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Experimental Investigation of Mechanical, Viscoelastic, and Dielectric behavior in Blended Hybrid Epoxy Composites Made with Coffee Bean Filler

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ABSTRACT

The study aims to understand the interplay between fillers and epoxy matrix in hybrid composite materials. The impact of coffee bean powder on the dielectric, tensile, flexural, impact, and dynamic properties of epoxy resin containing *Vachellia nilotica* (VN) is investigated in this work. The hand layup method was used to create the composite samples, which included various volume fractions of coffee beans (3, 6, 9, 12, and 15 v/v%) as a particulate filler. The VN content was maintained at a constant concentration of 15 v/v%. Additionally, experiments were conducted to ascertain the mechanical, viscoelastic, and dielectric characteristics of the hybrid composites. The findings clearly show that among the varying volume fractions, the one containing 9 v/v% coffee bean has substantially superior mechanical and dielectric properties. Additionally, it was observed that the epoxy composite with a filler percentage of 9 v/v% had better glass transition temperature and storage modulus. The TGA test revealed that the addition of coffee beans to the VN hybrid increased the composite's thermal strength. Further, the dielectric test findings demonstrated that the coffee bean filler increased the resin's dielectric strength. The tensile fracture of the composite was analyzed using scanning electron microscopy through assessing structural morphology.

Keywords: Biofibre; Composite; Coffee bean powder; Morphological analysis; *Vachellia nilotica*.

1. INTRODUCTION

Due to the depletion of crude petroleum sources and the environmental harm of petroleum-based polymers, researchers are exploring sustainable alternatives (Dhanaraj *et al*. 2021; Kumar *et al*. 2022). Single-use plastics are restricted due to their non-biodegradability (Yesuraj *et al*. 2021; Kumar *et al*. 2017; Santhanam and Chandrasekaran, 2014). Since biopolymers are derived from organic components like cellulose, proteins, sugars, polysaccharides, vegetable oils, and microbes, they may offer a solution to this problem (Venkateshwaran *et al.* 2011, Alavudeen *et al.* 2011, Vigneshwaran *et al.* 2018). Nguyen and Nguyen, (2021) tested composite compatibility by adding varying weight percentages of spent coffee grounds (SCG) to epoxy resin. Composites with 30% SCGs demonstrated outstanding compatibility without interfacial phase separation. The composite had 80.07 MPa flexural and 44.81 MPa tensile strength. The study showed that SCGs can improve the mechanical, thermal, and flame-retardant properties of the epoxy composites, making coffee grounds a more sustainable additive. Nayak *et al.* (2023) discovered that banyan aerial root (BAR) powder improved composite mechanical characteristics. When 4% BAR was added, the composites had the maximum tensile and flexural strengths of 407 and 339 MPa, respectively. Samples with 6% BAR powder had excellent impact strength (194.02 kJ/m^2). The results indicate a correlation between biofiller content and composite mechanical properties. Deepak *et al.* 2019 studied the mechanical characteristics of epoxy using dry floral waste as a biofiller at volume fractions of 0.04, 0.08, 0.12, 0.16, and 0.20 v/v. Impact strength, flexural properties, and tensile properties were examined and results revealed that the biofiller significantly influenced tensile and flexural modulus. Notably, 12% v/v biofiller composition achieved the best mechanical properties.

