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Experimental investigation of Sea shells filler on the *Vachellia Nilotica* Blended Hybrid Epoxy Composites

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ABSTRACT

This research work presents the influence of Sea shells powder on the mechanical, dynamic, and dielectric properties of the hybrid epoxy composites containing *Vachellia Nilotica* (VN). Hand layup method was used to prepare the composite specimens at different volume fractions of the Sea shells used as a particle filler (3, 6, 9, 12, and 15 v/v%). The *Vachellia Nilotica* content was kept at 15 v/v %. The mechanical, viscoelastic, and dielectric properties of the developed hybrid composites were characterized. The findings show that the composite containing 6v/v% Sea shells possesses better mechanical and dielectric characteristics compared to other volume concentrations. The filler was also found to significantly affect the epoxy composite in which the material with the highest glass transition temperature and storage modulus value containing 6 v/v % filler. The addition of Sea shells in the *Vachellia Nilotica* incorporated hybrid exposed had enhanced the thermal stability of the composite as evidenced by thermogravimetric analysis (TGA). Additionally, the dielectric test also showed that the Sea shells filler enhanced the dielectric strength of the composite. SEM equipment was used to examine the structural morphology of the tensile fracture of the composite in order to study the interface between the epoxy matrix and the fillers of the hybrid composites.

Keywords: Epoxy composite; Sea shells; *Vachellia Nilotica* resin; TGA analysis; Dielectric properties; SEM.

NOMENCLATURE

DMA	Dynamic Mechanical Analysis	SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis	μm	Micrometer
FDT	Final Degradation Temperature	IDT	Initial Degradation Temperature
MDT	Middle Degradation Temperature	ASTM	American Society for Testing and Materials

1. INTRODUCTION

Composite materials are widely utilised in various industries such as automotive, packaging, furniture, home goods, aerospace, shipping, defense, wind energy, and more. As weight concerns become critical, polymer-based composites are favorable for applications that require high strength to weight ratios. Originally, the polymer composites have been reinforced by glass, carbon, and aramid fibres for several years. There is much research being carried out at the moment to address the use of natural fibres and fillers as reinforcement as compared to synthetic products in terms of environment and carbon footprint (Palanikumar *et al.* 2018; Joseph Chandran *et al.* 2024; Hiremath *et al.* 2024). Utilising natural fibres and fillers offers a multitude of advantages. They are cost-effective, easily accessible, user-friendly, durable, sustainable, and have positive environmental impacts (Vijaya Rajan *et al.* 2020; Hadi and Mohammed 2022). In addition, studies (Kumar *et al.* 2020; Yesuraj *et al.* 2021) have indicated that the inclusion of natural fibres and fillers in polymer composites can enhance their mechanical properties when compared to synthetic materials. The properties of composite materials can be influenced by the presence of reinforcement fibres and fillers.

