

Effect of micro/nano-SiO₂ on mechanical, thermal, and electrical properties of silicone rubber, epoxy, and EPDM composites for outdoor electrical insulations

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Abstract: In this paper the influence of micro-sized (3 μm) and nano-sized (10 nm) silica (SiO₂) on mechanical, thermal, and electrical properties of silicon rubber (SiR), epoxy, and ethylene propylene diene monomer (EPDM) composites is presented. The micro- and nano-sized SiO₂ particle-filled SiR, epoxy, and EPDM composites were formulated with 20% microsilica and 5% nanosilica by weight respectively. Among these composites, SiR-SiO₂ amalgamation was performed by mixing using an ultrasonication procedure. Epoxy-SiO₂ was compounded in two steps, i.e. dispersion of fillers and mixing, whereas EPDM-SiO₂ compounding was performed with a two roll mill technique. With the addition of micro/nano-SiO₂, the composites showed enhanced tensile strength of ~ 2.7 MPa, improved hardness, and reduced elongation at break. Further incorporation of micro/nano-sized particles resulted in high thermal stability for SiR-nano composites (SNCs) as compared to epoxy and EPDM composites. Compared to SiR and EPDM composites, epoxy-nano composites showed the highest value of dielectric strength, i.e. 38.8 kV/mm. Meanwhile, the volume and surface resistivity of SNCs were found higher as compared to other investigated samples. Promising properties of SiR nanocomposites among all the investigated samples suggest that their application is more suitable for utilization in outdoor electrical insulations.

Key words: SiR, epoxy, EPDM, microcomposites, nanocomposites

1. Introduction

Due to their light weight, excellent insulation, simple installation and transportation, and outstanding mechanical, thermal, and electrical properties, polymeric insulators are gradually replacing customary ceramic insulators for high voltage (HV) transmission and distribution. Silicon rubber (SiR), ethylene propylene diene monomer (EPDM), acrylic resin, polythene, ethane, nylon, acetone, lignin, polypropene, and Bakelite elements have significant utilization in outdoor insulation applications. In order to enhance their utilization in HV industry, some built-in failures in these materials must be addressed [1–4]. To reduce their intrinsic drawbacks suitable inorganic fillers have been added into the polymer base matrix. Due to their thermal stability and better mechanical and electrical characteristics, SiO₂-composites have been concentrated on to rectify polymer composite properties. The amalgamation of SiO₂ with specific surface area (100–400 m²/g) has considerable impact on the mechanical properties of polymeric materials [5]. Improvement in mechanical, thermal, and

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electrical characteristics of the polymer insulation field is required to enhance its utilization for outdoor applications. It includes elongation at break, hardness, tensile strength, percentage weight loss, dielectric strength, volume, and surface resistivities [6]. Pure SiR, epoxy, and EPDM composites show comparatively little tracking and erosion resistance, weak tensile strength, hardness, lower thermal stability, weak dielectric strength, and lower volume resistivity. During the past decade, much effort has been made to enhance their insulation properties. One approach towards refinement of their characteristics is to reinforce natural or artificial resins by incorporation of micro/nano inorganic oxide fillers (like alumina trihydrate, SiO_2 , zinc oxide, titanium oxide, calcium carbonate, barium titanate), fibers, whiskers, and platelets [7,8]. Sim et al. [9] reported elevated thermal conductivity and thermal pads in SiR- Al_2O_3 /ZnO composites. Plesa et al. [10] reported the influence of SiO_2 , alumina (Al_2O_3), and titania (TiO_2) on the relative permittivity and loss tangent of epoxy resin. Composite properties can be modified due to networked dispersion of filler in the polymer matrix; therefore, it is feasible to enhance material properties at lower filler concentrations as well as reducing the negative impact of higher filler incorporation on additional properties [11]. Thus, amalgamation of 2–5 wt.% nano- SiO_2 into the polymer matrix would be satisfactory for enhancement of electrical properties [12]. Consequently, SiR nanosilica composites at 2.5 wt.% showed improved erosion and tracking resistance compared to microsilica composites [13,14]. Some researchers also reported hybrid (micro-nano) polymer composites [15, 6]. However, the impact of micro- and nano- SiO_2 on polymer properties is not well investigated for use in outdoor insulants.

