*Research Article*

# Dielectric Properties of TiO$_2$/Silicone Rubber Micro- and Nanocomposites

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Room temperature vulcanized (RTV) silicone rubber SR/TiO$_2$ nanocomposites and microcomposites are developed and characterized, and their dielectric behaviour and electrical conductivity are studied in this paper. We demonstrate that the surfactant Triton X-100 greatly improves the dispersal of micro- and nanoparticles across the surface to produce more homogeneous composites that have improved dielectric properties. This heightened dispersal with the presence of a surfactant is also confirmed by SEM analysis. We also discuss the influence of the filler concentration and particle size on the dielectric behaviour of the nanocomposites and the microcomposite surfaces having a frequency range of 40 Hz to 2 MHz. The dielectric properties are improved by the introduction of 5 wt.% and 10 wt.% TiO$_2$ nano- and microparticles. Furthermore, there is an improvement in the permittivity values for the microcomposites compared to the nanocomposites for all frequencies. This finding is of great importance for high-voltage electrical insulation.

## 1. Introduction

Over the past decade, there has been growing interest in nanocomposites, a new class of reinforced polymers produced by the introduction of nanoparticles into a polymeric matrix [1]. The large specific surface area of nanoparticles makes them very reactive, thereby giving nanocomposite coatings unique and remarkable properties. Multiple characteristics, including hydrophobicity, flame resistance, and the UV resistance of coatings, can be improved by adding small quantities of nanoparticles [2–4]. Furthermore, these coatings can be applied to all surfaces. The difficulties encountered during the elaboration of nanocomposites generally involve the choice of base polymer as well as the nature of the nanoparticles (both in terms of size and quantity), the optimization of the production process, and, in particular, the verification of the homogeneous dispersal of nanoparticles within the polymeric matrix [5].

The degree of nanoparticle agglomeration in a sample reflects the quality of the dispersal. One of the limitations to the development of nanocomposites is the ability to develop manufacturing processes that favour the homogeneous and reproducible dispersal of nanoparticles within nanocomposites. Poor dispersal increases the number and size of the aggregates, leading to the deterioration of the properties of the materials [6–8].

Moreover, the instability of nanoparticles with regard to agglomeration poses a real obstacle in controlling their size. Three approaches are generally adopted to stabilize nanoparticles against agglomeration and/or growth: stearic stabilization, electrostatic stabilization, and microemulsion synthesis. From these approaches, we used stearic stabilization by a surfactant. This technique allows the surface of the nanoparticles to be protected by the chosen surfactant, thus generating a stearic effect. This method is favoured as it is not very sensitive to the nature of chemical species present in the environment.

The surfactant molecules concentrate generally at interfaces and position themselves in such a way to increase the attractive interactions. As a result, these surfactants are adsorbed, which may cause a decrease in the surface tension. Triton X-100 (C$_{14}$H$_{22}$O (C$_2$H$_4$O)$_n$, where $n$ = 9-10) was used

as a surfactant by Ramirez et al. [9] and showed that Triton X-100 improves the dispersal of nanosilica particles to produce more homogeneous nanocomposite surfaces [9].

A wide variety of insulating materials are used for high-voltage outdoor insulation. Of these, silicone rubber has received the most attention. This polymer has unique properties, namely, high water repellency, ultraviolet (UV) resistance, and a good resistance to mechanical and climatic constraints. However, silicone rubber (SR) loses its hydrophobic behaviour over time.

To improve some of the silicone rubber properties and to reduce costs, nanoparticles may be added to the base polymer. The addition of these particles can increase the relative permittivity, surface hydrophobicity, and electrical conductivity of the composite silicone [10, 11]. Semiconducting materials, such as $TiO_2$ or ZnO nanoparticles, can also ensure the homogenization and distribution of electric fields on the insulators to reduce electrical stresses, thereby minimizing the surface flashover of insulators [6]. Cherney et al. [10] demonstrated that the introduction of barium titanate contributed to increasing the relative permittivity of silicone composites. Numerous studies have demonstrated that the obtained increase in relative permittivity is a function of concentration and the type of filler [12–17]. Momen and Farzaneh [17] have developed nanocomposite coatings of silicone rubber/ZnO and showed that permittivity increases with an increased concentration of ZnO in the coatings. Furthermore, these coatings favour a reduction in ice accumulation. Also, Feng et al. [18] have developed nanocomposite films of polyimide/$TiO_2$ composites by in situ dispersive polymerization. The pinning effect of $TiO_2$ nanoparticles to polyimide chain motion reduces relative permittivity of composites at lower doping concentrations (0–3%). As $TiO_2$ doping concentration increases (5%), the increased number of $TiO_2$ nanoparticles and the polarization effect of nanoparticle interfaces surpass the pinning effect and enhance the relative permittivity of composites above that of pure polyimide.