Kumar *et al.* (2020) investigated conch shell-dispersed GFRP composites. It was reported that, addition of 35% conch shell particles improved the tensile strength by 11.23% to 408.5 MPa, the improvement was attributed to uniform stress distribution with the incorporation of filler particles. However, the flexural strength was reported lesser due to the brittleness of the filler particles. Impact resistance improved significantly with 29.75 J impact energy. Dynamic mechanical analysis revealed that the loss modulus decreased but the glass transition temperature (Tg) increased. This indicated the energy dissipation as heat energy. Koçhan (2019) studied the effect of mussel shell waste reinforcement in the epoxy composites. The composite material was prepared using vacuum-assisted resin infusion molding (VARIM). It was reported that the composites have 170 HV micro-hardness, 24 MPa ultimate tensile strength, and 36.72 GPa flexural modulus. These experiments had shown that the recycled mussel shells was shown better than coconut and walnut shells. An eco-friendly bio-filler may improve polymer composite mechanical characteristics. Santulli *et al.* (2023) investigated the use of algae waste and mollusc shells as sustainable composite fillers. It was reported that mussel shells increased composite hardness and thermal stability as they are rich in calcium carbonate. However, algae fibres increased flexural and tensile moduli. Vasanthkumar *et al*. (2022) examined the thermal and thermo-mechanical characteristics of a seashell-reinforced Nylon-6 polymer composite incorporated with varying fractions (3, 6, 9, 12, 15, and 18 wt%) of sea shell particles. Maximum mechanical and thermal properties were reached with 15% SS, increasing glass transition, heat deflection, and vicat softening temperatures (VST). When the filler is added more than 15 wt%, the mechanical characteristics decreased as interfacial bonding became weak at higher filler concentration. They investigated using natural fillers in epoxy formulations to improve mechanical and thermal qualities. A study indicated that cashew nut and coconut shell powder increase specific strength and modulus. However, epoxy matrix bonding and moisture absorption remain issues. Nor Azwin *et al*. (2023) investigated clams, cockles, and mussels as epoxy composite fillers. The study found that 10% filler epoxy composites had the maximum tensile strength. However, filler particle agglomeration decreased strength as filler loading increased. Clumps absorbed less oil than cockles and mussels at lower filler loadings. Fombuena *et al*. (2014) characterized bio-based epoxy matrices bonded with green composites made from seashell-derived calcium carbonate. Shore D hardness and flexural modulus improved significantly with 30% seashell powder. Additionally, Shore D hardness increased by over 6% and flexural modulus by over 50%. Silane coupling agents improved particle-matrix interactions, raising the glass transition temperature by 13%. Gopal Krishna *et al*. (2021) studied epoxy-based polymer composites with glass fibres and seashell powder. Stress transmission and excellent reinforcement-matrix distribution gave the composite outstanding hardness and tensile strength. The mixture was 25% glass fibre and 35% seashell. The 40% seashell composite had poor mechanical performance, presumably due to decreasing powdered seashell-fibre contact as the filler proportion increased. Owuamanam and Cree (2020) studied polymer composites with seashell and eggshell bio-calcium carbonate. Surface modifiers can improve tensile and flexural strength, according to the study. The review recommended more research in filler loadings and testing these composites' durability.

The literature study highlights various seashell-reinforced polymer composite research gaps. Due to adhesion and agglomeration, seashell fillers lose efficiency as concentrations increase, making optimization difficult. Existing research on the long-term durability and environmental resistance of these composites, notably in humid circumstances, UV radiation exposure, and wide temperature ranges, is lacking. Seashell fillers vary in chemical and mechanical qualities due to species and processing methods. Discrepancies can affect composite performance consistency and reliability. Hence in this work the effect of sea shell waste powder is utilized as a natural filler material in epoxy-based composites. The composite specimens were tested for mechanical and dynamic mechanical analysis.

2. MATERIALS AND METHODOLOGY

2.1 Materials

The experiment utilized Epoxy Resin (Araldite LY 556), a thermosetting polymer known for its better thermal, chemical stability and strong adhesive characteristics. The hardener (HY951) was combined with the resin at a 10:1 ratio. The cured epoxy exhibits remarkable resistance to a wide range of acids, bases, and organic solvents, rendering it highly suitable for application as a protective coating in challenging conditions. The procured material has a tensile strength of 75±8 MPa as per the supplier specifications. This strength can be influenced by the precise formulation and reinforcement components used. The little shrinkage observed during the curing process ensures dimensional stability, which is a critical necessity for precise applications like packaging and outer covers. The hybrid matrix was formed by employing powdered *Vachellia Nilotica* (Babul), a natural resin procured from Vimal Enterprises in Kanchipuram, India.

The organic resin is gathered from the outer surface of Babul tree's bark, dried and then ground into a fine powder by employing a ball mill and the particle size of the powder obtained is between forty to fifty microns. The seashells were applied as the fillers to the epoxy matrix chosen for the application of the epoxy resin. The seashells are chiefly composed of calcium carbonate that includes the mineral calcite and another mineral called aragonite. The sea shell trash is ground into fine powder with a particle size of between 40 and 50 microns by means of ball milling. The incorporation of seashell powders was to improve the mechanical properties of the composite through the incorporation of the impressive hardness and toughness observed in the nanoscale arrangement of seashells. Seashell fillers are incorporated into the epoxy matrix positively affected the mechanical properties, dimensional stability, and solvent resistance of the composites. These were materials ideal for testing applications in automotive, sporting goods, aerospace industries. The hand layup technique was used to prepare the composite specimens with different volume fractions of sea shells used as particle filler. These volume fractions of the sea shell particulate filler were as 3 v/v %, 6 v/v %, 9 v/v %, 12v/v% and 15 v/v %.

