronic applications Future research will investigate the impact of nanoparticle addition on the structure, morphology, and mechanical properties of PVDF, exploring its potential applications in various industrial areas

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