

Original Article

Development of high performance granite fine fly dust particle reinforced epoxy composites: structure, thermal, mechanical, surface and high voltage breakdown strength properties



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ABSTRACT

The granite waste generated from the granite cutting and polishing industries contributes to lot of environmental contamination and health hazards. In the present work, an attempt has been made to assess the method to minimise environmental problems associated with granite dust pollution to the possible extent by converting the granite dust waste into value added high voltage electrical insulator products. Two types of epoxy matrices comprising bisphenol-A diglycidyl ether-phenalkamine (ER-1) and bisphenol-A diglycidyl ether-methyl hexahydrophthalic anhydride (ER-2) separately reinforced with varying weight percentages of (20, 40, 60, 80 and 100 wt%) of granite dust. The matrices and composites obtained were characterized for their structural, morphology, hydrophobic properties, thermal stability, mechanical strength, thermal conductivity, thermal resistivity, electrical resistivity, breakdown voltage and dielectric strength behaviour using appropriate analytical testing methods. Data resulted from different studies, it was inferred that the composites prepared using ER-2 possess better properties than those of ER-1. The composites with 100 wt % of cutting waste granite fly dust reinforced ER-2 possesses the higher values of electrical surface resistivity (5.22 \times 10¹² Ω), and electrical volume resistivity (5.60 \times 10¹³ Ω) than those of neat ER-2 matrix (surface resistivity $4.82 \times 10^9 \Omega$ and volume resistivity $2.48 \times 10^{13} \Omega$). The breakdown voltage and dielectric strength of ER-2 matrix were increased from 28 ±1 kV and 14 \pm 0.5 kV/mm to 34 \pm 1 kV and 17 \pm 0.5 kV/mm respectively, when reinforced with

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100 wt % granite dust. The value of breakdown voltage was increased by 22% for 100 wt% granite dust reinforced composites when compared to that of neat epoxy matrix.
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1. Introduction

Granite is a type of closely packed natural stone, which is widely used in the field of civil engineering sector such as wall covering panels, floors, shelves, sinks, basins, fireplace mantle, table tops and monuments. An advancement of granite cutting and polishing processing industries generate massive wastes. It is well known that the millions of tons of granite waste generated from the granite cutting and polishing industries around the world. These granite waste causes lot of environmental problems by contaminating land, water and air, which in-turn contribute to health hazards [1]. The effective method of utilization of granite fly dust particulates for the development of new industrial products will mitigate the environmental problem to certain extent.

Ceramic materials were utilized widely in medium and high voltage electrical insulation applications. The ceramic materials possess better electrical resistance and have long lifetime. However, heavy weight, high energy required for material production and less performance in prolusion contaminated environments are the limitation with the ceramic materials. Polymer insulator is a counter part of ceramic insulator. Light weight, easy processes, economic design, and hydrophobic nature are main advantages of polymer insulator. Usually, silicone rubber, ethylene propylene diene monomer, epoxy resin and ethylene vinyl acetate, polytetrafluoroethylene, polyolefin elastomer and highdensity polyethylene, polyurethane are used for low voltage and high voltage insulation applications. These polymeric materials contribute to an excellent hydrophobicity, good mechanical strength, excellent breakdown strength and higher volume resistivity. The polymer insulator systems are developed in the form of hybrid composites. Usually, metal oxides and non-metal oxides are used as a reinforcing material for high-voltage insulation materials such as silica, alumina, titanium oxide, zinc oxide, magnesium oxide, boron nitride, aluminium nitride, silicon nitride and silicon carbide. Previously our research group have been developed bio-silica hybrid benzoxazine composites for high voltage insulation application [2].

It is well known that the different epoxy based composites were utilised for various applications such as, coatings [3], friction materials [4—6], adhesives [7], military [8], building materials [9], electronic circuit boards [10], electrical insulation, etc. Majorly, the silica reinforced epoxy composites have been used for high voltage insulation application [11]. In this work two type of composite systems were prepared for different environment. The ER-1 is considered as a tough polymer system, which was derived from DGEBA and phenalkamine [12]. The phenalkamine is a cardanol based hardener [13] which contains a long aliphatic chain in *meta* position of phenol and triethylenetetraamine placed at ortho position of cardanol [14]. The ER-1 is a room temperature curable system. The ER-2 is considered as a brittle nature polymer system, which is derived from DGEBA and methyl hexahydrophthalic anhydride [15]. The ER-2 is considered as high temperature curable system, which will cures at about 100 °C [16]. The ER-2 is most widely used polymer system for high voltage insulation application [17].