This work aims to report the impact of micro/nanosilica on prepared composites with improved mechanical, thermal, and electrical properties, which will provide a promising prospective for use as HV outdoor insulants. In this work, the mechanical, thermal, and electrical properties of SiR, epoxy, and EPDM composites blended with micro- and nano-sized SiO_2 particles are investigated and studied.

2. Experimental

2.1. Materials

Epoxy resin (Eposchon) diglycidyl ether of bisphenol A with hardener meta-phenylene was purchased from China (Shanghai LiYi Science & Technology Development). The EPDM used in this work was bought from Exxon Chemical (Belgium). Room-temperature vulcanized silicon rubber (RTV 615), density ~ 0.970 g/mL and thermal conductivity ~ 0.14 W m^{-1} K^{-1} , was provided by Wacker-Chemie (Germany). The nano- SiO_2 with density ~ 2.18 g/mL and thermal conductivity of ~ 1.38 W m^{-1} K^{-1} was purchased from Degussa (USA), whereas micro- SiO_2 was bought from Sigma-Aldrich Chemicals (UK). Other materials like zinc oxide (ZnO), stearic acid, sulfur (S), tetra methyl thiuram tetra sulfide (TMTD), mercaptobenzothiazole (MBT), and dioctyl phthalate (DOP) were purchased from a local market. Different parts of the micro/nanocomposites are schematically described in Figure 1.

2.2. Preparation

Silicon rubber composites with different weight% of silica loading were prepared by following the ASTM-D 1418-10a standard procedure [17] as shown in Figure 2. Development of epoxy- SiO_2 samples included the spreading of filler powder in ethanol until the alcohol was completely evaporated from the mixture. This mixture (filler + epoxy) was then mixed with a hardening agent before putting the mixture in a die for the desired shape. Prepared samples are shown in Figure 3. The preparation of EPDM- SiO_2 composites was carried out by two roll mill technique in accordance with ASTM D-3182-07 [18]. Different materials for the EPDM composite samples were amalgamated according to the standard procedure as described in Figure 4. These composites

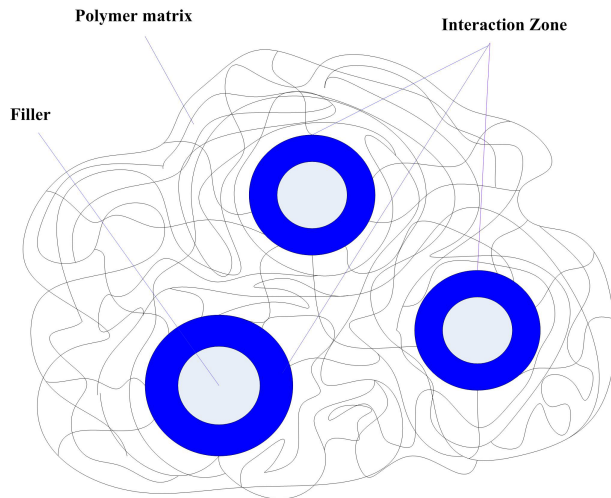


Figure 1. Parts of polymer micro/nanocomposites.

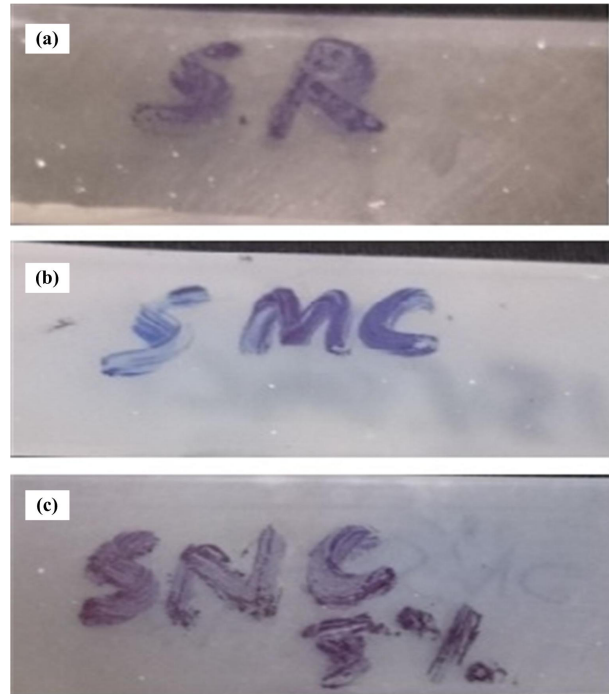


Figure 2. SiR produced samples: (a) neat, (b) micro, and (c) nano.