Abdul-Hussein *et al.* (2014) examined the effect of $CaCO_3$, K_2CO_3, and Na_2CO_3 powders on the mechanical characteristics of glass fibre reinforced epoxy composites. Epoxy resin was used as the matrix, with varying volume fractions of industrial powder fillers and 6% glass fibre reinforcement. Industrial powder fillers improved composite characteristics like moisture intake, flexural strength, hardness, and shear stress. The rise in volume fraction correlated with these desired features. A constant dispersion of industrial powder and its reinforcing characteristics improve the epoxy/glass fibre composite performance. Koyuncu, 2021 examined the tensile and flexural strengths of epoxy composites augmented with pumice powder and maize shell. The composites with 20 and 15 wt% pumice powder had the maximum tensile and flexural strengths, thus improving the mechanical characteristics. Similarly, Lutfi and Jassim (2023) observed that adding cantaloupe peel powder to epoxy composites at varying weight percentages (1% to 10%) improved hardness, flexural strength, compression strength, and impact resistance. Srinivasan *et al*. (2022) found that 30% v/v of epoxy resin and *Pterocarpus marsupium* resin powder produced the best results. They also investigated the effect of turtle shell powder (TSP) filler and found that 1%, 3%, and 5% TSP weight percentages affect composite tensile, flexural, impact, and hardness. Md-Shah, *et al*. (2021) conducted dynamic mechanical tests and found that bamboo powder increases the stiffness of epoxy composite. Jenkins *et al*. (2019) examined how reduced graphene oxide (rGO) affects carbon fiber incorporated shear and flexural characteristics of epoxy composite at different temperatures. They found that with rGO the flexural strength and modulus of composites increase by 62% and 44%, respectively. It was also noted that with rise in temperatures, the benefits decreased.

Jagadeesh *et al*. (2021) found that particulate powder derived from *Pongamia pinnata* shell significantly affected Kevlar-Sisal hybrid mechanical characteristics of the composite. The study found that 2% filler composites had higher interlaminar shear and flexural strengths, while the composites with 6% filler had high hardness and flexural modulus. Also, this study reveals that 2.5 wt% basalt powder enhances composite rigidity and thermal stability. These results are comparable to those got by adding coffee bean powder to epoxy composites. Majhooll *et al.* (2019) showed that adding 10% natural hydroxyapatite (nHAp) powder to epoxy increased flexural strength by 77% and tripled the impact strength. The SEM and EDX showed consistent nHAp particle dispersion in the epoxy matrix, whereas the FTIR study revealed that extra nHAp powder increased phosphate group intensity in the composite.

Sathish *et al.* (2022) analysed the epoxy composites reinforced with cellulose fibres for the mechanical and thermal characteristics. They developed hybrid composites using coir, bamboo, and jute fibres. The composite JC8, made of 1.5% coir, 7.5% bamboo, and 21% jute had excellent tensile and bending properties. These fibres have better mechanical qualities due to their increased cellulose content. The thermal stability of composites was consistent throughout the study. The JC8 hybrid composite outperformed others in tensile, flexural, thermal characteristics, and surface morphology. The volume fraction also affects material characteristics. In this background, this study used coffee bean powder waste as biofiller to evaluate its effect on the mechanical properties of epoxy resin.

2. MATERIALS AND METHODS

This study employed a polyamine-type hardener (HY951) and an epoxy resin (LY556) matrix material. The biofiller used in this study was coffee bean powder, obtained from local traders in Kanchipuram, India. The powder underwent a drying process before being subjected to a ball milling technique, resulting in a particulate size varying between 10 and 50 µm. The density of the powdered coffee bean biofiller was found to be 1.05 g/cm³. Composite samples were fabricated using the hand layup technique. The samples were generated in different volume percentages of coffee bean powder, ranging from 3% to 15% v/v. The *Vachellia nilotica* content was maintained at a concentration of 15 v/v%. An application of a removal agent was used on the surface of the mould to facilitate easy removal of the cured sample. The hardener and epoxy resin were combined in a weight ratio of 10:1 to achieve this matrix. Coffee bean powder was added to the epoxy mixture in line with the volume fractions that had been predetermined. The mixture was manually stirred for 15 minutes. After cautiously pouring into a wooden mould, the liquid was then gently brushed to create a smooth surface. To ensure that every test specimen was identical, the composite (after curing) was cut into pieces using ASTM dimensions.

The evaluation of tensile, impact, and flexural strength of the hybrid epoxy-coffee bean powder samples was conducted in accordance with ASTM standards. Tensile strength was evaluated following the ASTM D 638 standard, utilizing a crosshead speed of 5 mm/min. The flexural properties were evaluated through a three-point bend test conducted at a rate of 1.5 mm/min, adhering to the ASTM-D710 standard protocol. The IZOD impact strength of the hybrid epoxy composite sample was evaluated using the IZOD Impact tester, following the procedures outlined as per the ASTM-D256 standard. Additionally, the analysis involved computing the mean of the outcomes for each assessment through a comparative evaluation of five samples.

