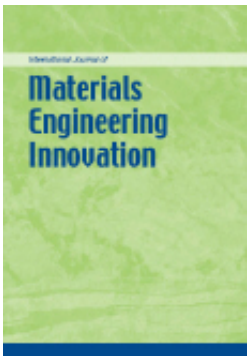

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Investigations into surface erosion characteristics and thermal stability of epoxy-based ZnO nanocomposites

Mihir N. Velani, Ritesh R. Patel

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Investigations into surface erosion characteristics and thermal stability of epoxy-based ZnO nanocomposites

Mihir N. Velani

Gujarat Technological University,
Ahmedabad-382424, Gujarat, India
and
Department of Electrical Engineering,
RK University,
Rajkot, Gujarat, India
Email: mihir.zalawad@gmail.com

Ritesh R. Patel*

Department of Electrical Engineering,
G.H. Patel College of Engineering and Technology,
Charutar Vidya Mandal University,
Vallabh Vidhyanagar-388120, Gujarat, India
Email: r2patel@yahoo.com
*Corresponding author

Abstract: The study examines the surface erosion characteristics and thermal stability of epoxy-based ZnO nanocomposites in various configurations. These include the effect of filler loading, co-loading of nano-micro fillers, and the synthesis process. The surface erosion experiments were conducted as per IEC60112, and the results were analysed using scanning electron microscopy (SEM). Thermogravimetric analysis (TGA) was carried to determine the materials' thermal stability. It was observed that with the high filler contents, ZnO nanocomposites offered smaller craters and discontinuous conducting channels, impeding the bulk erosion of epoxy. In addition, the composite exhibited stronger interphase with the epoxy matrix delaying thermal degradation at lower temperatures. The co-loading of nano-micro fillers reduced the mobility of epoxy resins and resulted in superior discharge resistance and thermal stability than the micro composite. The nanocomposite synthesised with heated ZnO particles in a solvent-free approach showed thermal decomposition beyond 250°C, which was superior among all.

Keywords: filler loading; nano-micro fillers; synthesis process; thermal decomposition; tracking resistance.

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Biographical notes: Mihir N. Velani received his BE in Electrical Engineering from Sardar Patel University, Vallabh Vidyanagar, Anand, India in 2008 and MTech in 2013 with specialisation in Electrical Power Systems from Nirma

University, Gujarat. He is currently pursuing his PhD from Gujarat Technological University, Gujarat. His research areas comprise electro-thermal analysis of power apparatus, conduction and breakdown studies of dielectric materials and characterisation of polymeric nanodielectric materials.

Ritesh R. Patel received his Bachelor's in Electrical Engineering from North Gujarat University, Master's in Electrical Power Engineering from M.S. University of Baroda and PhD from Indian Institute of Technology Kanpur. His extensive research areas cover electrical characterisation of insulating materials for high voltage applications, modelling of composite materials, condition monitoring of electrical apparatus, breakdown characterisation of gaseous, solid and liquid insulation systems, modelling of EHV power systems, and stability analysis of power systems.

1 Introduction

Polymeric insulators have drawn more attention than ever because of their excellent insulation and thermal characteristics. In the energy industry, various polymer materials, including thermoplastics, thermosets, glass, and natural fibres, have been employed because of their superior performance and long lifespan in extreme environments (Durai, 2014). Epoxy resins with appropriate nano and micro-organic particles have gradually replaced polymeric insulators and found their applications in various power apparatuses and devices (Banerjee et al., 2021; Cherney, 2013). Also, machining procedures like drilling and grinding have found a use for epoxy-based composites (Jamal and Sandeep, 2016). The performance of the nanocomposite materials chiefly depends on the combination of base and host materials, nano-micro fillers, and the synthesis process (Thomas and Alun, 2017; Toshikatshu and Takahiro, 2017). Therefore, it is challenging to achieve a perfect blend of various properties. Many configurations of base and host materials have been worked out to improve electrical, mechanical, and thermal properties. However, the nearly ideal combination is being tested. Our computational study (Velani and Patel, 2021, 2020) compared ZnO nanofillers to Al_2O_2, BeO, BN, SiO_2, and TiO_2 and showed they have good electrical and thermal properties. Concerning the findings, the current work examines the epoxy-based ZnO nanocomposites' resistance to surface erosion and thermal stability.

Significant work has been presented in improving resistance to electrical treeing (Wan et al., 2014) and thermal steadiness of epoxy (Mathioudakis et al., 2014; Noor et al., 2020; Farimah et al., 2020) and silicone-based polymer nanocomposites (Nazir et al., 2018). Also, different techniques have been applied to quantify the surface degradation of polymeric insulation under electric stresses (Chintan and Ritesh, 2022). Suchitra et al. (2018) derived a correlation of free space length and surface energy of glass epoxy composites incorporating nano-micro-ATH fillers with the help of transmission electron microscopy (TEM) along with a comparative tracking index for outdoor applications. Electrical tree growth and partial discharge in epoxy exposed to AC and DC voltages were explored by Ibrahim et al. (2018). Divya and Preetha (2021) performed simulation studies on epoxy-based nanocomposites inserting alumina, zinc oxide and titania nanofillers. They found that when epoxy nanocomposites are stimulated by DC current, the nanoparticles significantly slow down the growth of the trees.