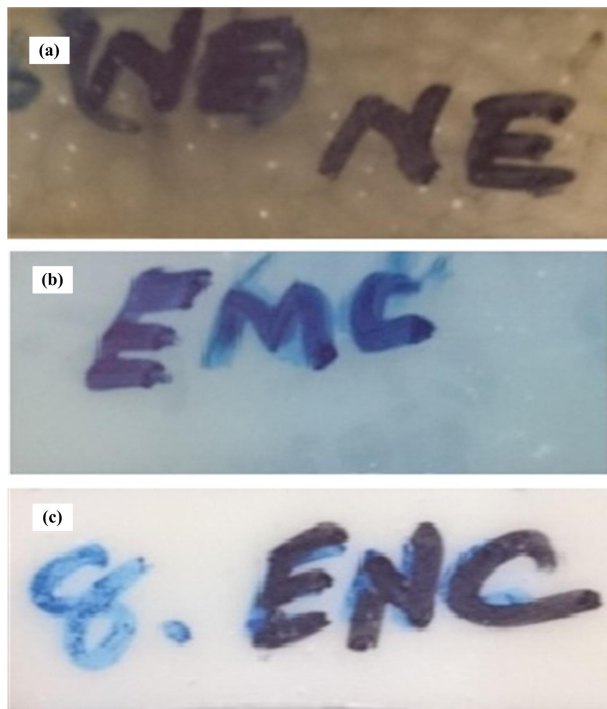


Figure 3. Epoxy produced samples: (a) neat, (b) micro, and (c) nano.

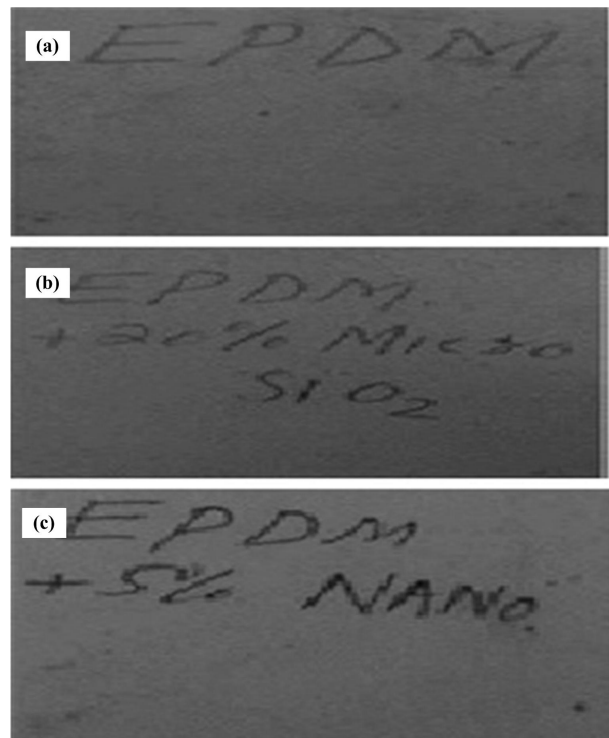


Figure 4. EPDM produced samples: (a) neat, (b) micro, and (c) nano.

were developed in the form of 5-mm-thick and 100-mm-diameter films having different wt.% ratios of silica particles. Mechanical, thermal, and electrical properties of SiR, epoxy, and EPDM composites were compared with neat samples.

2.3. Characterization methods

To study diverse properties of the developed samples, various characterization tests were performed as described below.

2.3.1. Mechanical evaluation

Mechanical testing (tensile strength, elongation at break) of the prepared samples (5 cm × 5 cm × 5 mm) was carried out according to the ASTM D 1708-6a [19] standard procedure by using an Instron 4465 instrument. At ambient temperature (23 °C), 5–10 samples were characterized and tested according to the standard procedure. All experiments were performed @100 mm/min. Likewise, for hardness of micro/nanocomposite samples a durometer Model 408 (type A) was used in accordance with ASTM D2240 [20]. For each composition five to ten samples were used and average results were calculated.