The viscoelastic characteristics such as storage modulus, loss modulus and glass transition temperature of the epoxy composite samples were investigated using Dynamic Mechanical Analysis (DMA). This test yields critical information regarding the mechanical and thermal characteristics of materials under different temperature and stress conditions. In the course of the experiments, it was recommended that particular parameters be chosen to align with the established experimental configuration.

The morphological features of the specimens were studied by taking pictures with a Hitachi S 3400N scanning electron microscope (SEM). Thus, based on the observations of the cross-sectional morphology of the specimens, it was possible to evaluate the bonding between the epoxy matrix and the bio particulate filler. To prepare the materials for analysis, samples were first cut into thin sections and then coated with a layer of carbon. When testing, an accelerating voltage of 20 kV was used.

The thermal properties of the epoxy coffee bean powder composites were assessed in a nitrogen (N_2) environment using a Thermogravimetric Analyser (TGA, NETZSCH STA 2500). The measurements were performed over a 30 ℃ to 600 ℃ temperature range at the rate of 10 ℃ per minute. An analysis of weight loss curves was conducted using the TGA data, examining various stages of decomposition.

3. RESULTS AND DISCUSSION

3.1. Mechanical Properties

Following an analysis of the stress-strain graphs, it was determined that the tensile, flexural, and impact properties of epoxy composites are significantly influenced by the volume percentage of the biofiller. The incorporation of coffee bean biofiller affected the tensile strength parameters of the epoxy composite, as illustrated in Fig. 1. The composite material demonstrated a maximum tensile strength of 14.5 MPa when subjected to a biofiller loading of 9% by volume under the experimental conditions. The composite material, which exhibited a filler loading of 9% v/v, exhibited substantial flexural and impact strengths. These values were determined at 28 MPa and 1.7 J/m^2, respectively. It can be observed that the introduction of granular filler material has as minimum impact on the overall mechanical properties of the hybrid epoxy coffee bean composite.