Verginadis et al. (2019) studied surface discharges and flashover characteristics in the presence of water droplets. It was derived that the higher filler contents reduce flashover voltages possibly due to particle agglomeration and interaction zones. Further, the lower flashover voltages were caused by increased filler contents, probably due to particle agglomeration and interaction zones. Zhenlian et al. (2016) analysed surface resistance of fluorinated epoxy resin to corona discharges in sulphur hexafluoride (SF_6) gas through attenuated total reflectance infrared (ATR-IR) spectroscopy and scanning electron microscopy (SEM). They showed that SF_6 gas would cause chemical modification leading to changes in surface morphology and properties. In the presence of SF_6, nitrogen, dry air, and epoxy, Herie et al. (2020) performed discharge tests on neat epoxy in a non-uniform electric field. They found that surface insulation performance can be improved in an SF_6-free environment. Du and Yamano (2005) performed DC resistance measurements to tracking on a range of polymeric materials, including glass-cloth epoxy resin, using the IEC 60112 approach. Also, Ispirli et al. (2018) simulated and experimented with tracking phenomena for polyurethane resin based on IEC 60112 test standard. Xing et al. (2019) explored surface tracking of MgO/Epoxy nanocomposites according to IEC 60112. The SEM results signified that the increased MgO contents enhanced anti-tracking performance. Regarding surface erosion studies as per IEC 60112, ZnO nanoparticles have yet to be explored much.

Takari et al. (2021) performed molecular dynamic simulation and thermal characterisation on epoxy-based TiO_2 and SiO_2 nanocomposites. The thermogravimetric analysis (TGA) curves showed higher thermal stability due to the addition of the nanoparticles but did not discuss the affecting factors. Qilong et al. (2022) carried out experimental and finite element-based computational study to reduce electric field and power loss. The experiments were conducted on epoxy-based aluminium nitride (AIN) filled nano-micro composites. According to the findings, adding the right amount of filler (20 weight percent micro-AlN and 5 weight percent nano-AlN) to epoxy micro-nano hybrid composites will lower power loss and the electric field at 150°C and 1 MHz when operating in an AC environment. Weiyu et al. (2021) adopted a surface modification approach for epoxy/SiO_2 composites to improve thermal performances. They pre-grated epoxy chains on surface of nano-SiO_2 to form cross-linked networks to improve dispersion. Using nano-ZnO that had been silane-modified, Suresh et al. (2017) observed the thermal and dielectric properties of epoxy. The samples were subjected to kinetic analysis, differential scanning calorimetry (DSC), and TGA, but only a cursory examination of the thermal stability was done. Yasmine et al. (2020) investigated the impact of modified ZnO on the thermal characteristics of epoxy resin. The TGA plots demonstrated that adding functionalised ZnO delayed thermal degradation by raising degradation temperatures. However, they still have to investigate the impact of filler loading.

The presented research stands out because it evaluates surface discharge resistance and thermal stability according to IEC 60112 (International Electrotechnical Commission, 2003) and ASTM E1131 (ASTM International, 2010), respectively, in differently synthesised epoxy-based ZnO nanocomposites. Among the configurations are:

1 the influence of filler loading

2 the co-loading of nano-micro-fillers

3 the synthesis processes.

2 Materials and specimen preparation

2.1 Materials

The epoxy (Huntsman Araldite CY230-1, density 1.19 g/cm^3 at 23°C) and hardener (Aradur HY951, density 0.95 g/cm^3 at 23°C) were blended to synthesise the epoxy specimens. For each specimen, the weight ratio of epoxy to hardener was 10:1. The ZnO particles were purchased from Sigma-Aldrich, USA, with a density of 5.68 g/cm^3 at 22°C. The nanofillers' and microfillers' average particle sizes are 80–100 nm and 5 μm, respectively. The specimens in appropriate proportionate in various configurations were synthesised. These configurations include varying the nanofillers' percentage, combining epoxy and nanofillers, and treating the nanofillers. The ultrasonication process was used to prepare the epoxy-based ZnO nanocomposites.

2.2 Specimen preparation

2.2.1 Specimen preparation using an ultrasound probe sonicator

The appropriate proportional ZnO nanofillers were added to 100 ml of methanol (0.792 g/cm^3) as a solvent and manually churned for 10 minutes in a beaker. The mixture was then successfully sonicated for 20 minutes at 20 kHz using a 12 mm diameter acoustic probe (Ultrasonic Probe Sonicator, 750-Watt, Frontline, India). To avoid overheating and excessive methanol evaporation, the mixture was housed in an ice bowl and sonicated in an ON-OFF mode (5 min ON, 5 min OFF). The methanol-filler solution was then supplied with 100 ml of epoxy, which was manually churned for 10 minutes. The resultant mixture was sonicated for an additional 30 minutes. This solution was heated on a hot plate (Anlon, India) up to 78°C to evaporate methanol entirely. The integrant weights were monitored continually throughout the heating process. In a vacuum desiccator, the mixture was degassed until it reached room temperature. The mixture was combined with the hardener, then poured into acrylic moulds (105 mm × 215 × mm × 3 mm), and allowed to cure for 24 hours at room temperature. All specimens were placed in a hot air oven for 24 hours at 90°C to hasten the cross-linking of epoxy resin, and they were then allowed to cool to room temperature. The specimens were cut into the suitable sizes to conduct the necessary tests.