In this study, titanium dioxide ($TiO_2$) is selected as a filler due to its dielectric properties and, among other characteristics, its high permittivity relative to the RTV silicone rubber polymer. This relatively large permittivity is responsible for significant variations in the effective permittivity with changes of the $TiO_2$ concentration in the matrix. Also, this semiconductive material helps improve the performance of insulating materials in polluted environments by controlling the distribution of the electrical field, thereby preventing moisture condensation by heating [10]. To date, little has been published regarding the properties of $TiO_2$-filled silicon rubber for outdoor applications, and published studies related to the electrical conductivity of nanocomposites and microcomposites are very scarce. Moreover, existing studies mostly focus on conducting fillers.

Given this, the present study investigates the dielectric behaviour and electrical conductivity of RTV silicone rubber reinforced with different $TiO_2$ concentrations and sizes. For this purpose, $TiO_2$ particles were modified by adding Triton X-100, a surfactant, to improve dispersal of the filler particles. Samples of RTV SR/$TiO_2$ were characterized using a scanning electron microscope (SEM) and an impedance analyzer.

## 2. Experimental Procedures

For this study, we used a commercial RTV silicone rubber from a Xiameter that contains 40–70 wt.% alumina hydrate. We also used titanium dioxide ($TiO_2$) particles as filler. To assess the impact of $TiO_2$ particle size on the dielectric properties, average diameters of 25 nm and 1 $\mu$m were selected for $TiO_2$ particles supplied by Sigma Aldrich. The Triton X-100 surfactant was applied to disperse the fillers. Nanocomposites and microcomposites were prepared using a combination of two different processing techniques: mechanical mixing and ultrasonication. Initially, the $TiO_2$ particles were dried at 100°C for 24 hours before mixing with silicone rubber. A specific amount of filler particles (based on weight fractions) was mixed with 100 mL of hexane and 2 wt% of nonionic Triton X-100. It should be noted that beyond this percentage, the hydrophobicity of the surface was lost. Subsequently, the prepared solution was placed in an ultrasonic bath for 20 minutes and then mechanically stirred at 700 rpm for two hours to achieve a good dispersal of nanoparticles and a homogenized solution. Approximately 20 mL of RTV SR was added to the prepared solution. This solution was then placed back into the ultrasonic bath for 60 min and subsequently mechanically stirred at 600 rpm for 24 h.

Once prepared, the solution was poured into a 6.5 cm diameter mold that was kept in an oven at 70°C for 48 h to evaporate the solvent.

The morphological characterization was examined using a LEO scanning electron microscope (SEM). The dielectric permittivity was measured using an Agilent 4294A impedance analyzer over the frequency range of 40 Hz–2 MHz, and conductivity was determined using Glassman high voltage.

Conductivity was calculated from the resistance, using the following relationship:

$$U = RI, \tag{1}$$

where $U$ is the voltage (V), $R$ is the resistance ($\Omega$), and $I$ is the intensity of the electric current (A).

For this purpose, a discontinuous current (between 40 $\mu$A and 150 $\mu$A) and a maximum voltage of 50 kV were used. Resistance $R$ was determined by graphical analysis. Values of $I$ versus $V$ were plotted for a number of different voltage settings, and the correlation coefficient was obtained by linear fit to the data. Resistance $R$ was then obtained from the slope:

$$\varphi = R \cdot \frac{S}{L}, \tag{2}$$

where $\varphi$ is the resistivity of the coating ($\Omega \cdot$m), $R$ is the resistance ($\Omega$), $L$ is the sample length (m), and $S$ is the section of the coating (m$^2$).

In our case, the sample length is 8 cm:

$$\sigma = \frac{1}{\varphi}, \tag{3}$$

where $\sigma$ is the conductivity of the coating ($\Omega \cdot$m)$^{-1}$ and $\varphi$ is the resistivity of coating ($\Omega \cdot$m).

## 3. Results and Discussion

The morphology and dielectric properties of the produced coatings were subsequently evaluated.