The content of *Vachellia Nilotica* was kept constant at 15 v/v%.

2.2 Testing Methods

The composite samples were evaluated for their dielectric, mechanical, and dynamic mechanical properties using standard ASTM techniques. The material's mechanical properties were assessed using an Instron 5567 universal testing machine, with a crosshead speed of 2 mm/min, in accordance with the ASTM D638. The criteria encompassed both tensile strength and modulus. The flexural characteristics were determined using an Instron machine with a three-point bending fixture. The machine functioned at a velocity of 10 mm/min, adhering to the recommended guidelines outlined in ASTM D790. The impact strength was assessed using an impact tester (Ceast Resil Impactor, Italy) by Charpy method following the guidelines set by ASTM D256 standards. The analysis employed the mean outcome derived from evaluating five separate samples.

The SEIKODMAI-DMSC 6100 was used to test the dynamic mechanical parameters of storage modulus (E') and loss modulus (E") in tensile mode. The measurements were performed in accordance with the ASTM E1640 criteria, with a frequency of 1 Hz and a heating rate of 5°C/min. After the viscoelastic tests, the damping factor, Tan δ, was calculated by dividing the storage modulus by the loss modulus. A CEAST 6135 analyser, capable of operating at a maximum voltage of 60 kV, was used to measure the dielectric breakdown voltage of epoxy resin composites containing *Vachellia Nilotica* natural filler. This investigation followed the guidelines specified in the ASTM D149. The dielectric strength was determined by computing the average using a set of five identical specimens. The investigation employed a circular specimen with dimensions of 80 mm in diameter and 3 mm in thickness. The experimental setup involved applying an input voltage of 100 volts per minute to the sample, which was positioned between two circular conductors.

3. RESULTS AND DISCUSSION

3.1 Tensile Strength

The experimental results presented in Fig. 1 to Fig. 3 showed that both *Vachellia Nilotica* and Sea Shells particulate filler had a significant effect on modifying the mechanical properties of epoxy composites. The relationships among these components arise as a result of the remarkable mechanical effectiveness observed across a broad range of filler concentrations. The tensile results given in Fig. 1 1 showed a notable improvement of 8.1% in tensile strength and the maximum tensile strength of 11.9 MPa was observed for the composite with 6 v/v% seashell filler. The effectiveness of the Sea Shells filler is a result of the combined impact of *Vachellia Nilotica* gum and the epoxy matrix, along with the even load transfer facilitated by the Sea Shells. In this study, it was also found that *Vachellia Nilotica* has the ability to act as a natural adhesive as observed from the SEM images. This property allows it to greatly improve the bond between the epoxy matrix and the filler material, resulting in enhanced interfacial adhesion. As the filler loading of Sea Shells increases to around 9 v/v%, a noticeable decrease in tensile strength is observed. The decrease in performance can be linked to the inadequate dispersion and agglomeration resulting from the higher concentration of filler material. Agglomerates can cause poor surface wetting by the matrix, which can result in poor load distribution. However, by improving the adhesive bonding on the surfaces, *Vachellia Nilotica* helps to mitigate some of these unfavourable outcomes.

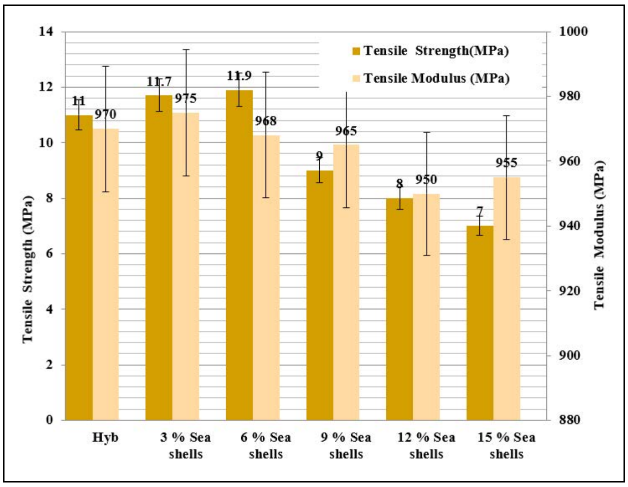

Fig. 1: Tensile strength of Sea shells filler - *Vachellia Nilotica* /epoxy matrix composite