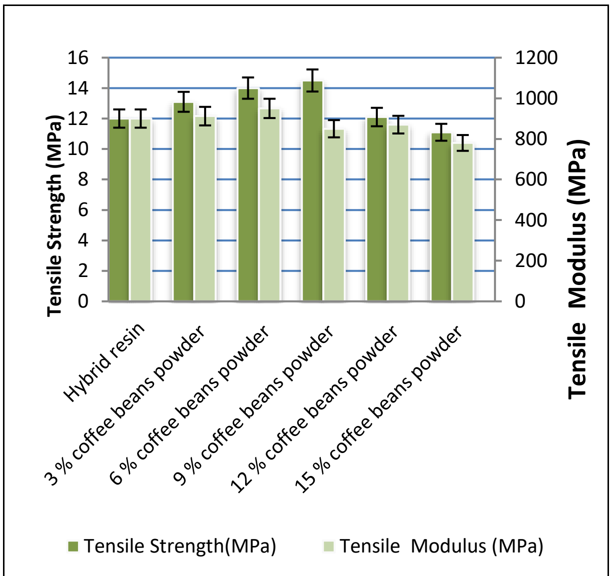

Fig. 1: Tensile strength of Coffee bean powder filler - VN/epoxy matrix

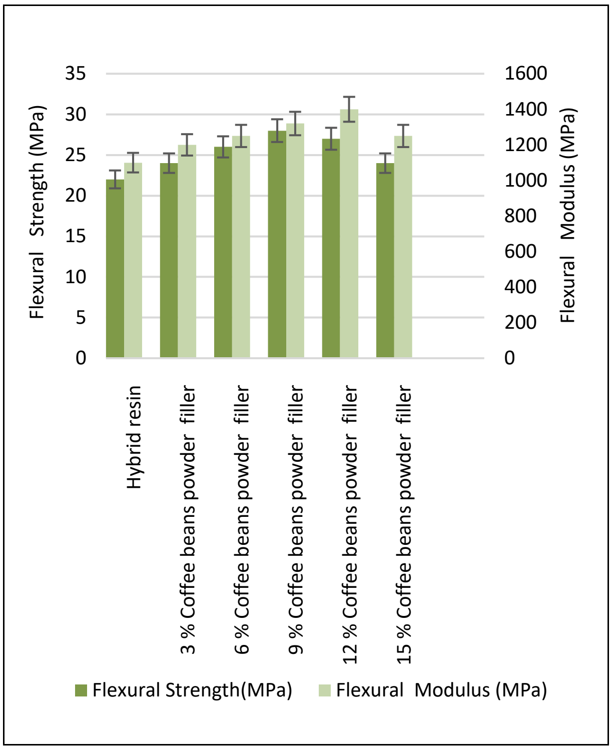

Fig. 2: Flexural strength of Coffee bean powder filler-VN /epoxy composite

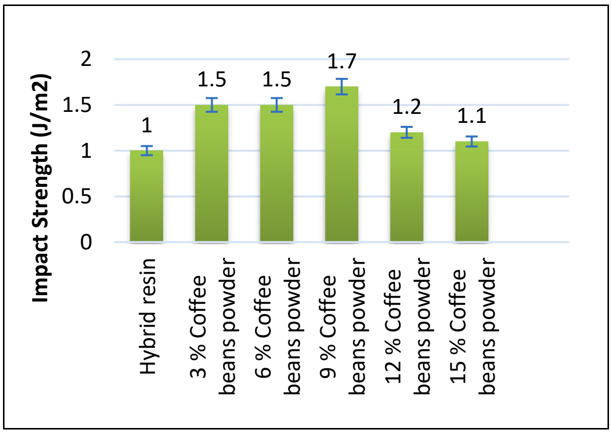

Fig. 3: Impact strength of Coffee bean powder filler-VN/epoxy matrix composite

Nevertheless, the mechanical moduli of the hybrid epoxy coffee bean composite material have been significantly enhanced as a consequence of the incorporation of biofiller. The results indicate that the addition of infill material to the composite material significantly improves the tensile and flexural moduli of the composite, resulting in a substantial increase in the composite's stiffness and rigidity. It has been demonstrated that composites with a biofiller volume fraction greater than 9% v/v manifest a substantial reduction in their mechanical properties. The observed behaviour can be ascribed to the fact that the epoxy matrix and the biofiller did not establish sufficient contact. The mechanical performance of the material may be adversely affected if the matrix coupling of the filler material is compromised. The experimental results are in concurrence with the studies conducted by Vieira *et al.* (2023), which involved the development of a biocomposite by combining eggshell with green polyethylene biopolymer. The results of this investigation underscore the importance of incorporating fibre elements into composites to enhance their mechanical, thermodynamic, and rheological properties.