The use granite fly dust is expected to replace the many of the traditional fillers [18–20], clay [21], and other conventional materials in the field of composite manufacturing sectors [22]. The granite is a silica rich stone, also contains of some metal oxides such as aluminium oxide, iron oxide, calcium oxide, magnesium oxide and trace of sodium and potassium oxides [23]. The silica rich granite dust material is a counter part of commercial silica fillers. Silica fillers are extensively used in the manufacture of polymer composites [24], in order to improve mechanical strength, hydrophobic nature, electrical resistivity, thermal stability and resistance to chemicals. In the recent past researchers and scientist around the world are concentrating towards converting waste materials to wealth and to eliminate the environmental problems associated with granite dust pollution [25-31]. Previously, granite dust was utilised in the field of ceramic, polymer composites for building materials, coating material and brake pads material applications. Gupta et al. reported on impact on mechanical properties of cement sand mortar containing waste granite powder [32]. Chen et al. inferred that the incorporation of granite polishing waste to reduce sand and cement contents and to improve the performance of resulting mortar [33]. Sadek et al. studied the reusing of marble and granite powders for making self-compacting concrete [25]. Lopes et al. studied on optimization of variation of particle size and pH of granite dust in paints [1]. Sugozu et al. developed granite dust based brake pad materials. Even though some of the earlier studies reported the utilization of granite dust for different industrial applications, still lot of new avenues to be developed in order to utilize granite dust more efficiently and economically to produce composite materials to widen the area of applications.

With this in view, an attempt has been made in the present work to produce high voltage electrical insulation granite fly dust waste and different epoxy systems. The two types of composites were prepared using varied weight percentages of granite fly dust with bisphenol-A diglycidyl etherphenalkamine (ER-1) and bisphenol-A diglycidyl ethermethyl hexahydrophthalic anhydride (ER-2) matrices. The composites developed were studied for their structural morphology, thermal stability, mechanical behaviour, thermal conductivity, electrical resistivity and break down voltage using appropriate analytical test methods. Data obtained from different studies are discussed and reported.



Scheme I – Hocess of granite my line dust remoted epoxy compos

2. Experimental section

2.1. Materials

Cutting waste granite fly fine dust was received from A Blue Hill Granites India Private Limited, Coimbatore, India. Bisphenol-A diglycidyl ether (DGEBA - epoxy resin)) and phenalkamine (phk) epoxy hardener were obtained from Roto polymers ltd, Chennai, India. Methyl hexahydrophthalic anhydride (mhhpa) was received from Hindustan speciality chemicals India ltd. Gujarat, India.

2.2. Preparation of granite fly dust filled epoxy composites

The collected granite fly fine dust were sieved with 38 μ m mesh to remove the bulk particle and other dust materials. Further, it was washed with distilled water and treated with 0.1 N hydrochloric acid to reduce the conductive metal oxide.



ab remoreca poly(bacbit phenaikamine)

Scheme 2 - Preparation of granite fly dust filled ER-1 (poly(DGEBA-phk)) composites.

Further, washed with 0.1 N sodium hydroxide solution for surface activation and subsequently washed with water, until reach the neutral pH. Further, the washed granite fly dust was dried at 80 °C for overnight (Scheme 1). The 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite fly dust was homogenously mixed separately with DGEBA epoxy resin and phenalkamine system (1:0.5) and are cured at room temperature. The neat epoxy matrix and homogeneously granite dust reinforced system were separately poured into the mould with required dimensions. Further the curing system was post cured at 60 °C for 2 h (Scheme 2).

Similarly, the 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite fly dust was homogenously mixed separately with DGEBA epoxy resin and methyl hexahydrophthalic anhydride (mhhpa) system (1:1). Then, the curing process of ER-2 system was accelerated by adding 2 g of N,N-dimethyl aniline (Scheme 3). The granite fly dust reinforced epoxy resin system was poured into the required size of the Teflon coated aluminium mould and cured at 100 °C for 3 h. Then, the completely cured samples were separated from mould and placed for characterisation.

2.3. Measurements

The functional group of materials were studied by FTIR spectral technique (SHIMADZU FTIR Spectrophotometer). The thermal degradation behaviour of the granite flay dust filled epoxy composites was analysed by Thermogravimetric technique (HITACHI STA 700 series). Tensile strength and flexural strength of neat epoxy matrix and granite dust reinforced epoxy composites were determined through INSTRON 8801 instrument as per ASTM-D638 and ASTM-D790 standards respectively. The thermal conductivity and thermal resistivity of neat epoxy matrix and granite dust reinforced epoxy composites were analysed by heat flow meter (HFM). Surface

resistivity and volume resistivity behaviour were studied using Fischer Elektronik TE50, Germany. Water contact angle of granite dust reinforced epoxy composites was analysed by goniometer. The breakdown voltage (BDV) of composites was measured by 100 kV high voltage transformer setup. The dielectric strength of granite dust reinforced epoxy composites was calculated from the values of breakdown voltage (BDV).

3. Results and discussion

The granite fly dust reinforced polymer composites were subjected to different analysis such as molecular structure, morphology, thermal stability, mechanical behaviour, thermal conductivity, hydrophobic behaviour and electrical resistivity, and high voltage insulation behaviour and the data obtained are discussed and reported.