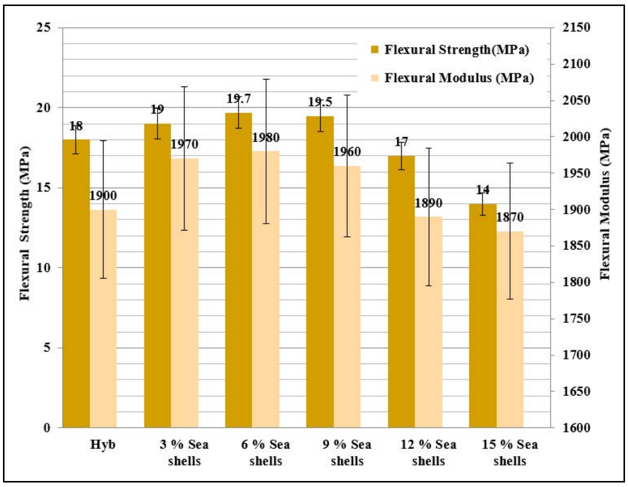

Fig. 2: Flexural strength of Sea shells filler and *Vachellia Nilotica* /epoxy matrix composite

The flexural strength and modulus achieved their maximum value of 19.7 MPa and 1980 MPa at a filler content of 6 v/v% which is showed in Fig. 2. The flexural characteristics show a similar trend to the tensile values. By combining seashells and *Vachellia Nilotica*, the composite's resistance to bending forces is significantly improved. The natural gum improves the adhesion between the epoxy and the Sea Shells filler, resulting in increased stiffness and flexural strength of the composite. The improved distribution of stress within the epoxy matrix is attributed to the enhanced contribution of the natural gum. At higher levels of filler, the decrease in flexural strength is primarily caused by the agglomeration at certain places leading to stress concentration at those points. Fig. 3 shows the impact strength of the epoxy composite. When Sea Shells filler is incorporated, the impact strength is slightly enhanced, resulting in a higher overall impact strength attributed to the presence of *Vachellia Nilotica*. Maximum impact strength value of 0.65 J/m^2 was observed for the composite with 6% sea shells filler. This study explored the role of the gum in dissipating energy during impact and improving the adhesion between filler particles and the matrix. The improvement in the impact strength is substantial, with a 30% improvement in the impact strength over the composite without any filler particle.

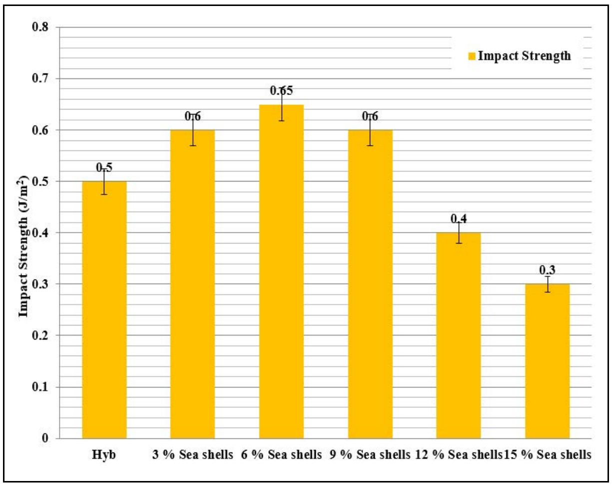

Fig. 3: Impact strength of Sea shells filler *Vachellia Nilotica* (VN)/epoxy matrix composite

3.2 Fractography Observation of Sea shells filler dispersed *Vachellia Nilotica* (VN)/epoxy Composites

Scanning electron microscopy (SEM) is utilised to study the surface morphology of the tested composite specimens. The tensile tested specimens were used for this purpose. Figs. 4 (a) to 4 (d) presents the SEM images taken in this study. The better bonding can be attributed to *Vachellia Nilotica*, as observed in the micrographs when the filler content is at its ideal level of 6 v/v%. The distribution of the Sea Shells filler is uniform which is clear from the images 4 (a), and 4 (b). This study confirms the adhesive capabilities of natural gum and its impact on the formation of a cohesive contact between the epoxy matrix and the filler. The findings reveal that this interaction leads to efficient stress transfer and enhances the mechanical properties of the material. However, when the filler quantity is above 6 v/v% as shown in Figs. 4 (c) and 4 (d), a significant aggregation of the seashell filler and a decrease in bonding within the composite is observed.