The combinations of nano-micro fillers have been selected based on a round-robin study (Thomas and Alun, 2017). The nano-fillers are mixed with shear force into the epoxy resin in the first step to disperse them. The nano-filler-dispersed epoxy resin and the micro-fillers are combined in the second step. The remaining steps were carried out as previously mentioned.

2.2.2 Specimen preparation with preheated ZnO fillers

In the hot air oven, the requisite proportional (2 wt%) ZnO nanofillers were heated for 24 hours at 150°C. The epoxy was then infused with the heated ZnO nanofillers, as was described in the previous section.

2.2.3 Specimen preparation with bath sonicator

The epoxy was mixed with the needed quantity of ZnO nanofillers. A bath sonicator from Unimarks Lab, India, operating at 40 kHz and 180 W, was used to sonicate the mixture for 45 minutes at 55°C.

The prepared specimens in various configurations are listed in Table 1.

Table 1 Specimen configurations

Specimen specifics	*Specimen code*
Neat epoxy	EP
Epoxy with 2 wt% Nano ZnO fillers with solvent and probe sonicated	E2NZS
Epoxy with 4 wt% Nano ZnO fillers with solvent and probe sonicated	E4NZS
Epoxy with 6 wt% Nano ZnO fillers with solvent and probe sonicated	E6NZS
Epoxy with 2 wt% Nano ZnO fillers without solvent and probe sonicated	E2NZ
Epoxy with 8 wt% Micro ZnO without solvent and probe sonicated	E8MZ
Epoxy with 2 wt% Nano ZnO and 8 wt% Micro ZnO without solvent and probe sonicated	E2N8MZ
Epoxy with preheated 2 wt% Nano ZnO without solvent and probe sonicated	E2HNZ
Epoxy with preheated 2 wt% Nano ZnO without solvent and bath sonicated	E2NZB

3 Experimental

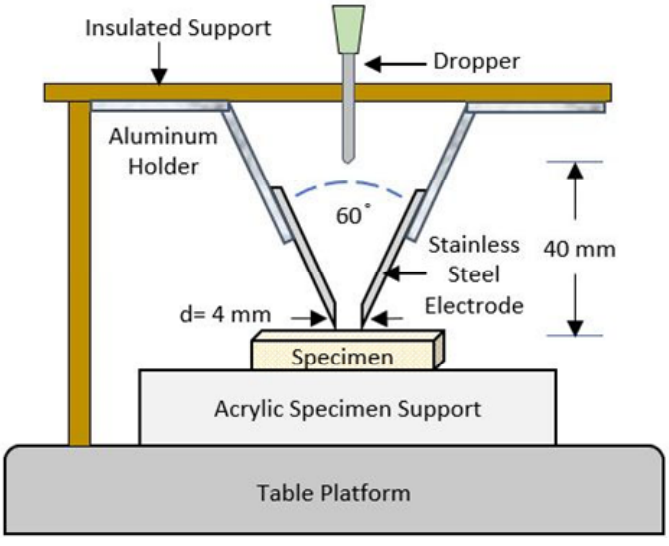
3.1 Surface erosion test

Solid insulations should impede the progressive formation of conduction channels, commonly known as tracking and electric erosion. The purpose of the test is to observe the performance of the specimens against the combined effects of electric stress and electrolytic contamination; however, determining the comparative tracking indices is not the motive of the study.

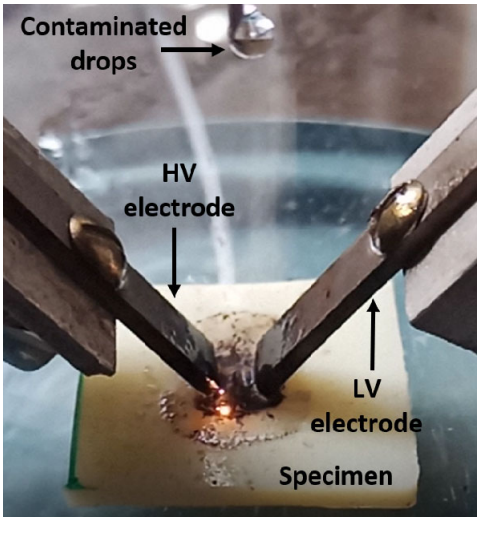
The specimens have been tested according to the IEC60112 standards. However, the study does not derive comparative tracing indices and the test results are not directly suitable for the evaluation of safe creepage distances when designing electrical insulation. The setup is displayed in Figure 1. The stainless-steel electrodes are 4 mm apart and oriented at a 60° angle. The test solution was prepared with 0.1% reagent grade anhydrous ammonium chloride ($NHCl_4$) in the deionised water (EMPLURA®, Merck). It was ensured that the solution's conductivity was not more than 1 mS/m at 23°C. The dropper was arranged to fall on the specimen nearly centrally between the electrodes from a height of 40 mm. The specimens were cut into 2.5 cm × 2.5 cm and further cut to 1.0 cm × 1.0 cm for the microscopic analysis. The tests were conducted for the test period of 50 drops at an interval of 30 seconds. The ambient temperature was maintained between 23°C to 28°C. The electrodes were energised by a 1-phase sinusoidal source of 325 V_{rms} at 50 Hz. There were no occurrences of persistent flame between the electrodes; however, the discharge current ranged from 0.02–0.14 A. When the contaminated drops fall on the specimens under electric stress, the electrical discharges erode the surface by producing carbonised fillers. The carbonised fillers form conducting channels helping the

degradation of the material. The eroded specimens are shown in Figure 2. The erosion is significant at the edge of the electrodes because the charge density is larger there.