3.1. FTIR analysis

The structural behavior of neat and granite fly dust reinforced epoxy composite materials were analyzed by FTIR and SEM-EDX spectral techniques. The functional groups of the neat epoxy matrix and granite fly dust reinforced epoxy composite materials were analyzed by FTIR spectroscopy and are presented in Fig. 1. The peaks observed at 2933 cm⁻¹ and 2861 cm⁻¹ were confirm the symmetric and asymmetric stretching vibration of the $-CH_2$ - (methylene) groups respectively. The peak noticed at 1725 cm⁻¹ represents the stretching vibrations of carbonyl (-CO-) and 1178 cm⁻¹ infers the presents of -C-O- link of the ester groups respectively. The stretching vibration of -C-N- and ether (-C-O-) linkages were observed at 1233 cm⁻¹ and 1034 cm⁻¹, respectively. The stretching vibration bands appeared at 1077 cm⁻¹ and



Scheme 3 – Preparation of granite fly dust (GD) filled ER-2 (poly(DGEBA-mhhpa)) composites.



Fig. 1 — FT IR spectra of neat epoxy matrix and granite fly dust (GD) reinforced epoxy composites of (a) ER-1 (poly(DGEBAphk)) and (b) ER-2 (poly(DGEBA-mhhpa)).

980 cm⁻¹ were confirm the presence of Si–O–Si (siloxane) and -Si-OH (silanol) linkages in the composites.

3.2. Microscopy analysis

Further, the homogeneous dispersion of granite fly dust reinforced epoxy composites were studied using scanning electron microscopy (SEM) and transmission electron microscope (TEM) techniques. The raw granite dust, neat epoxy matrix and 100 wt% granite fly dust reinforced ER-1 and 100 wt % granite fly dust reinforced ER-2 composites were taken as a representative sample for the morphological analysis through SEM and TEM analysis. The SEM and TEM images of raw granite dust and the fractured surface of neat polymer matrix and 100 wt% granite fly dust reinforced ER-1 and 100 wt% granite fly dust reinforced ER-2 composites are shown in Figs. 2 and 3 respectively. From the SEM and TEM images, it was observed that the raw granite fine dust particles are obtained as spherical and amorphous particles. As seen from SEM image of the granite fly fine dust reinforced epoxy composites, the dust particles show uniform distribution and compact interface structures, which are no obvious clusters or voids in the composites. The compact interface and homogeneous distribution could be explained by the intra-molecular interaction between granite dust particle and epoxy resin matrix. The interface between granite dust particle and epoxy matrix looks more indistinct, which is attributed to the occurrence of covalent bonding interaction and contribute to the strong and efficient compatibility.

In addition, the elemental analysis of granite dust, neat epoxy matrix, 100 wt% granite fly dust reinforced ER-1 and 100 wt% granite fly dust reinforced ER-2 were analysed by EDX spectral technique (Fig. 5) and elemental mapping (Fig. 4), and the results obtained are presented in Figs. 4 and 5. From the EDX spectrum and elemental mapping (Figs. 4 and 5a), granite fly dust waste contains major element of SiO₂ and trace of other oxides viz. Al, Ti, Fe, Cu, Cr and Ca. Fig. 2c, d and 5b ascertain that the smooth neat epoxy matrix possesses only carbon, oxygen and nitrogen atoms. The homogeneous dispersion of granite flay dust present in ER-1 and ER-2 composites were noticed from Fig. 5c and d. From Fig. 5c and d, it is noticed that the elements above mentioned are obtained in composite materials.

3.3. XPS analysis

XPS analysis is employed to study of the functional groups and are shown in Fig. 6. For the case of Si, the peaks appeared at around 98 eV and 162 eV were conform the Si 2p, Si 2s. The peaks noticed at around 125 eV and 75 eV were ascertain the Al 2s and Al 2p, respectively. The spectra of C1s noticed at 286 eV, corresponds to C–Si, C–C, C–O, C–N and C=O, respectively. In the case of N element, a peak appeared in the rage of 395 eV infers the N 2s of N–C group. The peak noticed at 455 eV represents the Ti 2p. A peak obtained at 533 eV indicates the O 1s of O–C, O–Si, O=C, O–N and Oxides of metals (O-M). The peak observed at 592 eV confirms the Cr 2p. For the Fe element, a peak appeared at around 705 eV ascertains the Fe 2p. The peak obtained at around 945 eV represents the Cu 2p.

3.4. Thermal behaviour

The thermal behaviour of granite fly dust reinforced epoxy composites namely thermal stability, thermal conductivity and thermal resistivity nature obtained are discussed below.

3.5. Thermal stability

The thermal stability of neat epoxy matrix and granite dust reinforced ER-1 and ER-2 composites were studied by thermogravimetric analysis (TGA) under N_2 environment with the order of increment of 20 °C/min. The recorded TGA data of neat epoxy matrix and granite fly dust reinforced ER-1 and ER-



Fig. 2 – FESEM micrograph of (a), (b) granite fly dust particle, (c) neat ER-1, (d) neat ER-2, (e) 100 wt% GD reinforced ER-1 and (f) 100 wt% granite fly dust reinforced ER-2 composites.



Fig. 3 – TEM micrograph of (a–c) granite fly dust particle (d–f) 100 wt% GD reinforced ER-1 and (g–i) 100 wt% granite fly dust reinforced ER-2 composites.



Fig. 4 - Elemental mapping of a granite fly dust particle.