*3.1. Surfactant Influence on the Morphology of Nanocomposites.* The dispersal of the fillers within the silicone rubber was evaluated using a SEM before analysing the morphology of the nanocomposites. Samples of approximately $1\,mm \times 1\,mm$ were prepared by cutting the nanocomposites at different locations.

Dispersal of the nanofillers is critical for obtaining consistent properties for the nanofilled material. To investigate the effects of the surfactant on the dispersal of the nanoparticles in the polymer matrix, RTV SR/TiO$_2$ samples containing 10% titanium by weight, with and without the addition of surfactant, were prepared.

The nonionic surfactant Triton X-100 was used to improve particle dispersal in the polymer. Particles are highly charged and thus tend to agglomerate and form larger particles. This agglomeration reduces markedly the particle surface to volume ratio that compromises the unique particle properties in the polymer [19]. Figures 1(a) and 1(b) show that the fabricated nanocomposite has a large number of agglomerates and poor nanoparticle dispersal in the polymer. The sample also has cracks stemming from poor dispersal (Figure 1(b)).

The microstructure of the surface at different magnification levels (Figure 2) indicates that the nanoparticles are uniformly dispersed in the polymer matrix. This illustrates how the introduction of Triton X-100 leads to a significant reduction in agglomerates by reducing the surface energy and increasing the separation of agglomerations, thereby improving the dispersal of nanoparticles. A good dispersal is achieved when the repulsive forces between the particles are greater than the Van der Waals forces of attraction or the mechanical couplings between particles [20].

*3.2. Effect of Surfactant on Relative Permittivity.* Poor dispersal of the particles in the polymer generally results in nanocomposites of poor and nonhomogeneous quality. It can also generate unwanted effects such as a drop in material performance. These problems can be solved by modifying the surface of the TiO$_2$ nanoparticles with the use of surfactants. Kim et al. [21] showed that phosphoric acids can modify the surface of BaTiO$_3$ nanoparticles by promoting the bond between the organophosphonate group and the oxide surface to create a stable organic oxide interface. This results in a good compatibility with the matrix and an improvement in the electrical properties of the material. Similarly, Cherney et al. [10] demonstrated that the introduction of inorganic fillers into the silicone rubber improves its dielectric properties. After the addition of 30% BaTiO$_3$, the relative permittivity of the polymer increased from 2.7 to 10. The study also showed that the use of materials having a high relative permittivity can reduce considerably electrical stress and improve the distribution of the electric field.

In this work, the dielectric properties of composites were determined using Agilent 4294A impedance analyzer. The studied frequencies ranged from 40 Hz to 2 MHz.

By way of comparison with the dielectric response, the variation of permittivity against frequency was studied for two samples composed of silicone rubber filled with a percentage of 10% wt of TiO$_2$ nanoparticles: one having 2% wt Triton X-100 and the other sample without a surfactant (Figure 3).

As illustrated in Figure 3, the permittivity of the two samples increases as frequency decreases: at low frequencies, all free dipolar groups can move, resulting in a high permittivity. At higher frequencies, these groups struggle to move, causing a reduction in permittivity.

However, the homogeneous distribution of particles is the main factor to explain the improvement in dielectric properties. In fact, the presence of Triton X-100 provides a chemical compatibility between the titanium dioxide particles and the silicone rubber, resulting in a more uniform particle distribution characterized by an increase in dielectric permittivity from 3.93 to 5.16 at 60 Hz between the samples.

Several factors contribute to the improvement of the properties of the composite, in particular the adhesion of the polymer matrix to the surface of the particles. An improvement in adhesion to the interface can be achieved by treatment of the particles with a dispersal agent (solvent). In our case, isopropanol was chosen as a solvent to provide a more homogeneous solution. Dielectric measurements confirmed the more uniform morphological structure resulting in an increased relative permittivity in the case of the composite with Triton X-100 when compared to the surface nonhomogeneity and weak permittivity in the other sample.

*3.3. Influence of Filler Concentration on Relative Permittivity.* The dielectric behaviour as a function of frequency in our nanocomposites is mainly determined by polarization related to RTV silicone rubber, TiO$_2$ nanoparticles, and interfacial polarization.

The variation of the real portion of the permittivity for RTV silicone rubber reinforced with different concentrations of TiO$_2$ nanofillers at 165 Hz is shown in Figure 4.