Figure 1 Surface erosion test setup, (a) schematic and (b) experimental (see online version for colours)



(a)



(b)

The scanning electron micrographs were captured on JEOL SEM IT300 at the magnification of 500 µm (×50) at three different spots (2 mm, 1 mm, and 0.5 mm away from the HV electrode) on the eroded specimens, indicated in Figure 3.

Figure 2 Eroded specimens (see online version for colours)

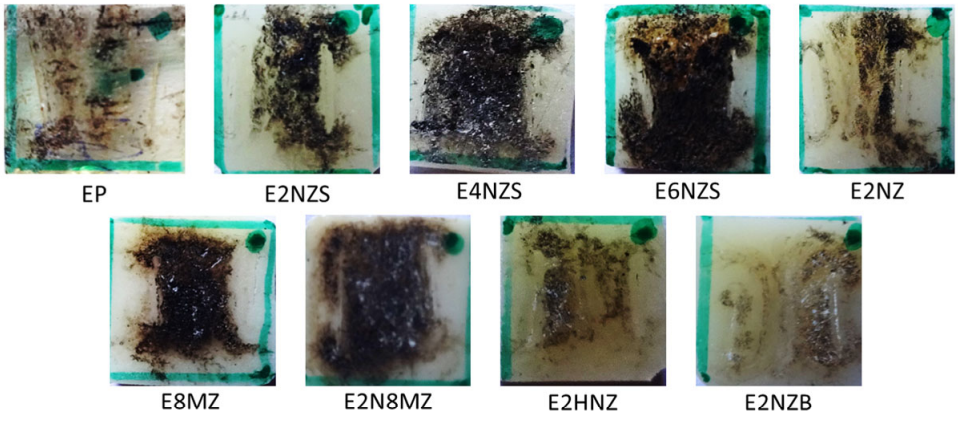
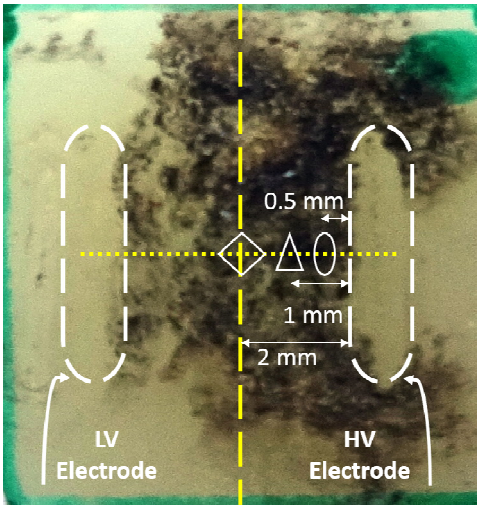


Figure 3 Micrographs captured at three different spots (see online version for colours)



For conciseness, all the micro scans have been displayed for only EP. All the SEM images are displayed 0.5 mm away from the HV electrode for the rest specimens. The greyer/blackish regions point to the impact craters that have endured erosion, whereas the whiter/snowier ones denote islands where degradation has occurred in lower or more subtle proportions.

3.2 Thermogravimetric analysis

The insulators are exposed to high voltage field stresses and extreme weather conditions, leading to material properties' degradation over time. As a result, the insulator gradually develops heat spots at lower leakage currents may lead to the short-term or long-term degradation. Hence, it is desired that the solid insulators should offer excellent thermal properties ensuring thermal endurance.

The epoxy polymers are categorised by low thermal stability and high flammability. It is imperative to know the inherent temperature-dependent properties of epoxy-based composites as they prompt molecular changes beyond its glass transition temperature (T_g) and degradation temperatures (T_{on} and T_{off}).

A TGA is a thermo-analytical method to examine the thermal stability of a material and its fraction of unstable constituents by observing the weight change occurring at a constant heat rate. The tests were performed on TGA 4000 by Perkin Elmer according to ASTM E1131. The specimens of about 2 mg were analysed in a nitrogen environment. The temperature ranged from 30°C to 300°C at 10°C per minute. The TGA thermogram generally displays high volatility first weight loss, low volatility second weight loss, combustion of compounds, and residues. Only one-stage decomposition can be seen in all the TGA plots.