2 are presented in Fig. 7 and Table 1. Thermal stability of polymer composites is an important parameter for the device fabrication, to predicts, its service temperature. The behaviour of stability was measured using the weight loss at every moment observed for ER-1 composites is presented in Fig. 7a. The 5% weight loss ($T_{5\%}$) of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of ER-1 composites were noticed at

around 309 °C and that there was no significant changes noticed. The 10% weight loss ($T_{10\%}$) of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of ER-1 composites were noticed at 325 °C, 326 °C, 327 °C, 329 °C, °C, 329 °C and 331 °C, respectively. The maximum degradation temperature (T_{max}) of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of ER-1 composites were observed at 364 °C, 369 °C, 370 °C, 372 °C,



Fig. 5 – EDX spectra of (a) granite fly dust, (b) neat epoxy matrix (c) 100 wt% granite fly dust reinforced ER-1. and (d) 100 wt% granite fly dust reinforced ER-2 composites.



Fig. 6 - XPS spectra of granite dust (GD) reinforced epoxy composites.



Fig. 7 – TGA thermogram of neat epoxy matrix and granite fly dust (GD) reinforced epoxy composites of (a) ER-1 and (b) ER-2.

 $374 \,^{\circ}$ C and $375 \,^{\circ}$ C, respectively and that of char yield (residual) percentage at 850 $\,^{\circ}$ C was noticed at 6%, 24%, 31%, 36%, 43% and 49%, respectively.

In the case of ER-2 composites (Fig. 7b and Table 1), the 5% weight loss of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of ER-2composites were observed at 270 °C, 314 °C, 319 °C, 326 °C, 338 °C and 350 °C, respectively. The maximum degradation temperature noticed at 371 °C, 383 °C, 385 °C, 387 °C, 389 °C, and 391 °C, respectively. The char yield was obtained at 10%, 23%, 33%, 46%, 55% and 64%, respectively. Thermal stability of both granite fly dust reinforced ER1 and ER-2 composites was increased with increasing concentration of granite fly fine dust particle. The ER-2 composites possess higher stability in thermal environment than that of ER-1 composites, because of the presence of thermally stable skeletons of methylhexahydro phthalic anhydride curative. Among the composites, 100 wt% of granite fly dust reinforced with ER-2 matrix have better stability under thermal

| epoxy composites. | | | | | |
|-------------------------------|-------------------|--------------------------|--------------------------|--------------------------------|-----------------------------|
| | Thermal stability | | | | |
| Sample | T₅% (°C) | T _{10%} (°C) | T _{max} (°C) | Char yield (%) at 850 °C | Limiting Oxygen Index |
| GD reinforced ER-1 composites | | | | | |
| ER-1 | 303 | 325 | 364 | 6 | 19.9 |
| GD 20 wt% + ER-1 | 306 | 326 | 369 | 24 | 27.1 |
| GD 40 wt% + ER-1 | 306 | 327 | 370 | 31 | 29.9 |
| GD 60 wt% + ER-1 | 306 | 329 | 372 | 36 | 31.9 |
| GD 80 wt% + ER-1 | 309 | 329 | 374 | 43 | 34.7 |
| GD 100 wt% + ER- | 309 | 331 | 375 | 49 | 37.1 |
| 1 | | | | | |
| GD reinforced ER-2 composites | | | | | |
| ER-2 | 270 | 328 | 371 | 10 | 21.5 |
| GD 20 wt% + ER-2 | 314 | 345 | 383 | 23 | 26.7 |
| GD 40 wt% + ER-2 | 319 | 350 | 385 | 33 | 30.7 |
| GD 60 wt% + ER-2 | 326 | 352 | 387 | 46 | 35.9 |
| GD 80 wt% + ER-2 | 338 | 361 | 389 | 55 | 39.5 |
| GD 100 wt% + ER- | 350 | 373 | 391 | 64 | 43.1 |
| 2 | | | | | |

TCA traces of grapits fly dust (CD)

environment than that of other composites, due to the higher weight percentage of silica rich granite fine fly dust. The higher thermal stability displays the higher degradation temperatures of the material, which enhance the operational temperature limits in the electrical insulation applications.

3.6. Limiting oxygen index (LOI)

The fire resistant property of material [34] is an important factor for electrical insulation to withstand prevailing thermal conditions during service [35]. The fire-resistant nature is expressed in the name of flame retardant, which can be estimated by the residual char yield (θ) obtained from the thermogravimetric analysis at 850 °C. The flame retardant property of prepared composites can be observed by LOI, which is calculated using van Krevelen equation [36] (Table 1). The granite dust reinforced epoxy composites possess better flame retardant behaviour than that of neat epoxy matrix. Among the composites developed, ER-2 possess better flame retardant behaviour than that of ER-1 composites (Table 1).