It can be seen that the real relative permittivity ($\varepsilon_r$) increases as the TiO$_2$ content in the RTV-SR is increased. As the individual permittivity of TiO$_2$ is greater than that of pure silicone rubber, it influences the value of the resultant nanocomposite permittivity. At a frequency of 165 Hz and a 20 wt.% TiO$_2$ loading, the real portion of permittivity is quadrupled. The addition of TiO$_2$ having a permittivity greater than that of the base polymer increases the permittivity of the polymer composite, mainly due to the influence of the filler permittivity. Increasing the nanofiller concentration in RTV silicone rubber causes the permittivity to increase.

*3.4. Effect of Particle Size and Concentration on the Relative Permittivity.* The use of high-permittivity materials greatly reduces electrical stress (partial electrical discharge) [10]. It is for this reason that the evolution of relative permittivity ($\varepsilon_r$) as a function of the frequency for the nanocomposites
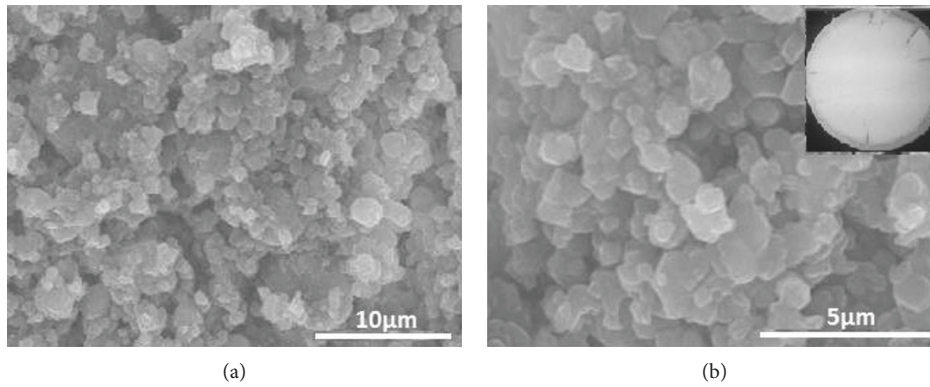
(a)                                                                          (b)

FIGURE 1: SEM images of silicone rubber/TiO$_2$ without the surfactant Triton X-100. (a) Low magnification; (b) high magnification.



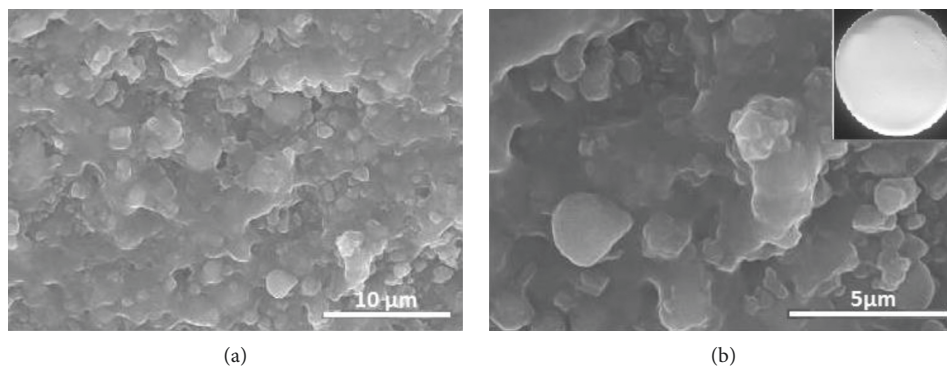(a)                                                                          (b)

FIGURE 2: SEM images of silicone rubber/TiO$_2$ with Triton X-100. (a) Low magnification; (b) high magnification.
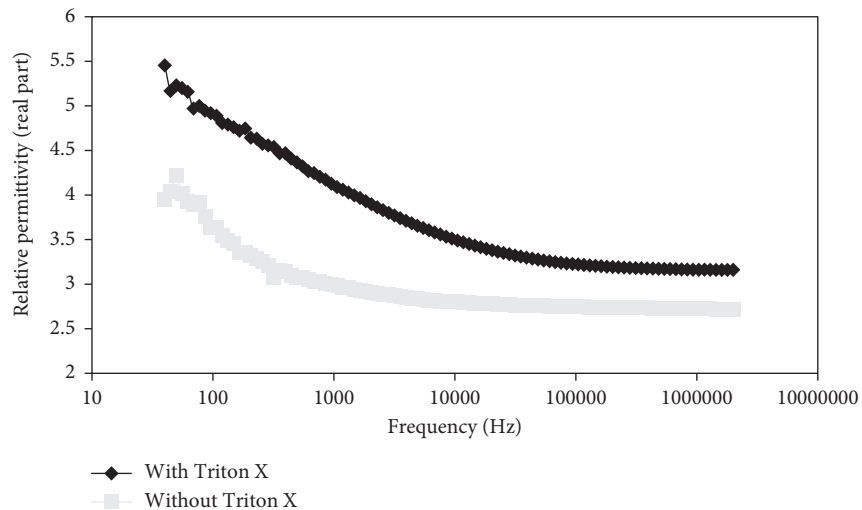