4 Results and discussion

4.1 Surface erosion test

4.1.1 Effect of filler loading

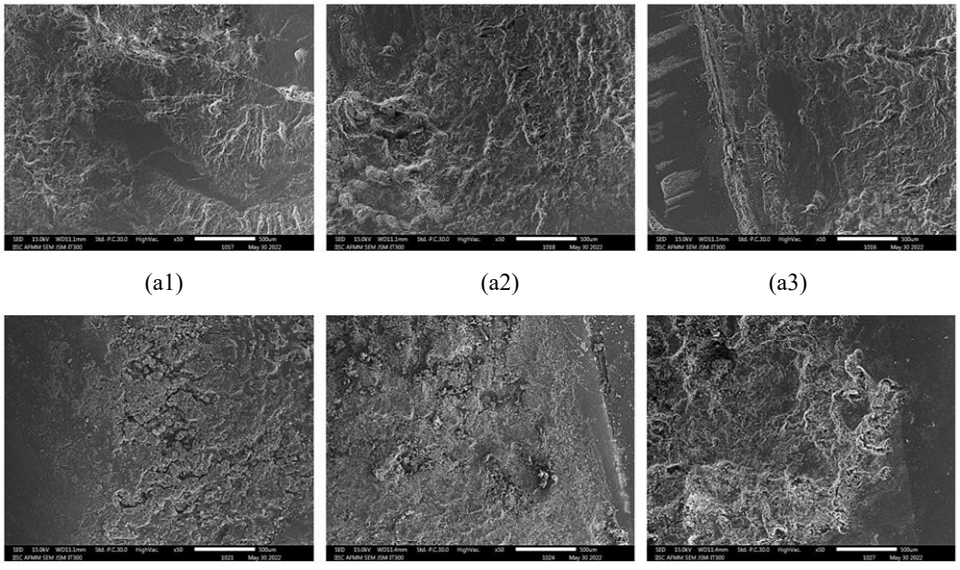
Figure 4 shows that in the neat epoxy-EP, the erosion is significant near the electrode surface and can be spotted as continuous tracking channels compared to the other two cases (1 mm and 2 mm away from the electrode surface, respectively). Also, the degraded channels are thick and continuous near the electrode. The formation of numerous large craters here signifies that the epoxy specimen has been almost eroded. The craters oversee directing the stream, which causes greater erosion of the material. On the opposite side, the formation of craters reduces with the increase in wt% of ZnO fillers. Notably, the craters become smaller and scattered and are comparatively less in E6NZS. The nanoparticles exfoliate from the epoxy matrix against the electrical discharges enduring the erosion of the base material. The interaction of the nanoparticles with the polymer matrix greatly influences the cumulative impact of the particles. For MgO/epoxy nanocomposites, similar findings were described by Xing et al. (2019) to explain surface tracking performance; Kangning et al. (2018) also obtained identical results for MgO/epoxy.

4.1.2 Effect of nano-micro co-loading

It is evident from Figure 5 that the erosion is remarkably significant in E8MZ compared to E2NZ and E2N8MZ composites. The tracking channels appear to have reduced in the nano-filled specimen compared to the only unfilled specimen-EP. The specimen-E2N8MZ with nano-micro fillers has restricted eroded volume of the neat epoxy exfoliating from the surface. The superior performance deduces the increased interaction between nano and micro fillers, infusing the unfilled gaps in the epoxy matrix. The fillers of different sizes might function as pinning to confine the mobility of polymer chains. The performance of polymer composites is governed by the weight percentage and dispersion of nano- or micro-sized particles into the underlying polymer matrix (Banerjee et al., 2021; Xiaoyang et al., 2022). Our findings are congruent with Parimal et al. (2008), who observed similar outcomes for nano and micro Al_2O_3/epoxy nanocomposites

exposed to corona discharges. Iyer et al. (2012) also concluded that co-filled epoxy improves electrical discharge resistance.

Figure 4 Effect of filler loading: SEM images of (a1), (a2), (a3) EP (b) E2NZS (c) E4NZS and (d) E6NZS



(a1) (a2) (a3)

(b) (c) (d)

4.1.3 Effect of synthesis process

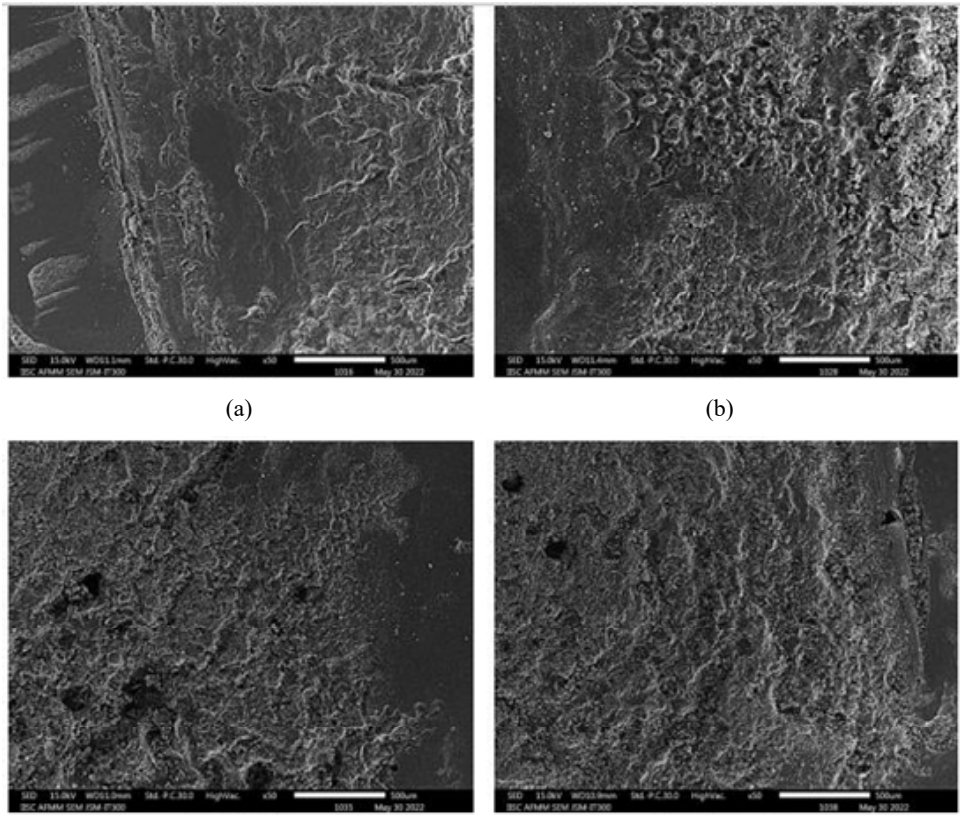
The synthesis process has affected the performance of the specimens against electrical discharges (Chen et al., 2021). The striking feature of the comparative analysis is that the specimen-E2HNZ prepared with heated ZnO fillers exhibited supercilious performance. Figure 6 illustrates that in the E2HNZ, the tracking channels are thinner and parted. These degraded channels have become outspread and more continuous in E2NZS and E2NZB and are ruinous in E2NZ. This erosion is postulated due to:

1 absorbed atmospheric moisture

2 particle agglomeration.

The studies have reported that the electrical properties of the polymers are substantially affected due to the dipolar and conductive nature of water uptake or moisture ingress (Yan et al., 2018; Nilsson et al., 2017). The inclusion of nanofillers forms numerous tracks affecting moisture migration in nanocomposites (Hui et al., 2013). It is featured that even E2NZB surfaced thinner track paths compared to E2NZ; the bath sonication being continuous in operation at 40 kHz, had generated more heat and would have removed moisture from the particles. The additional moisture would have accessed the interfacial surface of ZnO particles in E2NZ, creating moisture pockets responsible for the local conducting channels. Besides, agglomerated ZnO particles are responsible for the free volume in the E2NZ and have exposed the surface, eroding a greater volume of the epoxy.

Figure 5 Effect of nano-micro co-loading: SEM images of (a) EP (b) E2NZ (c) E8MZ and (d) E2N8MZ



(a) (b)

(c) (d)

4.2 Thermogravimetric analysis

4.2.1 Effect of filler loading

It is observed from Figure 7 that EP undergoes the first step of weight loss at about 122°C and continues to degrade to the residual polymer at about 227°C. The weight loss also includes the evaporation of water, solvent, and combustion of amalgams, such as carbon black or other fillers, and leave ash of inorganic fillers. During the transition from high volatility to low volatility, epoxy retained almost 84% mass along with all 2, 4, and 6 wt% composition. It specifies that weight loss mechanism did not change considerably with the addition of fillers. However, the degradation process was delayed with the filler loading. The degradation temperatures for all the specimens are indicated as T_{on} and T_{off} and are shown in Table 2. All the specimen began to decompose at 119–125°C and entirely decompose at 227–256°C.

Figure 6 Effect of synthesis process: SEM images of (a) EP (b) E2NZS (c) E2NZ (d) E2HNZ and (e) E2NZB

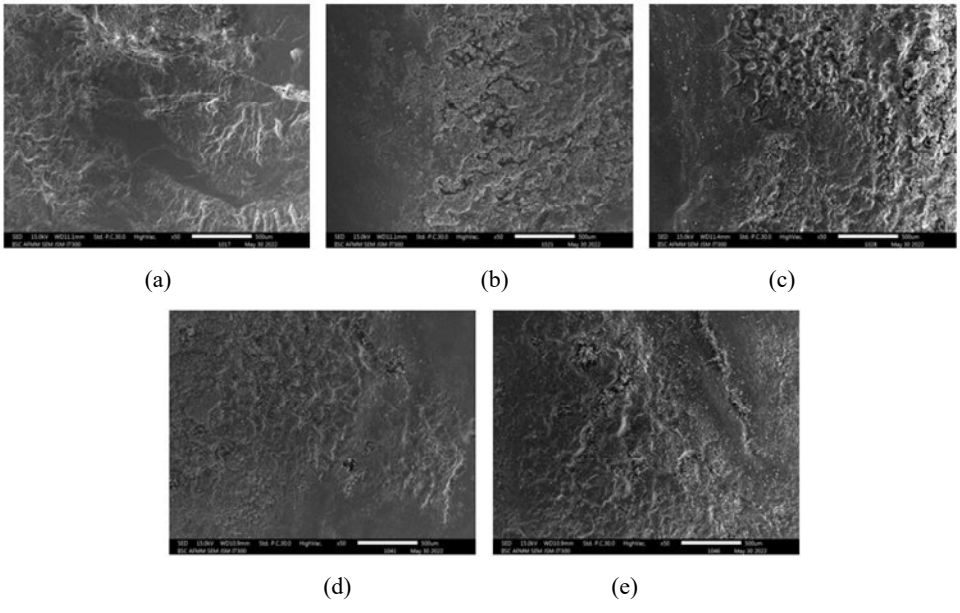
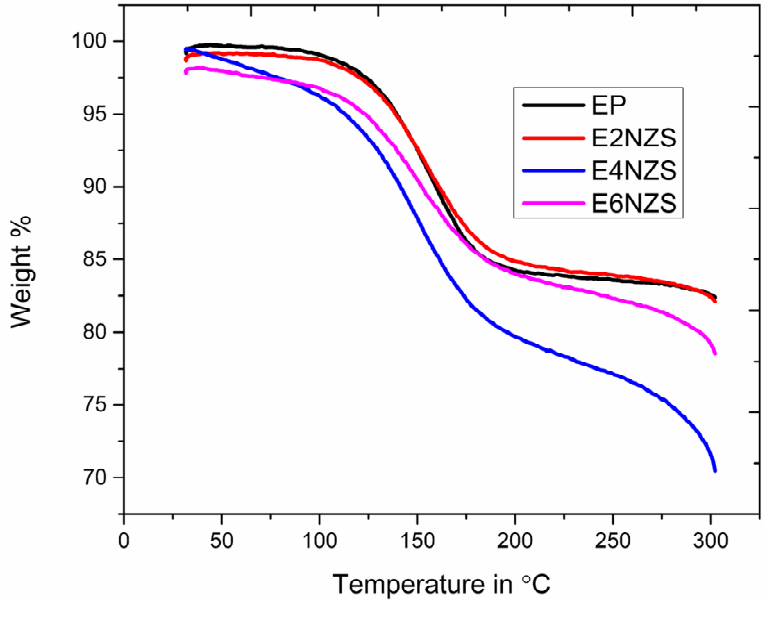