2.3.2. Thermogravimetric analysis (TGA)

To estimate thermal stability as well as weight reduction of composite samples TGA analysis was carried out using a TGA-Q50 TA instrument (USA). The measurements were conducted on 20 mg of samples of each composite at a rate of 20 °C/min from 0 °C to 1000 °C. Weight loss as a consequence of temperature rise was recorded for both pure and impure samples.

2.3.3. Electrical characteristics

Dielectric strength tests were performed according to ASTM D-149-13 [21] on a HV instrument (CS2674A, NC Instrument Co. Ltd.) having spherical electrodes of 2.5 cm in diameter. All readings were obtained using a high AC voltage generator varying from zero to breakdown value (@ 2.5 kV/s) according to the standard procedure. The ASTM D257-05 standard procedure was employed to compute volume and surface resistivities [22]. For each test operation five to ten samples were used and average results were recorded.

3. Results and discussion

3.1. Mechanical properties

Mechanical properties such as tensile strength, elongation at break, and hardness of SiR, EPOXY, and EPDM composites depend on the type of filler, loading, and bond strength with the polymer matrix [23]. These properties are reinforced and influenced by the amalgamation of SiO₂ particles. The obtained results are presented in Tables 1, 2, and 3, respectively. It can be observed that the tensile strength of all samples is improved by addition of micro-SiO₂ and nano-SiO₂ as summarized in Table 1. This improvement is 71.25% (i.e. from 1.11 to 1.9 MPa) for EPDM-microsilica, while it is 83.99% (i.e. from 1.11 to 2.04 MPa) for EPDM-nanosilica composites. For epoxy microsilica the increase in tensile strength is 33.3% (i.e. from 1.2 to 1.6 MPa), and it is 125% (i.e. from 1.2 to 2.7 MPa) in the case of epoxy nanosilica. Likewise, SiR-microsilica showed 80% (i.e. from 0.5 to 0.9 MPa) improvement in tensile properties as compared to neat samples, whereas compared to SiR-micro-SiO₂, the SiR-nanosilica composites showed 220% (i.e. from 0.5 to 1.6 MPa) improved tensile

properties. From tensile strength results of all produced samples it is inferred that with the decrease in particle size the tensile strength is increased, as was expected both for micro- and nanosilica particles. This was due to the strong H-bonding among OH-groups in the base matrix and silanol (Si-OH)-groups on the SiO₂ surface, which restricts chain mobility and consequently enhances cross-linking (tensile strength). Another reason is that the concentration of Si-OH groups on the SiO₂ surface and the particles' uniform dispersion in the polymer matrix and interaction zone (Figure 1) between silica particles and base rubber has a significant impact on mechanical properties. This distance (interaction zone) decreases with the decrease in particle size [24]. Among all prepared composites, the interface distance between microsilica and the polymer base is higher than that of nanocomposites. The interaction between nanosilica and the polymer matrix as well as the concentration of silanol groups on the SiO₂ surface is higher than that of microsilica; therefore, microsilica samples showed slightly lower tensile results than nanosilica samples, as summarized in Table 1. Hence, it was found that among all the blends, EPDM, epoxy and SiR nano-SiO₂ samples exhibited higher tensile strength compared with microsilica composites.

Table 1. Tensile strength (MPa) of SiR, epoxy and EPDM composites.

Sample	Neat	Micro	Nano
SiR	0.5	0.9	1.6
Epoxy	1.2	1.6	2.7
EPDM	1.11	1.9	2.04

Table 2. Elongation at break (% strain) of SiR, epoxy, and EPDM composites.

Sample	Neat	Micro	Nano
SiR	142	123	118
Epoxy	125	115	110
EPDM	244.5	223.4	220.2

Table 3. Hardness (shore A) of SiR, epoxy, and EPDM composites.