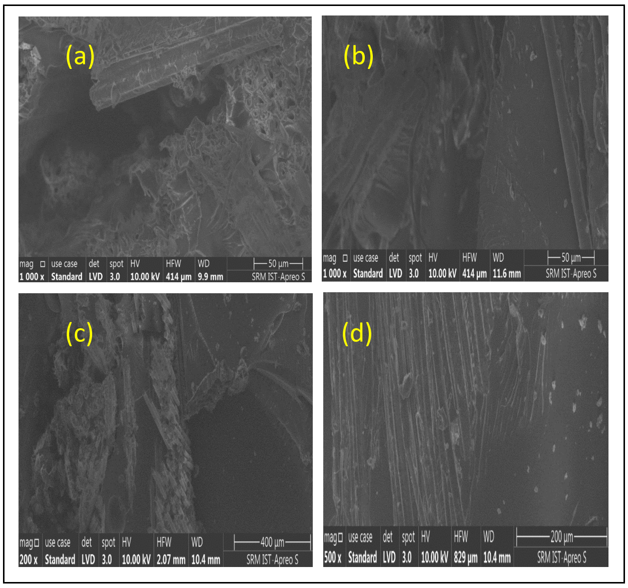

Fig. 4: SEM images of (a) 3%, (b) 6% and (c) 9% (d) 12% of Sea shells filler incorporated *Vachellia Nilotica* (VN)/epoxy-based composite

3.3 Dynamic Mechanical Analysis

As the temperature rises, the polymer chains become more mobile, causing a decrease in the storage modulus. This is a common behaviour seen in viscoelastic materials. However, the composites containing filler demonstrate a noticeably higher glass transition temperature (Tg) of approximately 100°C. As a result, the presence of fillers hinders the movement of polymer chains, causing a delay in the shift from a rigid to a flexible state. This finding is in line with prior research that has demonstrated the ability of natural fillers to improve the stiffness of hybrid composites by limiting the mobility of polymer chains. A vital indicator, known as the damping factor (Tan δ), reveals a material's viscoelastic properties and its ability to dissipate heat under cyclic stress. Figure 6 demonstrates the effect of filler content on the damping properties of epoxy composites reinforced with sea shells and *Vachellia Nilotica*. It is seen that there is a decrease in tan δ with increase in filler content of the composites. The results imply an improved ability to dissipate energy in the composite which was probably due to proper filler/matrix interface adhesion. The interaction is much better in the case of *Vachellia Nilotica* and the filler that is Sea Shells, and these are the factors that have led to the conclusions made in the research. All these components are very important when it comes to the dissipation of energy and the distribution of stress in this composite material. The obtained Tan δ values indicate that the level of energy dissipation decreases if the filler content exceeds 12%, which should be further investigated. This decrease is attributed to reduction in overall surface area which seems to be due to filler particles agglomeration.

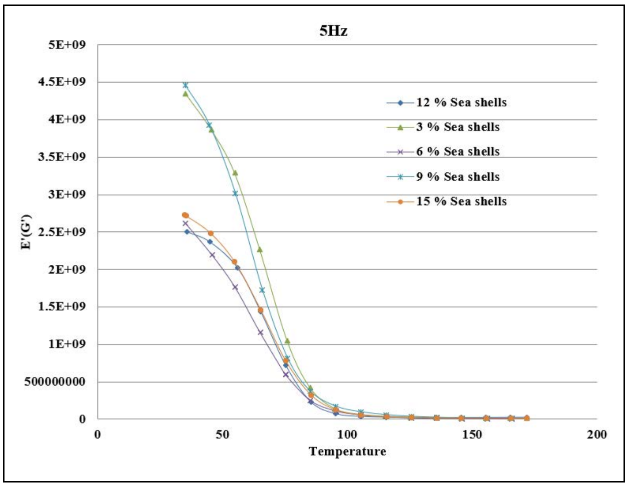

Fig. 5: Storage modulus of Sea shells filler reinforced epoxy and *Vachellia Nilotica* at 10 Hz Frequency