Figure 7 Effect of filler loading: TGA plots (see online version for colours)

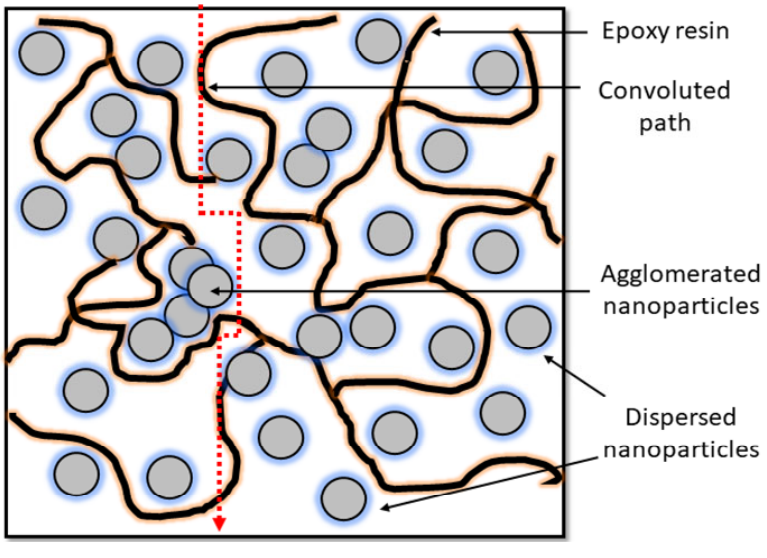


It is worth noting that the complete decomposition of the epoxy is delayed to 256°C with the increase in ZnO content, i.e., E6NZS. This shows that ZnO fillers exhibit stronger interphase with the epoxy matrix. The presence of the ZnO particles in the specimens impedes the motility of epoxy chains by creating convoluted paths as shown in Figure 8. Azeez et al. (2013) studied similar developments for epoxy-clay nanocomposites. Besides, Shettar et al. (2019) showed close results for nano clay-epoxy under different hygrothermal aging conditions.

Table 2 Degradation temperatures

Specimen	*Degradation temperature Ton (°C)*	*Degradation temperature Toff (°C)*
EP	119.43	227.27
E2NZS	120.70	236.36
E4NZS	124.00	239.40
E6NZS	124.60	256.06
E2NZ	125.75	236.36
E8MZ	124.24	237.87
E2N8MZ	122.72	243.93
E2HNZ	122.70	253.00
E2NZB	124.19	236.36

Figure 8 Convoluted path in nanocomposites (see online version for colours)



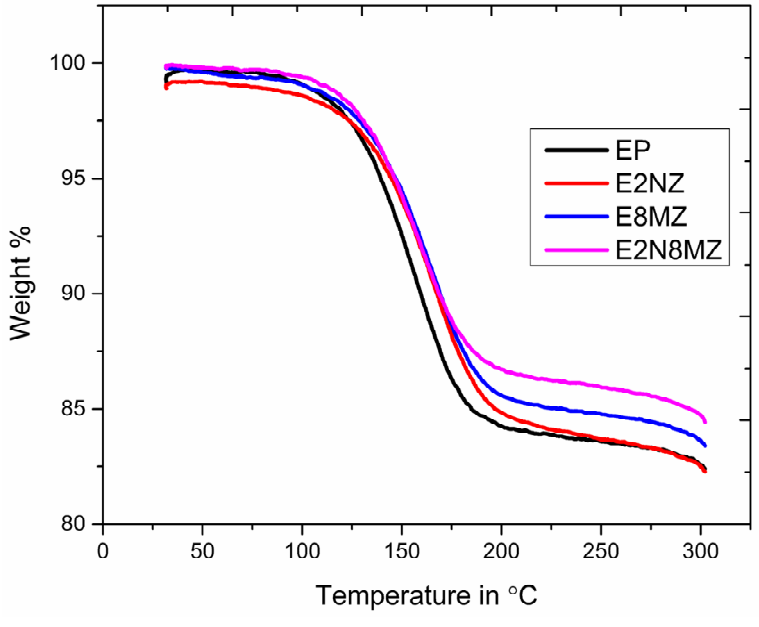
4.2.2 Effect of nano-micro co-loading

The nanocomposites were analysed for nano, micro, and nano-micro ZnO fillers-E2NZ, E8MZ, and E2N8MZ. The epoxy-nano/micro mixtures were sonicated in the probe sonicator without solvent. It is observed from Figure 9 that the sample E2NZ incurs a

weight loss of 15.17% till the low volatility step, whereas the sample E8MZ and E2N8MZ run up with 14.66% and 13.70%, respectively.