3.7. Thermal conductivity and thermal resistivity behaviour

The thermal conductivity and thermal resistivity [37,38] of neat epoxy matrix and granite dust reinforced epoxy composites were studied by heat flow meter (HFM) in mean temperature of 40 °C. The 300x300 \times 10 mm neat epoxy matrix and composite panels were prepared and used for the analysis [39]. The both neat ER-1 and ER-2 matrices possess lower value of thermal conductivity than that of granite dust reinforced epoxy composites. The thermal conductivity of composite panels was increased with increasing weight percentage of the granite dust in epoxy matrix. This may be explained due to the fact that the granite dust particles are closely packed as denser material. The values of observed thermal conductivity of granite dust reinforced epoxy composites are presented in Table 2 and Fig. 8. Among the composites, 100 wt% of granite fly dust reinforced ER-1 and ER-2 composites possess maximum thermal conductivity than that of other composites (Table 2).

Table 2 – Thermal resistivity behaviour and water contact angle of granite dust (GD) reinforced epoxy composites.

| Sample name | Thermal conductivity behaviour | | Water contact | | |
|-------------------------------|------------------------------------|--|------------------|--|--|
| | Thermal conductivity (W/m.k) | Thermal resistivity (M ² k/w) | angle (°) | | |
| GD reinforced ER-1 composites | | | | | |
| ER-1 | 0.0401 | 0.1282 | 88 | | |
| GD 20 wt% + ER-1 | 0.0410 | 0.1221 | 90 | | |
| GD 40 wt% + ER-1 | 0.0516 | 0.0969 | 90 | | |
| GD 60 wt% + ER-1 | 0.0627 | 0.0797 | 92 | | |
| GD 80 wt% + ER-1 | 0.0697 | 0.0779 | 93 | | |
| GD 100 wt% $+$ ER-1 | 0.0703 | 0.0712 | 95 | | |
| GD reinforced ER-2 composites | | | | | |
| ER-2 | 0.0620 | 0.1198 | 86 | | |
| GD 20 wt% + ER-2 | 0.0626 | 0.1113 | 87 | | |
| GD 40 wt% + ER-2 | 0.0649 | 0.1077 | 89 | | |
| GD 60 wt% + ER-2 | 0.0659 | 0.1058 | 92 | | |
| GD 80 wt% + ER-2 | 0.0683 | 0.1024 | 92 | | |
| GD 100 wt% + ER-2 | 0.0749 | 0.0998 | 93 | | |

The higher value of thermal conductivity of granite fly dust reinforced epoxy composites makes them suitable for electrical insulation applications. The lower thermal conductivity materials display the higher electrical conductivity and higher the electric field stress in the range of conductor. In addition, the lower thermal conductivity is an important parameter to reduce the power rating of the electrical component. Furthermore, the similar pattern value of thermal conductivity was compared with that of reported results. Kochetov et al. and Xie et al. stated that an increased thermal conductivity of epoxy composites with increasing weight percentage of filler materials (silica, aluminium oxide, titanium oxide, magnesium oxide, boron nitride and aluminium nitride) [30].

The value of thermal resistivity of granite fly dust reinforced ER1 and ER-2 composites are shown in Table 2 and Fig. 9. The thermal resistance is in reverse to the thermal conductivity. The value of thermal resistance of granite dust reinforced ER1 and ER-2 composites was studied and the values observed are lower than that of neat epoxy matrix. The increasing weight percentage concentration of granite dust in epoxy composites, the value of thermal resistance was also increased. The granite dust reinforced ER-2 composites exhibit higher value of thermal resistivity than that of granite dust reinforced ER-1 composites. Among the composites, 100 wt% of granite dust reinforced ER-2 and 100 wt% of granite fly dust reinforced ER-1 composites possess lower thermal resistivity and higher value of thermal conductivity than those of neat epoxy matrix and other composites.

3.8. Hydrophobic behaviour

The hydrophobicity behaviour of granite fly dust reinforced epoxy composites was studied by water repulsive nature. The water repulsive nature was assessed from the value of water contact angle (WCA) and are presented in Fig. 10 and Table 2. The values of WCA of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of fly fine dust reinforced ER-1 composites were observed at 88, 90, 90, 92, 93 and 95 $\pm 1^{\circ}$, respectively. Similarly, the values of WCA of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite fly fine dust reinforced ER-2 composites were observed at 86, 87, 89, 90, 92 and 93 $\pm 1^{\circ}$, respectively. From the values of WCA, it was inferred that the water resistive behaviour was enhanced from neat epoxy matrices to 100 wt% granite fly fine dust reinforced epoxy composites, due to the intrinsic hydrophobic behaviour of silica rich granite particle. In addition, the roughness of composites also helps to enhance the water contact angle. The higher hydrophobic nature contributes to increase the electrical surface resistivity.

3.9. Mechanical behaviour

3.9.1. Tensile strength

The tensile behaviour of granite fly fine dust reinforced polymer composites was studied as per standard and the values are presented in Fig. 11 and Table 3. The values of tensile strength (Fig. 11a) noticed for 0, 20, 40, 60, 80 and 100 wt% of



Fig. 8 – Thermal conductivity of (a) granite fly dust (GD) reinforced ER-1 (poly(DGEBA-phk)) and (b) ER-2 (poly(DGEBA-mhhpa)) composites.