FIGURE 3: Variation of the permittivity as a function of frequency for silicone rubber/TiO$_2$ nanocomposite.

and microcomposites is studied. The results are presented for particle sizes of 25 nm and 1 μm.

The permittivity of pure and charged polymers increases as frequency decreases (Figure 5) as permittivity is dependent on the frequency in polymer systems ($\varepsilon_r \propto 1/f$) [22].

For microcomposites, the permittivity of both the 5% and 10% TiO$_2$ microparticles tends to increase more sharply at lower frequencies in the case of nanocomposites. This difference between micro- and nanocomposites is likely due to the presence of a significant volume fraction of the nanoparticles at the interfaces in the material preventing the migration of ions. This may subsequently cause a reduction in the accumulation of charges and, therefore, lower the relative permittivity [20]. On the other hand, the strong interactions
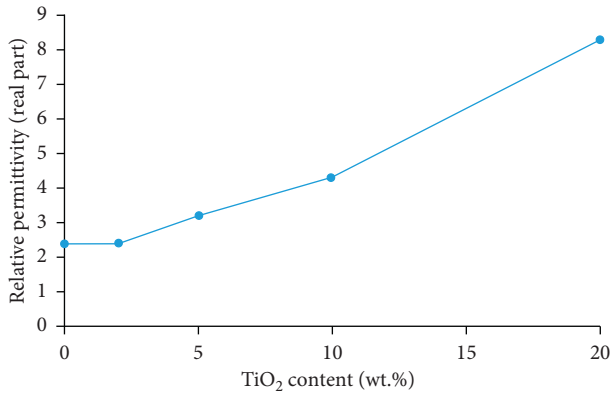
Figure 4: Effect of filler concentration on the real part of relative permittivity.
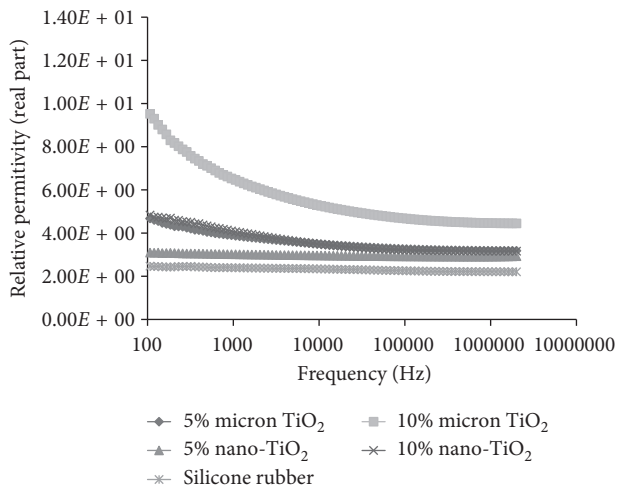


Figure 5: Real portion of relative permittivity at different loading fillers and its size in relation to frequency.

between the nanoparticles and the polymer can lead to a stable interface having fewer free ions and defects, reducing the interfacial polarization in the bulk nanocomposite.

At low frequencies, all free dipolar groups can move and thereby produce a high permittivity. At high frequencies, these groups struggle to move, causing a reduction in permittivity.

Particles of $TiO_2$ have a high ionic polarization due to the presence of $Ti^{4+}$ and $O^{2-}$ ions, increasing the static permittivity. This mechanism is similar for both nano- and microcomposites.

Furthermore, the relative permittivity ($\varepsilon_r$) increases as the concentration of $TiO_2$ increases (Figure 5). Since the individual permittivity of $TiO_2$ is greater than that of the polymer, the latter will have an influence on the permittivity obtained for the nano- and microcomposites.