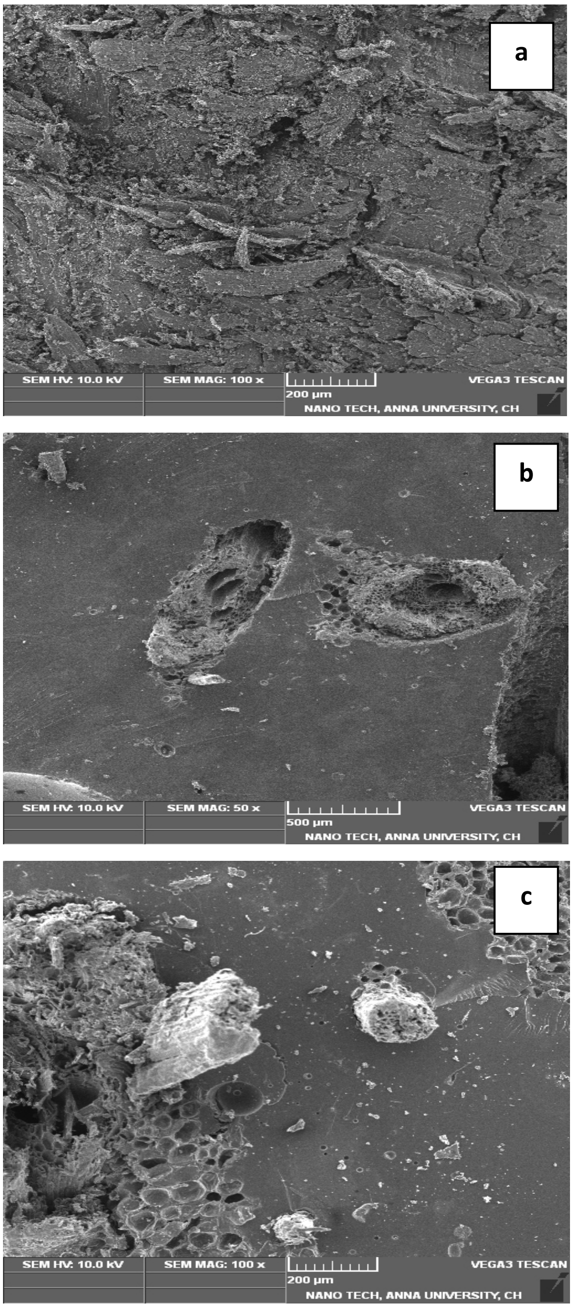

Fig. 4: SEM images (a) Composite with 3 vol% biofiller (b) Composite with 9 vol% biofiller (c) Composite with 15 vol% biofiller

3.2 Fractography Observation of Sea Shells Filler Dispersed VN/epoxy Composites

The SEM images shown in Fig. 4(a)–(c) offer vital insights into the dispersion and interfacial adherence of the coffee bean powder and *Vachellia Nilotica* in the epoxy matrix. The scanning electron micrographs clearly demonstrate the observable link amongst the biofiller and matrix. The specimens were obtained from the fractured surface under mechanical stress. Fig. 4a illustrate the composite that includes a biofibre volume of 3%. The existence of voids in the matrix suggests that the filler material is not evenly distributed and that the transmission of load amongst the coffee bean powder and the polymer matrix is not effective. In addition, it is evident that the matrix is experiencing stress in this particular scenario, suggesting a lack of strong bonding between the coffee bean filler and the epoxy matrix. Fig. 4b show the SEM images of a composite that includes 9% v/v coffee bean biofiller. The images show the presence of rough voids and fractured surfaces in the epoxy matrix, indicating a stronger bond between the epoxy and filler material. Through this enhanced contact, there is a notable increase in the efficiency of stress transfer across the surface, leading to enhanced mechanical characteristics. Alternatively, when the volume of bio fibre surpasses 15% v/v, as shown in Fig. 4c the epoxy matrix does not effectively cover the filler surface, leading to insufficient bonding amongst both the components. Inadequate load transmission between the filler and the matrix leads to a decrease in mechanical strength.

3.3. Dynamic Mechanical Analysis

The DMA provides insights into the effects of filler volume percent and temperature on the storage modulus at a frequency of one hertz (Hz). Based on the findings, it appears that incorporating the filler element into the composite improves the energy storage capabilities of the composite material. Fig. 5 clearly demonstrates the increased rigidity of the fundamental epoxy resin due to its lower storage modulus. The composite materials exhibit an elevated glass transition temperature (T_g) of 100 °C, which can be credited to the enhanced molecular motion of the epoxy polymer chain. The findings indicate that the integration of biofiller into the polymer matrix enhances the rigidity of the composite material. As the storage modulus increases, the influence of the filler material becomes evident in both the glassy and rubbery regions. Research indicates that the incorporation of a natural filler into the epoxy matrix has the potential to enhance the stiffness of both the brittle and ductile elements of hybrid composite materials.