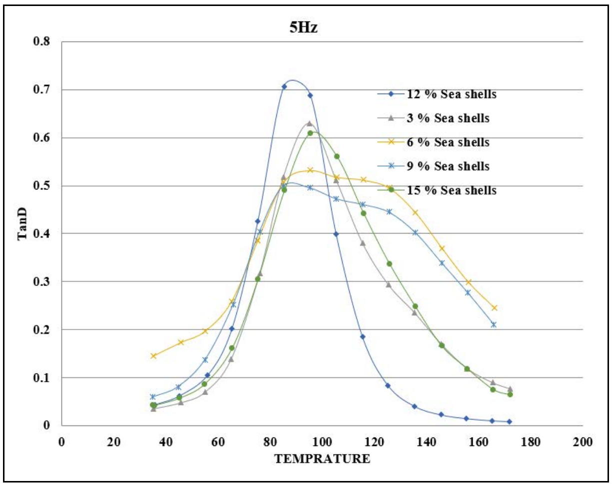

Fig.6: Effect of Tan δ at 10 Hz frequency on Sea shells filler reinforced epoxy and *Vachellia Nilotica* (VN) composite

The presence of particles hinders the flow of stress from the filler to matrix in a smooth manner. Also, the energy distribution of the material is affected by microvoids and imperfection when there is a significant amount of the filler content. In situations where there exists dynamic loading, these imperfections work as regions of stress concentration thereby leading to early failure. The loss modulus of a material describes its capacity to dissipate energy through internal friction during deformation, providing insights into its viscous behaviour. The loss modulus shows variations with changes in the filler content, as shown in Fig. 7. According to the findings, incorporating filler at a concentration of up to 6% is associated with a significant rise in the loss modulus. The findings suggest that there has been an enhancement in the interaction between the epoxy matrix and the Sea Shells filler and *Vachellia Nilotica* gum, which may have led to an improvement in internal friction. In addition, there is a noticeable decrease in the loss modulus as the concentration of filler increases. This trend is consistent with the observations made in Tan δ and the storage modulus. In the presence of higher amounts of filler, the loss modulus experiences a decrease. This can be attributed to the formation of filler agglomerates and the restricted mobility of polymer chains. These elements impede the efficient dissipation of energy.

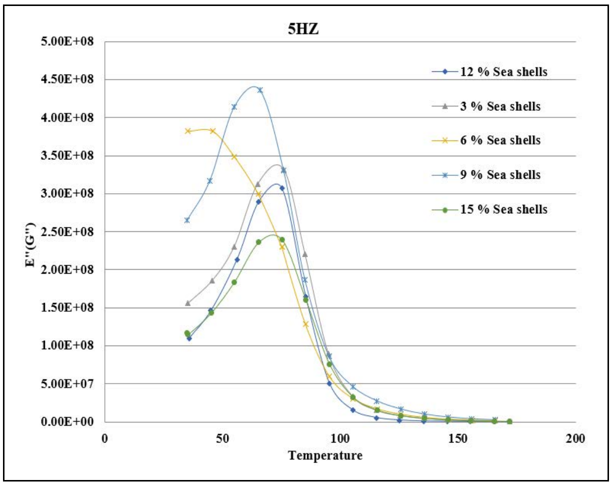

Fig.7: Effect of Loss modules at 10 Hz frequency on Sea shells filler reinforced epoxy and *Vachellia Nilotica* (VN) composite.