Sample	Neat	Micro	Nano
SiR	48	54	56
Epoxy	54	56	58
EPDM	48.4	57.3	58.1

The elongation at break of SiR, epoxy, and EPDM samples decreases with the incorporation of micro- and nano-SiO₂, as shown in Table 2. This decrease varies from 13.3% to 20.3% for SiR micro- and nanosamples. Likewise, epoxy and EPDM samples showed the same declining behavior, which varied from 8% to 12% and 8.6% to 9.93% for their micro- and nanosamples respectively. The reduction in elongation at break is due to improved cross-linking, strong hydrogen bonding, and reduced chain mobility for nanocomposites as compared to microcomposites. This improvement in cross-linking is due to reduction of interfiller distance with decrease in particle size, as depicted in Table 2.

Another physical property, the hardness of prepared samples, is also increased by incorporation of micro/nanosilica particles. Such enhancement is from 12.5% to 16.6% for SiR micro- and nanosamples. For epoxy and EPDM micro/nanocomposites, it varies from 3.7% to 7.4% and 18.3% to 20%, respectively, as illustrated in Table 3. From the hardness results it is obvious that hardness is increased both for micro- and nanocomposites, but nanosilica imparts greater hardness to the polymer matrix than microsilica samples. This is attributed to the large number of Si-O-Si groups and higher molecular interaction among nanosilica and base polymers compared with the poorer molecular interaction in micro-sized samples. Hence, due to the higher interaction and smaller particle size, cross-linking in nanosamples is greater than that in microcomposites, which leads to improved hardness of nanosilica samples. Hence, from all prepared samples, it is inferred that nanosamples exhibited greater hardness compared with micro-sized and neat samples.

3.2. Thermal properties

The influence of micro/nano-SiO₂ on the thermal stability of SiR, epoxy, and EPDM composites was analyzed by TGA. Thermal conductivity can be calculated by Eq. (1):

$$\lambda = [Q / (A \times (T_1 - T_2))] \times t, \tag{1}$$

where T₁ and T₂ are the sample top and bottom surface temperatures, Q is the heat strength, A is the sample contact area, t is the sample thickness, and λ is the thermal conductivity [25]. Figure 5 shows 10% and 50% weight loss as well as the polymer decomposition temperature (PDT) of EPDM samples. It can be seen that the temperature of 10% mass-loss of neat EPDM is 290 °C, whereas by adding microsilica and nanosilica the 10% mass-loss temperature (1% and 2.5%) is a little higher compared to pure EPDM samples. The same behavior is also exhibited at the 50% mass-loss temperature (1.51% and 2.7%) both for micro- and nanosamples. Samples with micro- and nanosilica displayed improved PDT, which varied from 1.51% to 2.7%, respectively. It appears that nano-SiO₂ provides better stability than micro-sized and neat EPDM samples, which is primarily due to SiO₂'s hygroscopic nature and immobility in the surrounding area of nano-SiO₂ particles. Similarly, TGA results of epoxy microsilica and nanosilica samples are compared in Figure 6. The 10% and 50% weight-loss temperature of neat epoxy is 157 °C and 392 °C, respectively, whereas its residual weight seen at 600 °C is 13%. Epoxy samples filled with micro-SiO₂ and nano-SiO₂ display improved thermal stability in the case of 10% weight-loss (i.e. 99.3% for microsilica and 58.5% for nanosilica) and 50% weight-loss (i.e. 4.5% for microsilica and 3% for nanosilica) as well as final residual weight (i.e. 21% for microsilica and 20% for nanosilica). Hence, we conclude that the highest mass reduction is exhibited in epoxy nanosilica samples (Figure 6). It seems that reinforcing particles in lower percentages has no significant effect on composite characteristics as compared to pure latex [26].

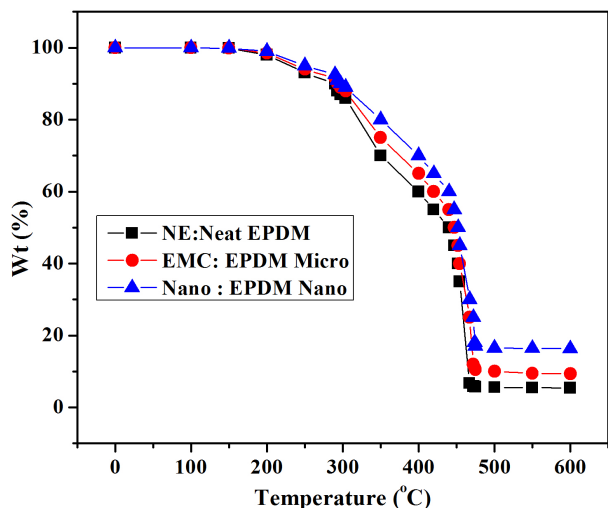


Figure 5. EPDM TGA results: weight loss as a function of temperature.