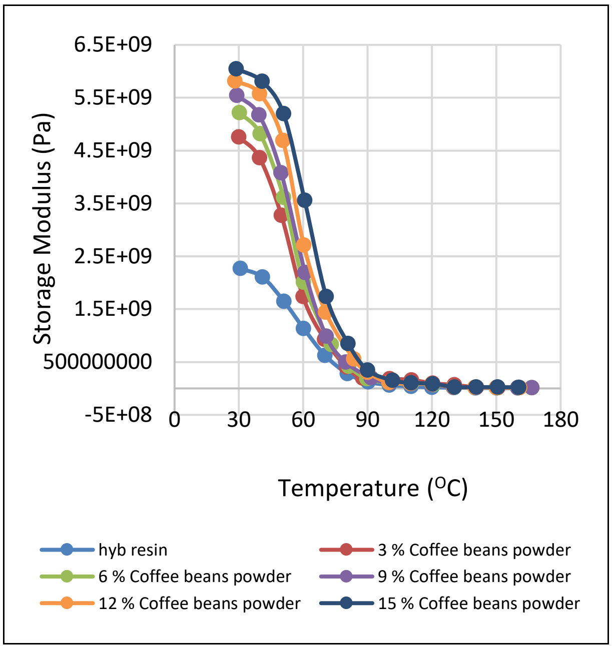

Fig. 5: Storage modulus of Coffee bean powder dust filler reinforced epoxy and *Vachellia nilotica* at 1 Hz frequency

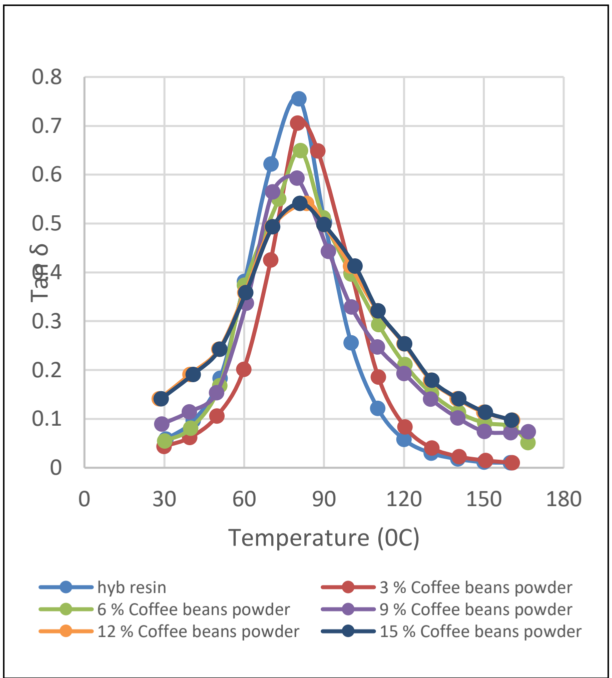

Fig. 6: Effect of Tan δ at 1 Hz frequency on Coffee bean powder dust filler reinforced epoxy and VN composite

The level of molecular mobility within the epoxy molecular chain and the material's ability to release energy under load are crucial factors when determining the damping factor. Calculating the ratio of storage to loss modulus is a key aspect of this investigation. Fig. 6 illustrates the damping factor of the composite material composed of *Vachellia nilotica*, coffee bean powder filler, and epoxy filler. Studies have demonstrated that as the volume proportion of filler loading decreases, there is an increase in the interface between the biofiller and the matrix. The composite material exhibits a higher energy release as a result of the improved contact. In contrast, Fig. 6 demonstrates that the dissipation of energy decreases with an increase in filler content (above 12% volume percent). In a comparison between an epoxy resin matrix and a composite material with a higher filler content, it was observed that the peak height of Tan δ in the epoxy resin matrix is relatively smaller. There is evidence of a decrease in peak height, which indicates a significant improvement in the bond between the resin matrix and natural filler. This suggests that there has been a notable enhancement in interfacial adhesion. In comparing the energy loss between the hybrid composite (epoxy matrix with natural filler) and the epoxy matrix resin, it is evident that the former exhibits a lower level of energy loss