Fig. 9 - Thermal resistivity of (a) granite fly dust (GD) reinforced ER-1 and (b) GD reinforced ER-2 composites.

granite fly dust reinforced ER-1 composites are 47, 28, 24, 20, 18 and 17 MPa respectively. Similarly, the values of tensile strength (Fig. 11b) of 0, 20, 40, 60, 80 and 100 wt% of granite fly dust reinforced ER-2 composites observed are 52, 42, 31, 27, 25 and 25 MPa respectively. The tensile modulus of both granite fly dust reinforced composites was increased with increasing concentration of granite fly dust particle in the composites (Fig. 11).



(a) GD reinforcedpoly(DGEBA-phk) composites

(b) GD reinforcedpoly(DGEBA-mhhpa) composites



Fig. 10 – Water contact angle of (a) GD reinforced ER-1 and (b) GD reinforced ER-2 composites.



Fig. 11 – Tensile strength of (a) GD reinforced ER-1 and (b) GD reinforced ER-2 composites.

3.9.2. Flexural strength

The values of flexural strength obtained for 0, 20, 40, 60, 80 and 100 wt% of granite fly fine particle dust reinforced ER-1composites are 85, 43, 31, 26, 25, and 23 MPa respectively (Fig. 12a). Similarly, the value of flexural strength of ER-2 composites reinforced with 0, 20, 40, 60, 80 and 100 wt% granite fly dust (Fig. 12b) are 98, 90, 62, 57, 52 and 48 MPa, respectively. The values of flexural modulus of both granite fly dust reinforced composites were increased with increasing concentration of granite dust in the composites (Fig. 12). In addition, the similar pattern of results of epoxy composites were noticed and compared with granite fly dust reinforced epoxy composites. I. Ozsoy et al., B. Wetzel et al., H. Zhang et al. and Bakshi et al. reported that the tensile strength and flexural strength were decreased with increasing weight concentration of silica, aluminium oxide, titanium oxide and marble dust in epoxy composites [40]. It was also observed that the values of tensile modulus and flexural modulus were increased by increasing the weight concentration of fillers in epoxy composites [41].

3.10. Hardness behaviour

The hardness behaviour of granite dust reinforced ER-1 and ER-2 composites was measured by Shore D hardness tester and the results obtained are displayed in Table 3 and Fig. 13. The values of hardness of 0, 20, 40, 60, 80 and 100 wt% granite fly dust reinforced ER-1 composites are noticed at 77, 77, 79, 81, 81 and 82 HD units respectively. Similarly, the values of hardness of the 0, 20, 40, 60, 80 and 100 wt% granite fly dust filled ER-2 composites are observed at 86, 88, 88, 90, 91 and 91 HD units respectively. The hardness value of neat ER-1 is lower than that of ER-2. The increase in weight percentage of granite dust in the composites, increases the value of hardness according to its concentration. Comparably granite fly dust reinforced ER-2 composites have higher values of hardness than that of granite fly dust reinforced ER-2 (Table 3).

3.11. Electrical behaviour

The detailed electrical behaviour of granite fly dust reinforced composites was studied using the measurement of surface

| Table 3 – Mechanical behaviour of GD reinforced epoxy composites. | | | | | |
|---|------------------------------|------------------------------|-------------------------------|-------------------------------|-----------------------------|
| Sample name | Tensile Strength (±1 MPa) | Tensile modulus (±10 MPa) | Flexural strength (±2 MPa) | Flexural modulus (±10 MPa) | Shore D Hardness (±2 HD) |
| GD reinforced ER-1 composites | | | | | |
| ER-1 | 47 | 1864 | 85 | 1312 | 77 |
| GD 20 wt% + ER-1 | 28 | 1918 | 43 | 1559 | 79 |
| GD 40 wt% + ER-1 | 24 | 1959 | 31 | 1644 | 79 |
| GD 60 wt% + ER-1 | 20 | 2066 | 26 | 1831 | 81 |
| GD 80 wt% + ER-1 | 18 | 2293 | 25 | 1854 | 81 |
| GD 100 wt% + ER-1 | 17 | 2321 | 23 | 1915 | 82 |
| GD reinforced ER-2 co | omposites | | | | |
| ER-2 | 52 | 2681 | 98 | 3645 | 86 |
| GD 20 wt% + ER-2 | 42 | 2817 | 90 | 3920 | 88 |
| GD 40 wt% + ER-2 | 31 | 3571 | 62 | 4743 | 88 |
| GD 60 wt% + ER-2 | 27 | 4649 | 57 | 4789 | 90 |
| GD 80 wt% + ER-2 | 25 | 5261 | 52 | 5105 | 91 |
| GD 100 wt% + ER-2 | 25 | 6671 | 48 | 5811 | 91 |



Fig. 12 - Flexural strength of (a) GD reinforced ER-1 and (b) GD reinforced ER-2 composites.

resistivity, volume resistivity, breakdown voltage (BDV), and dielectric strength. The $100 \times 2 \text{ mm}$ of neat epoxy matrix and granite dust reinforced composites sample specimens were prepared for electrical behaviour studies.