The specimens E8MZ and E2NZ with 8 wt% micro fillers (< 5 μm) and 2 wt% nanofillers (<100 nm), respectively, introduce a higher surface area and, interestingly, offer a solid interfacial adhesion with the epoxy matrix. The convoluted paths created by nano-micro fillers (E2N8MZ) resist the heat access in the epoxy resins. The reduction in % weight loss signifies that the dispersion of the fillers in the epoxy matrix leads to stronger interphase with the base material. It is also observed that the nanocomposites are relatively more stable than micro companions mostly due to stimulative effect on cross-linking between fillers and the base material (Muthamma et al., 2021; Ghosh et al., 2012). In addition, the nano-micro composition deferrals the complete decomposition of the composites to about 244°C, roughly 5–7°C, more than E2NZ and E8MZ composites.

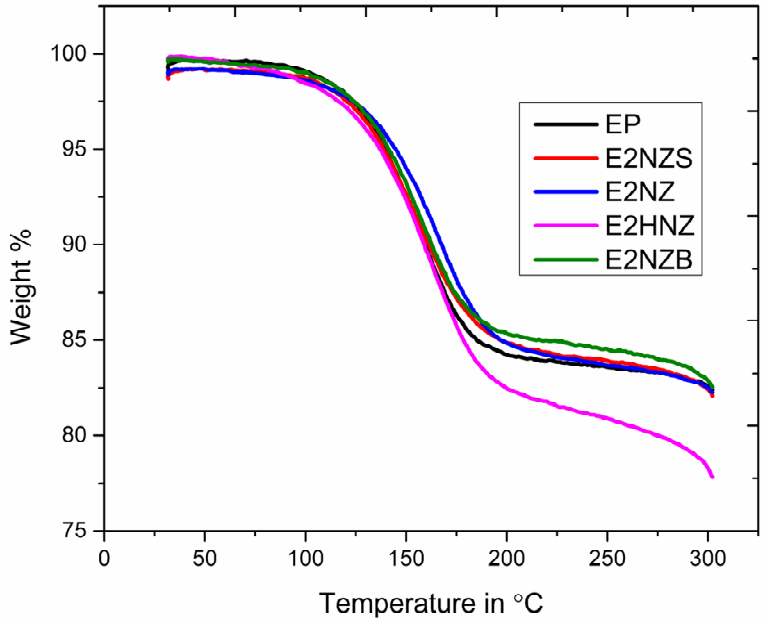
Figure 9 Effect of nano-micro co-loading: TGA plots (see online version for colours)



4.2.3 Effect of synthesis process

The synthesis process plays a critical role in the performance of nanocomposites under various conditions (Landge et al., 2018). The test results (Figure 10 and Table 2) have been compared for E2NZS, E2NZ, E2HNZ, and E2NZB. The specimen composed of heated ZnO fillers mixed in the epoxy with a probe sonicator exhibited increased thermal stability at T_{off} – 253°C. The heated ZnO fillers at 150°C for 24 hours removed the absorbed moisture leading to the enhanced crosslinking with the epoxy resins. Moreover, the absence of the solvent avoids the sedimentation of ZnO particles in the epoxy matrix [as the density of the solvent is lower (0.79 g/cm^3) than that of the ZnO particles (5.61 g/cm^3)]. However, the weight is about 18.74% signifies the improved bonding between the epoxy matrix and the filler particles. The E2NZS, E2NZ, and E2NZB showed nearly similar performance in all thermogravimetric aspects.

Figure 10 Effect of synthesis process: TGA plots (see online version for colours)



5 Conclusions

The epoxy-based ZnO nanocomposites were prepared in different constitutions. The surface erosion tests were conducted following IEC60112 at 325 V_{rms} in the presence of NH_4Cl for 50 drops. The qualitative analysis of the eroded specimens was carried out using SEM. The thermal stability was studied through TGA at a rate of 10°C per minute from 30°C to 300°C. The following conclusions can be drawn from this study:

1 The nanocomposite with higher filler contents (6 wt%) has a noticeably improved resistance to surface erosion. It prevented the loss of epoxy weight by preventing the formation of craters and conducting channels in a more significant chunk. Further, at this proportionate of the fillers, the more potent interface between the epoxy matrix and nanoparticles delayed the thermal decomposition at 256°C, whereas the neat epoxy disintegrated at almost 227°C.

2 The co-loading of nano-micro fillers enhanced cross-linking with the base material. As a result, the mobility of the epoxy resin was constrained, preventing material deterioration from electrical discharges. Additionally, compared to composites with only nano and micro fillers, the blended composite delays thermal runaway by 5–7°C.

3 The nanocomposite synthesised with heated ZnO nanofillers in a solvent-free environment made the tracking channels smaller and fragmented. In terms of thermal stability, it also fared better than the other three compositions. The complete decomposition was deferred at 253°C, 30°C higher than the neat epoxy, and roughly 17°C higher than the two different formulations, i.e., E2NZS, E2NZ, and E2NZB.

Considering all the discussions, the epoxy composite filled with nano and micro ZnO fillers enhances discharge erosion resistance and thermal stability. The presented study can further determine the threshold limit of the filler contents, suitable proportionate filler size, and in situ-based synthesis approaches.

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