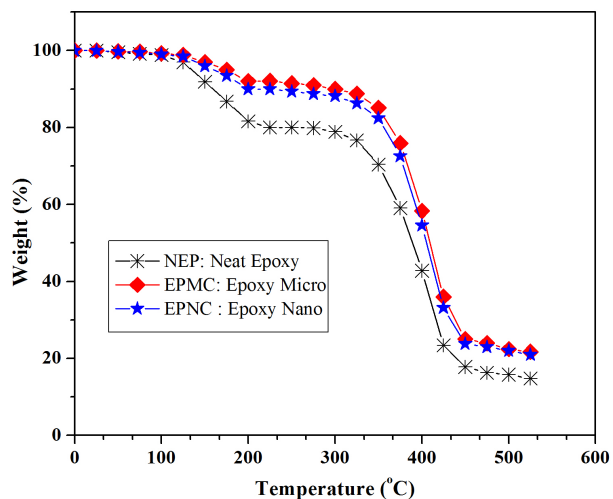


Figure 6. TGA results: weight loss vs. temperatures of epoxy.

From the TGA results of SiR samples (Figure 7) it is obvious that micro/nano-SiO₂ also grants thermal substantiality in the case of 10% weight-loss (i.e. 7.8% for microsilica and 8.2% for nanosilica) and 50% weight-loss (i.e. 68% for microsilica and 90% for nanosilica) as well as final residual weight (i.e. 32 % for pure SiR, 60%

for microsilica, and 73% for nanosilica). The smaller the gradient, the higher the remaining mass, as revealed from TGA graphs. Hence, overall greater residual mass is seen for SiR nanosilica samples compared with microsilica samples, attributed to the substantial amount of Si-OH groups in nano-SiO₂. Thus, from thermal studies of the developed samples it is concluded that, by amalgamation of nano-SiO₂ particles in the SiR matrix, substantial enhancement in degradation temperature, higher residual weights, and thermal stableness can be obtained.

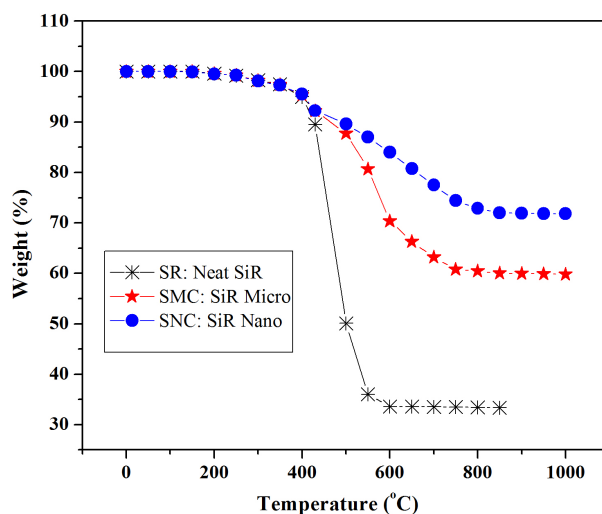


Figure 7. TGA results: weight loss vs. temperatures of SiR composites.

3.3. Electrical properties

Insulation failure is the disastrous collapse of dielectrics due to outside stress following leakage current flow as well as morphological degradations. Research on polymer insulant decomposition for outdoor applications has been a subject of interest. Due to molecular configurations, reinforcing particles, and inherent flaws, the dielectric strength of insulants varies even among the same polymer composites [27]. The influence of micro/nanosilica on breakdown strength (BDS) of the prepared samples is imperceptible. Breakup experiments were conducted on prepared samples of EPDM, epoxy, and SiR to explore the influence of micro/nano-SiO₂. The relation between dielectric BDS and volume resistivity is given by Eq. (2):

$$BDS = 2.83 \log[\rho_v / (\Omega_r \times \tan \delta)] + 15.24, \tag{2}$$

where ρ_v is volume resistivity, Ω_r is relative permittivity, and $\tan \delta$ is loss tangent [28].