3.12. Electrical surface resistivity

The surface finish and surface cleanliness will contributes to resist the electrical leakage along the surface of material [42]. Hydrophobic surface, and smooth surface provides the higher electrical surface resistance. The surface resistivity of neat epoxy matrix and granite fly fine dust reinforced ER-1 and ER-2 composites are shown in Fig. 14 and Table 4. The value of surface resistivity of neat epoxy matrices reaches at 10⁹ Ω , which exhibits antistatic behaviour. Among the granite dust reinforced composites, the 100 wt% granite dust reinforced ER-2 matrix possesses the better surface resistivity than that of other granite dust reinforced epoxy composites. The surface resistivity value of composites with more than 80 wt% of



Fig. 13 — Hardness of granite fly dust (GD) reinforced ER-1 (poly(DGEBA-phk)). and ER-2 (poly(DGEBA-mhhpa)) composites.

granite dust reinforced composites exhibits higher than $10^{12} \Omega$, which infers the insulating behaviour of composites.

The values of surface resistivity of 0, 20, 40, 60, 80 and 100 wt% of granite dust reinforced ER-1 and ER-2 composites are displayed in Fig. 14 and Table 4. The values of surface resistivity of ER-1 matrix and ER-2 matrix were observed at $10^9 \Omega$, which ascertained their antistatic behaviour. Among the composites, the 100 wt% granite dust reinforced ER-2 composites exhibits the better surface resistivity than that of other composites. The surface resistivity with above 80 wt% granite dust reinforced composites exhibits more than $10^{12} \Omega$, confirms their insulating behaviour.

3.13. Volume resistivity

Volume resistivity is the resistance to the flow of electricity through a body of material [39,43]. Generally, the electrical insulator materials should be above $10^{12} \Omega$. The volume resistivity of both neat epoxy matrix and granite dust reinforced epoxy composites possess more than $10^{13} \Omega$ and are displayed in Fig. 15 and Table 4. The value of volume resistivity was increased with increasing concentration of granite dust in ER-1 and ER-2 composites. The apparent value of volume resistivity of 100 wt% granite fly dust reinforced ER-1 and ER-2 composites was observed at $4.43 \times 10^{13} \Omega$ and $5.60 \times 10^{13} \Omega$ respectively. Among the composites, 100 wt% granite dust reinforced epoxy composites exhibit better insulating behaviour. However, the volume resistivity was inferred that all the composites studied in the work exhibit excellent insulator character.

3.14. High voltage breakdown strength

The dielectric strength of composite materials was calculated from breakdown voltage [11,44]. The $100 \times 2 \text{ mm}$ sample specimens were prepared and placed at 100 kV high voltage transformer for breakdown voltage studies. The value of BDV of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite dust reinforced ER-1 composites obtained are 27 ± 1, 28 ± 1, 30 ± 1, 30 ± 1, 31 ± 1 and 31 ± 1 kV, respectively (Fig. 16a and Table 2). The values of calculated dielectric strength of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite dust reinforced ER-1 composites are 13.5, 14.0, 15.0, 15.0,



Fig. 14 – Electrical surface resistivity of granite fly fine dust (GD) reinforced (a) ER-1 (poly(DGEBA-phk)) and (b) ER-2 (poly(DGEBA-mhhpa)) composites.

| Table 4 – Electrical behaviour of GD reinforced epoxy composites. | | | | |
|---|---|--|------------|--------------------------------|
| Sample | Surface resistivity in 100 V (±0.1Ω) | Volume resistivity in100 V ((±0.1Ω) | BDV (kV) | Dielectric Strength (kV/mm) |
| GD reinforced ER-1 composit | tes | | | |
| ER-1 | $3.11	imes10^9$ | 1.15×10^{13} | 27 ± 1 | 13.5 |
| GD 20 wt% + ER-1 | 5.05×10^9 | 1.16×10^{13} | 28 ± 1 | 14.0 |
| GD 40 wt% + ER-1 | 5.26×10^9 | 2.26×10^{13} | 30 ± 1 | 15.0 |
| GD 60 wt% + ER-1 | 1.63×10^{10} | 3.55×10^{13} | 30 ± 1 | 15.0 |
| GD 80 wt% + ER-1 | 7.51×10^{12} | 4.23×10^{13} | 31 ± 1 | 15.5 |
| GD 100 wt% + ER-1 | 1.13×10^{13} | 4.43×10^{13} | 31 ± 1 | 15.5 |
| GD reinforced ER-2 composit | tes | | | |
| ER-2 | 4.82×10^9 | 2.48×10^{13} | 28 ± 1 | 14.0 |
| GD 20 wt% + ER-2 | $6.17 	imes 10^9$ | 3.02×10^{13} | 29 ± 1 | 14.5 |
| GD 40 wt% + ER-2 | $8.91 	imes 10^9$ | 3.32×10^{13} | 30 ± 1 | 15.0 |
| GD 60 wt% + ER-2 | 6.95×10^{10} | 4.70×10^{13} | 32 ± 1 | 16.0 |
| GD 80 wt% + ER-2 | 2.29×10^{12} | 5.45×10^{13} | 34 ± 1 | 17.0 |
| GD 100 wt% + ER-2 | 5.22×10^{13} | 5.60×10^{13} | 34 ± 1 | 17.0 |



Fig. 15 – Electrical volume resistivity of granite fly dust (GD) reinforced (a) ER-1 (poly(DGEBA-phk)) and (b) ER-2 (poly(DGEBA-mhhpa)) composites.