Table 1. Effect on thermal stability of composites on adding coffee bean powder

Composition	Degradation temperature (°C)			Residue (%)
	Stage 1	Stage 2	Stage 3	
Epoxy + 12 Biofiller	202	112	100	0.4
3% Coffee bean powder filler + Hybrid epoxy	204	113	101	0.6
6% Coffee bean powder filler + Hybrid epoxy	206	117	101	0.9
9% Coffee bean powder filler + Hybrid epoxy	198	121	102	1.4
12% Coffee bean powder filler + Hybrid epoxy	180	132	103	1.6
15% Coffee bean powder filler + Hybrid epoxy	175	141	104	1.5

3.4 Thermogravimetric Analysis

Table 1 presents the findings regarding the thermal stability of the epoxy matrix under ambient conditions at a temperature of 200°C. The heat stability of the composite with filler surpasses that of a pure epoxy matrix. From the results it is clear that adding coffee bean powder and *Vachellia nilotica* filler improves the thermal stability of the epoxy matrix. The rise in TGA levels can be ascribed to the filler components present in the composite material. By utilizing a dust filler made from coffee bean powder, the epoxy matrix demonstrates improved durability in high temperatures. Through careful analysis, it was observed that a specific temperature range (602–677) °C marks the critical point where residues begin to accumulate and all flammable substances are completely eradicated. Throughout the study, the VN/epoxy composite demonstrated exceptional stability, maintaining a residual VN content of 1.6%, even at a high VN content of 12%. However, the composite degrades quickly at temperatures below this threshold, leaving only a small residue of 0.4%.

3.5. Dielectric Strength

The inclusion of coffee bean powder infill is thought to have enhanced the dielectric strength of the VN/epoxy matrix (Bhuvaneswari *et al*. 2018). Table 2 presents the dielectric strength of Coffee bean powder filler incorporated *Vachellia nilotica*/epoxy matrix composite samples. When no tension is applied to the epoxy composites, the dielectric strength of the epoxy/VN resin sample reaches a maximum of 2.89 KV/mm. This particular hybrid composite stands out for its exceptional dielectric strength. It is made up of a composite material infused with a 3 vol.% coffee bean powder dust. The coffee bean powder dust filler is evenly spread within the epoxy matrix at low concentrations (0% and 3%), resulting in an improved interfacial polarisation. This phenomenon leads to a higher dielectric strength due to the effective trapping of charges at interfaces, as supported by the Maxwell-Wagner-Sillars (MWS) effect. When the concentration of particles exceeds 3%, they have a tendency to come together and form larger particles. This accumulation of particles creates small gaps and imperfections, leading to a reduction in the electrical insulation capacity and the concentration of electric charge in specific areas. The increased filler content in the composite leads to a compromise in its mechanical strength, causing the development of internal tension. High filler loadings result in the formation of clusters and microcracks, leading to a decrease in dielectric performance. When faced with strong electric fields, mechanical defects can act as catalysts for electrical failure.

Table 2. Dielectric strength of Coffee bean powder filler incorporated *Vachellia nilotica* /epoxy matrix composite

Sample ID	Coffee bean powder (%)	Volume fraction of bio filler (%)	Volume fraction of Epoxy Resin (%)	Dielectric Strength (kV/mm)
1	0	12	88	16.54
2	3	12	85	18.30
3	6	12	82	15.62
4	9	12	79	11.40
5	12	12	76	10.26
6	15	12	73	8.26

4. CONCLUSION

This study examined the mechanical, dielectric, and thermal properties of hybrid epoxy resin composites with fillers from *Vachellia nilotica* and coffee bean powder. Filler content varied, with a maximum of 9% v/v, beyond which bonding and mechanical strength declined. A strong filler-resin bond maximized energy absorption and impact resistance of the material.

Scanning electron microscopy showed consistent filler dispersion at 9% v/v, enhancing glass transition temperature, storage modulus, rigidity, and energy retention in both glassy and rubbery stages. Thermogravimetric analysis indicated that fillers improved thermal stability and degradation temperatures, added to increased residue percentages, when compared to pure epoxy. At 3% coffee filler, dielectric strength peaked due to increased polarization but decreased at higher filler levels due to flaws, agglomeration, and micro gaps. Optimal performance was achieved at specific filler concentrations, with excess causing clustering and reduced adhesion.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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