SEM assessment of the morphology of the nano- and microcomposites structure shows a uniform dispersal of nano- and microparticles in the polymer, confirming the efficiency of the synthesis of these materials process (Figures 6(a) and 6(b)). The morphology of both nano- and microcomposites samples is nearly identical.

### 3.5. Electrical Conductivity as a Function of Particle Size and Concentration.

An improvement in the electrical performance of insulators subjected to pollution conditions is achieved by providing an electrical conductive insulating material [10]. The electrical conductivity of micro- or nanocomposites is conditioned by the formation of a three-dimensional network of particles within the polymer. Two main mechanisms are responsible for the electrical performance of micro- and nanocomposites [23]. The simplest case occurs when there is direct contact between the particles as electrons can be transferred from one particle to another. The second mechanism is the tunnel effect [24] that occurs when there is a thin polymer layer separating two particles. Even if there is no contact between the particles, electron transfer is still possible despite an insulating barrier between the particles. When the potential difference reaches a certain threshold, the electrons are able to jump from one particle to another. For these mechanisms to come into play, it is first necessary that a grid forms within the micro- or nanocomposites. However, various factors influence the presence or absence of this network. Particle concentration, for example, should allow the formation of the network through the entire sample if sufficiently high.

The evolution of the relative permittivity ($\varepsilon_r$) as a function of frequency for both nanocomposites and microcomposites has been assessed for two sizes of particles: 25 nm and 1 $\mu$m.

The formation of an electrical path in micro- or nanocomposites can be determined by measuring the electrical conductivity of these nanocomposites at different concentrations.

Table 1 summarizes the results for the conductivity ($\sigma$) measurements obtained for the nano- and microcomposites. These values were obtained from the measurement of the resistance, using the relationships (2), (3), and (4).

The conductivity of pure SR is $1.21 \times 10^{-5}$. By adding nanoparticles and microparticles, this conductivity increases slightly as $TiO_2$ content is increased (to 5 wt.%), due to the semiconducting properties of $TiO_2$. From this concentration, there is a decrease with excess $TiO_2$ content (10 wt.%) (Figure 7, Table 1). The decrease in conductivity may be due to particle blockage of the conduction path by the $TiO_2$ particles embedded within the polymer [25]. In addition, increasing the concentration of $TiO_2$ for nano- and microcomposites produced a large number of polarons when the coupling of interpolarons became large. This phenomenon led to a severe pinning effect of the polarons that limited their contribution and resulted in reduced conductivity. Moreover, the recovery of $TiO_2$ particles by the polymer prevents contact between the particles, inhibiting the semiconducting properties of $TiO_2$.

## 4. Conclusion

Despite the great interest in nanocomposites, there are, to our knowledge, very few studies addressing the elaboration and the development of nanocomposites for outdoor insulation applications. However, microcomposites have been used in commercial materials and offer a potential use in these outdoor situations.
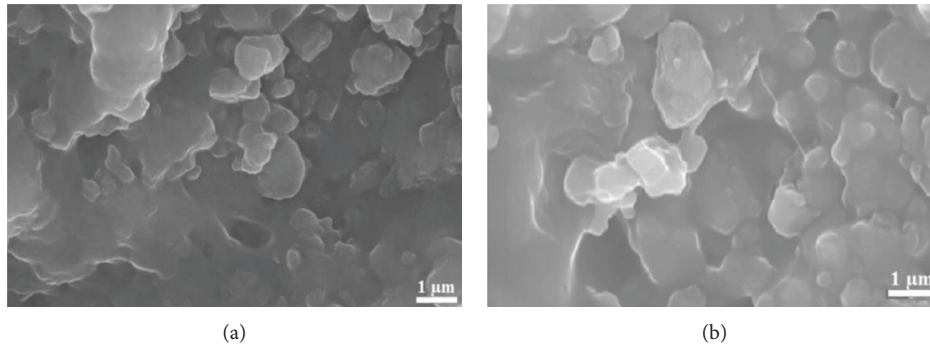
(a)



(b)

FIGURE 6: SEM images of silicone rubber/$TiO_2$. (a) Nanocomposites; (b) microcomposites.

TABLE 1: Conductivity of micro/nanocomposites relative to the concentration of $TiO_2$.