Breakdown strengths of the investigated composites are summarized in Table 4. Micro-SiO₂ and nano-SiO₂ particles displayed improved dielectric BDS for SiR (i.e. 7.1% for microsilica and 12.7% for nanosilica), epoxy (i.e. 10% for microsilica and 21% for nanosilica), and EPDM (i.e. 7.9% for microsilica and 17% for nanosilica) composites. The improvement in BDS may be due to large numbers of Si-O-Si bonds in SiR nanocomposites than microcomposites and/or it may be due to stronger Si-O bonds than C-C bonds of other polymers. Additionally, due to the incorporation of nano-SiO₂ into the polymer matrix, the energy gap between valance and conduction bands increases, which accordingly enhances the BDS and electrical resistivity. Furthermore, due to homogeneous dispersion of nanosilica in the base matrix, the interparticle distance decreases and it behaves as a barrier for applied voltage stress, which leads to improved BDS [29].

Table 4. Dielectric strength (kV/mm) of SiR, epoxy, and EPDM composites.

Sample	Neat	Micro	Nano
SiR	19.7	21.1	22.2
Epoxy	32	35.2	38.8
EPDM	19	20.5	22.3

The volume and surface resistivities of micro/nano-SiO₂ blended EPDM, epoxy, and SiR composites are presented in Tables 5 and 6, respectively. Superficial impedance is the opposition to leakage current on the insulator surface, whereas volume resistance is the opposition to leakage current throughout the whole insulator structure. Thus, greater superficial and volume resistances cause less leaking current as well as inferior conductivity of polymer composites [30].

Table 5. Volume resistivity (Ω cm) of SiR, epoxy, and EPDM composites.

Sample	Neat	Micro	Nano
SiR	1.8 E+15	7.1 E+15	8.9 E+15
Epoxy	5.7 E+13	7.2 E+13	1.5 E+14
EPDM	3.0 E+14	3.4 E+14	3.6 E+14

Table 6. Surface resistivity (Ω cm) of SiR, epoxy, and EPDM composites.

Sample	Neat	Micro	Nano
SiR	1.2 E+15	6.3 E+15	7.7 E+15
Epoxy	2.3 E+13	4.4 E+13	7.2 E+14
EPDM	2.1 E+14	2.3 E+14	2.5 E+14

Enhancement in both volume and surface resistivities of all the investigated samples was observed due to incorporation of micro and nano-SiO₂ particles. However, nano-SiO₂ filler-based composites showed higher volume and surface resistivities than microcomposites. It was found that decreasing the silica particle size from micro to nano increased the volume and surface resistivities of prepared samples, as shown in Tables 5 and 6. Among all investigated composites the SiR blends showed higher resistivities. This may be due to stronger bonds between silicone and oxygen atoms and increased interactions between SiO₂ and the base polymer. Due to smaller size, nanosilica exhibited the highest surface-to-volume ratio as compared to micro and neat samples, which leads to increased chain immobilization and improved cross-linking and molecular network structure. As a consequence, this enhances their resistivities [29].

4. Conclusion

In this work the preparation and effects of micro- and nano-sized SiO₂ on mechanical, thermal, and electrical properties of SiR, epoxy, and EPDM composites were studied for their utilization as outdoor insulants. The mechanical results of EPDM-SiO₂ (neat, micro, and nano) composites revealed that EPDM nano-SiO₂ samples demonstrated better characteristics compared with epoxy and SiR samples. Such improvement was 71.25% for EPDM-microsilica and 83.99% for EPDM-nanosilica. From thermal results, silicon rubber nano-SiO₂ (i.e. 73% final residual weight) composites showed superior performance as compared to EPDM and epoxy composites. Likewise, electric characteristics of the developed samples demonstrated outstanding dielectric features, but increase in dielectric strength was observed in epoxy nanosamples (38.8 kV/mm), whereas silicon rubber nanocomposites performed well in volume and surface resistivities as compared to other samples. Overall characterization reports of the developed micro- and nano-SiO₂ composites validate the superiority as well as the homogeneous dispersal of filler particles in the prepared samples, verifying them as fine competitors for HV insulator applications.

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