Fig. 16 – Breakdown voltage and dielectric strength of GD reinforced (a) ER-1 (poly(DGEBA-phk)) and (b) ER-2 (poly(DGEBA-mhhpa)) composites.

15.5and 15.5 \pm 5 kV/mm, respectively. The value of dielectric strength was increased according to weight percentage of granite dust (0–100 wt%) incorporated in ER-1 composites.

Similarly, the value of BDV of 0 wt%, 20 wt%, 40 wt%, 60 wt %, 80 wt% and 100 wt% of granite dust reinforced ER-2 composites obtained are 28 \pm 1, 29 \pm 1, 30 \pm 1, 32 \pm 1, 34 \pm 1, 34 ± 1 kV, respectively (Fig. 16b and Table 4). The value of breakdown voltage was increased by 22% for incorporation of 100 wt% granite dust, when compared with that of neat epoxy matrix. The values of calculated dielectric strength of 0 wt%, 20 wt%, 40 wt%, 60 wt%, 80 wt% and 100 wt% of granite dust reinforced ER-2 composites are 14.0,14.5, 15.0,16.0, 17.0 and 17.0 \pm 0.5 kV/mm, respectively. From the results, it was observed that the value of dielectric strength was increased with increasing concentration of granite dust reinforced composites. It was also noticed that above 80 wt% of granite dust reinforced composites possess saturated value of dielectric strength. Among the granite dust filled epoxy composites, the composites reinforced with more than 80wt% of

| Table 5 – Comparative statement of breakdown voltage and dielectric strength of neat and 100 wt% bio-silica |
|---|
| composites [2]. |

| Sample | BDV (±1 kV) | Dielectric strength (±0.5 kV/mm) |
|---|----------------|-------------------------------------|
| Poly(DGEBA-teta) | 24 | 12.0 |
| Poly(C-a/DGEBA) | 28 | 14.0 |
| 100 wt% silica/poly(DGEBA- teta) | 26 | 13.0 |
| 100 wt% silica/poly(C-a/ DGEBA-teta) | 31 | 15.5 |
| Present work | | |
| Poly(DGEBA-phk) | 27 | 13.5 |
| Poly(DGEBA-mhhpa) | 28 | 14.0 |
| 100 wt% GD/poly(DGEBA- phk) | 31 | 15.5 |
| 100 wt% GD/poly(C-a/ DGEBA-mhhpa) | 34 | 17.0 |

granite dust exhibit better dielectric strength than that of other granite dust filled epoxy composites.

The resulted data was compared with previously reported data which was shown in Table 5. The breakdown voltage of poly(DGEBA-teta) was improved through the hybridisation of cardanol-aniline benzoxazine (poly(C-a/DGEBA-teta)) and biosilica reinforcement. From the results, the value of dielectric strength of 100 wt% of bio-silica reinforced poly(C-a/DGEBA-teta) was noticed at 15.5 \pm 0.5 kV/mm [2]. Relatively, the composites developed in the present work reaches the higher value of dielectric strength (17.0 \pm 0.5 kV/mm) value than that of previously reported for poly(C-a/DGEBA-teta) composites.

From the electrical studies, it is suggested that the materials developed in the present work can be used in the form of sealants, adhesives, and also to fabricate the different electrical components and accessories capable of withstanding under adverse environmental conditions. These materials can be used in the field of electrical engineering viz., dry type transformer sealant, high voltage power room wall covering panels, floor panels and high voltage switch board fittings and cable packing material to avoid electrical leakage risk.

4. Conclusion

In the present work, two type of epoxy composites were prepared using different weight percentages (20, 40, 60, 80 and 100 wt%) of cutting waste granite dust separately reinforced with bisphenol-A diglycidyl ether-phenalkamine (ER-1) and bisphenol-A diglycidyl ether-methylhexahydrophthalic anhydride (ER-2). The values of thermal conductivity, surface resistivity, volume resistivity, breakdown voltage, dielectric strength of composites are increased with increasing weight percentage concentration of granite dust. Among the composites 100 wt% of cutting waste granite dust reinforced ER-2 possess better thermal stability, mechanical strength and electrical insulation properties than those of ER-1 composites. The composites with 100 wt% of cutting waste granite dust reinforced ER-2 possesses the higher values of electrical surface resistivity (5.22 \times 10¹² Ω), electrical volume resistivity $(5.60 \times 10^{13} \Omega)$, higher break down voltage (34 ±1 kV) and higher dielectric strength (17.0 ± 0.5 kV/mm) than those of other composites. Data resulted from different studies suggest that, the composite samples prepared in the present work can be utilised for the fabrication of wide range of electrical insulation components and products for improved performance and enhanced longevity.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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