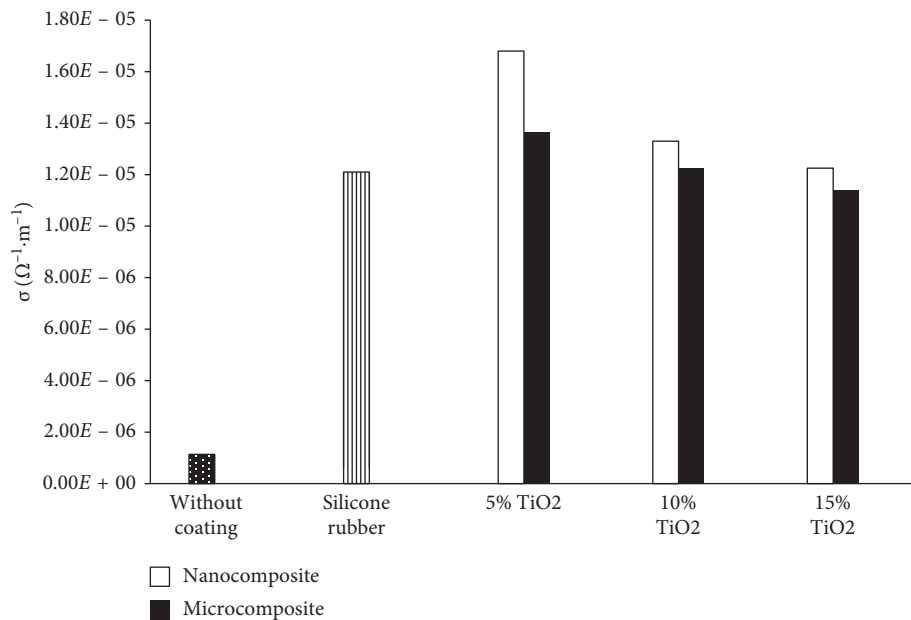| % nano-$TiO_2$ | Conductivity $\sigma$ ($\Omega^{-1} \cdot m^{-1}$) | % micro-$TiO_2$ | Conductivity $\sigma$ ($\Omega^{-1}$ $m^{-1}$) |
|---|---|---|---|
| SR (0%) | $1.21 \times 10^{-5}$ | SR (0%) | $1.21 \times 10^{-5}$ |
| 5 | $1.68 \times 10^{-5}$ | 5 | $1.366 \times 10^{-5}$ |
| 10 | $1.33 \times 10^{-5}$ | 10 | $1.225 \times 10^{-5}$ |

SR: silicone rubber.



FIGURE 7: Evolution of the electrical conductivity of the microcomposites and nanocomposites based on the concentration of $TiO_2$.

In this study, RTV silicone rubber/titanium dioxide nanocomposites and microcomposites were prepared and analyzed at different concentrations of $TiO_2$. We observed that a surface treatment of $TiO_2$ with a surfactant can improve the dispersal of particles in silicone rubber. SEM analysis showed a decrease in particle agglomeration confirming that the surfactant Triton X-100 improves the dispersal of the particles. We investigated the dielectric properties of nano- and microcomposite coatings. For microcomposites, the permittivity tended to increase very strongly in comparison with nanocomposites. This pattern may be due to interfacial polarization that is higher in microcomposite systems. Also, the results showed that relative permittivity increases with increased $TiO_2$ concentrations. Finally, we studied changes in electrical conductivity in relation to particle size and $TiO_2$ concentration. Conductivity increased slightly for the nanocomposites that had a low concentration of $TiO_2$ (5 wt.%) compared to other samples. Conductivity decreased slightly at higher concentrations of $TiO_2$. This decrease can be attributed to the blocking of conduction paths by $TiO_2$ particles embedded in the polymer.

## Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.
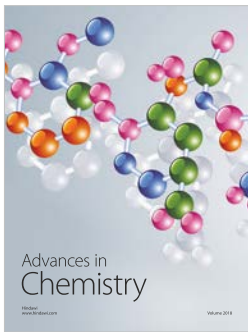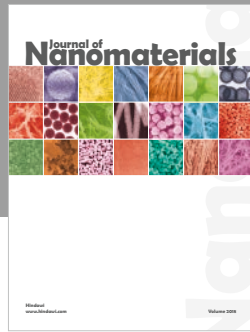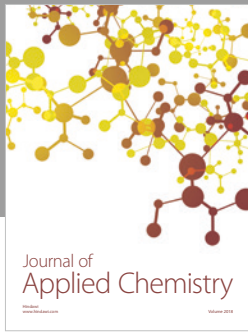
## Conflicts of Interest

## Acknowledgments

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