3.4 Thermogravimetric Analysis

The thermal stability of the composites can be described using thermogravimetric analysis (TGA), which calculates the amount of weight lost as temperature rises. Table 1 presents a brief overview of the composites' degradation temperatures and residual weights, demonstrating the impact of various filler volumes. The TGA results show that adding *Vachellia Nilotica* and sea shell filler improves the thermal stability of epoxy composites significantly. The composites have higher degradation temperatures than pure epoxy resin, implying that the fillers have a role in delaying the onset of thermal breakdown. This particular enhancement can be attributed to the inorganic composition of the Sea Shells filler. It works as a heat barrier, substantially slowing the degradation of the polymer matrix. The residual weight at the end of the TGA examination reveals a clear link with increasing filler quantities, supporting the hypothesis that the fillers improve the composite's thermal stability. *Vachellia Nilotica*'s strong organic makeup contributes significantly to the formation of char during decomposition, providing an additional layer of protection against heat degradation. Previous research on the thermal characteristics of composites reinforced with natural fillers (particularly seashellpaper-Materials) has found that when the filler loading is 15%, the composite exhibits increased resistance to heat degradation.

Table 1. TGA Analysis

Composition	Degradation temperature (°C)			Residue (%)
	Stage 1	**Stage 2**	**Stage 3**	0.018
Epoxy + 12 Bio filler	702	612	400	0.4
3% *Sea shells* filler + Hybrid EPOXY	704	613	401	0.6
6% *Sea shells* filler + Hybrid EPOXY	706	617	401	0.9
9% *Sea shells* + Hybrid EPOXY	710	621	402	1.4
12% *Sea shells* + Hybrid EPOXY	770	632	403	1.6
15% *Sea shells* + Hybrid EPOXY	777	641	404	1.5

3.5 Dielectric Strength

Table 2 shows the composites' dielectric strength, which indicates their ability to withstand electrical breakdown when subjected to high voltage conditions. Furthermore, the use of Sea Shells filler, particularly at lower filler concentrations, increases the dielectric strength of *Vachellia Nilotica*/ epoxy composites. At a filler loading of 6%, the dielectric strength peaks, probably due to the even distribution of Sea Shells filler inside the matrix, resulting in better interfacial polarity. By achieving a constant distribution, dielectric performance improves greatly because to the Maxwell-Wagner-Sillars (MWS) effect, which efficiently traps charges. Nonetheless, the dielectric strength decreases as the filler level surpasses 6%. The reduction in size is thought to be due to the formation of microscopic void and intrinsic defects induced by the clumping of the particles that fill the substance. These flaws act as isolated locations where electric charges might accumulate, potentially causing early degeneration of electrical systems. Mechanical flaws found at greater filler loadings also contribute to reduced dielectric strength because of their ability to produce electrical failure under strong electric fields. In keeping with previous research, the findings indicate that a high filler content has a negative impact on dielectric performance, increasing the probability of mechanical and electrical failures.

Table 2. Dielectric strength of Sea shells filler incorporated *Vachellia Nilotica* /epoxy matrix composite

Sample ID	*Sea shells* (Vol %)	Bio Filler (Vol %)	Epoxy Resin (Vol %)	Dielectric Strength (kV/mm)
1	0	12	88	22
2	3	12	85	24
3	6	12	82	26
4	9	12	79	21
5	12	12	76	20
6	15	12	73	17

5. CONCLUSION

The studies show that adding Sea Shells filler to composites can improve their mechanical properties, with tensile strength improving by up to 9 vol%. The presence of filler agglomeration reduces mechanical strength because it interrupts matrix continuity and decreases interfacial bonding, especially when filler volume is more than 9%. Improving the composite's interfacial adhesion leads to increased energy absorption and dispersion upon impact, as well as improved tensile characteristics. The SEM testing showed that the homogeneous filler dispersion at 9 vol% improves toughness and energy absorption. Dynamic Mechanical Analysis (DMA) reveals that the inclusion of VN and Sea Shells filler greatly improves the composite's rigidity. This is reflected in the greater glass transition temperature (Tg) and enhanced storage modulus. The damping factor reaches its maximum at 9 vol% filler, indicating the better level of energy dissipation.

According to TGA studies, this work demonstrated the possibility for improving thermal characteristics, as indicated by greater thermal stability at increasing filler concentrations (up to 9 vol%). This finding has important implications for applications that require high thermal resistance. The dielectric strength reaches its peak at lower filler levels (6 vol%) due to effective charge.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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