



Research article

Effect of bioderived calcium carbonate/thermal cycling treatment on morphology and mechanical properties of reinforced epoxy biocomposites

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ABSTRACT

This study assessed the potential of snail shell (SSs) derived calcium carbonate (CaCO_3) particles as reinforcements in epoxy-based composites (ESS) and as well investigate the effects of cyclic thermal treatment and SSs content on the mechanical and physical properties. The composites were developed through stir casting using an open mold technique after producing the snail shell particles (SSp). After curing, parts of the developed samples were post-cured by subjecting them to cyclic thermal treatment and both sets of samples were tested and characterized in accordance with the relevant standards. Analysis of the results revealed that, thermal cycling treatment and 10–20 wt% reinforcement content were the most suitable conditions/parameters for the development of snail shell reinforced epoxy composites for structural applications. The incorporation of SSp into epoxy-based composites significantly improved mechanical properties, with 20 wt% treated SSp (ESS20) yielding the highest tensile strength (4.56 MPa) that culminated to a 94% increase over the treated control while in the untreated condition, the ESS20 was with 260.3% over untreated control sample (ESS0). Flexural strength was at peaked for 25.3 MPa in untreated ESS20 while treated composites showed a maximum flexural modulus of 0.75 GPa at ESS10. Impact strength was highest in ESS10 (1.82 kJ/m²) for untreated and ESS20 (2.09 kJ/m²) for treated composites. Hardness was at maximum at ESS20 (65.6 HS) for untreated and ESS15 (65.4 HS) for treated. Water absorption was limited by the thermal cycling treatment carried out on the samples while the density was enhanced as the SSp content increases in addition to thermal treatment. The use of SSp as a reinforcing filler also presents significant environmental and economic advantages worth considering for real-world applications. These attributes make SSp reinforced composites promising for low-cost-sensitive industries such as construction, packaging and automotive.

1. Introduction

In response to the growing emphasis on sustainable development and ecological demands, there are several efforts to develop composites from several natural materials [1–3]. Concerns about environmental pollution have also heightened the need for the scientific community and the

public to oppose non-biodegradable composites strongly [4]. Numerous industries have been utilising composites for various applications, including food, textiles, water treatment, chemicals, cosmetics, and nanofillers, among others [5,6]. As a result, naturally occurring fibres (NFs) and mostly biodegradable polymer-based composites emerge as viable alternatives for producing environmentally friendly materials [7,

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8]. For instance, a biodegradable polymer combined with biological or biodegradable nanofillers to create biodegradable nanocomposites [9–12]. Numerous studies have demonstrated the successful utilisation of biomass waste-derived NFs as fillers in polymeric composites [13–15]. Notable examples include rice husk [16–18], wood [19,20], coconut shell [21–23], fish scale [24–26], curaua [27], sisal [28], and peanut shells [29]. These fibres are characterised by their properties such as strength, low density, and cost, among other favourable properties. The fact that they are also readily available at low cost makes them acceptable for use in several areas of application.

Researchers have explored various natural fibres (NFs) to reinforce polymeric matrices and create composites with enhanced properties [30]. NFs can be classified as organic and inorganic fibres, and researchers have studied organic fibres such as curaua, cellulose, algine, sisal and bamboos [5,31]. Also, inorganic fillers such as eggshells, snail shells, shellfish, oyster shells, mollusc shells, etc., have been utilised by researchers as reinforcement in composites due to the presence of high calcium carbonate (CaCO_3) content [32,33]. Calcite-based bio-fillers have been discovered to enhance the mechanical and other properties of developed composites [34–36]. Among the inorganic fibres, snail shell particles have emerged as one of the highly effective reinforcement materials in polymer composites. They are noted for significant enhancements in tensile strength, hardness, and other mechanical properties compared to traditional fillers like coconut shell ash [37]. Snail shells are primarily classified as ceramic-based biogenic waste. They are characterised by their unique compositions that offer several functional advantages. Their inherent hardness and stiffness make them well-suited for reinforcement applications where rigidity and dimensional stability are critical [38]. Also, compared to lignocellulosic fillers, snail shells exhibit greater thermal stability and lower hygroscopicity, reducing the risk of moisture-related degradation in composite materials. Additionally, they present a low-cost, abundant, and sustainable alternative to mined CaCO_3 fillers without sacrificing performance [39]. Unlike synthetic or commercially processed CaCO_3 , snail shell particulates retain a biologically derived microstructure and morphology that may enhance interfacial interactions within an epoxy matrix. These properties distinguish snail shells from both plant-based and conventional mineral fillers, making them a promising material for sustainable composite development [40].

Studies have shown that smaller particle sizes and optimal filler content (typically around 10–20 wt%) lead to notable improvements in tensile strength, stiffness, and impact resistance [41–44]. Due to their high calcium carbonate content, snail shells contribute significantly to mechanical performance enhancement while also serving as an eco-friendly alternative material [45]. Their application extends to interior automobile components and wastewater treatment, particularly for the removal of heavy metals and textile dyes, thus aligning with growing environmental regulations that prioritise biodegradable and sustainable solutions [46,47]. Therefore, snail shell particles represent a promising, low-cost, and sustainable option for pollution remediation efforts [48,49].

Moreover, epoxy resin has gained significant attention in modern composite industries [50,51]. Known as a thermosetting resin, it offers valuable properties such as excellent adhesiveness, compatibility with various curing agents, and superior mechanical and corrosion resistance characteristics [52–54]. Due to its exceptional performance, epoxy is widely used for coatings, electrical insulation, adhesives, and structural materials, as well as in industries where composite materials are often required. However, its cross-linking capability due to its molecular structure often resulted in inherent brittleness and poor crack-propagation resistance [55–57]. These limitations may cause characteristics such as brittleness and low impact strength, which have restricted its application in coatings when used as an engineering composite [58]. To address these issues, extensive efforts have been made to enhance epoxy properties through practical curing approaches. Studies have shown that incorporating a unique material, such as an

inorganic component, into an epoxy matrix significantly improves its impact strength [59,60].

Building on this background, this work investigates the effects of snail shell-derived calcium carbonate particles and a thermal post-curing process on the mechanical, physical, and morphological properties of epoxy-based biocomposites. This approach evaluates how the thermal post-curing process can be effectively applied to enhance the overall performance of epoxy composites.

Various thermal treatments have been widely used for structural modifications and enhancement of properties in metals and ceramics, as well as the quality of their products. For instance, annealing, normalising, hardening, nitriding, and tempering are generally done on ferrous metals in a single, repeated, or cyclic form [61,62]. However, limited information is available on the use of cyclic thermal treatment for polymer-based composites. Hence, this research examines the potential of cyclic thermal treatment in enhancing the properties of SSP reinforced composite for advanced materials applications. A series of characterisations was conducted, including mechanical tests (tensile, flexural, impact, and hardness), physical assessments (water absorption and density), and structural/morphological analyses (SEM and XRD). These investigations provided systematic insights into the structure-property relationships of the developed composites under both treated and untreated conditions. Therefore, the research contributes to the growing exploration of non-fibrous bio-wastes in advanced composite applications.

2. Experimental

2.1. Materials

Snail shells, as shown in Fig. 1(a), were sourced from a farmland in Akure, Ondo State, Nigeria. The epoxy resin and hardener were obtained from Pascal Scientific, a local vendor in Akure, Ondo State, Nigeria.

2.2. Processing of the snail shell

The SSs were soaked in water for 24 h and subsequently washed with ethanol to remove residual organic matter and adherent hard particles. The cleaned SSs were then oven-dried at 110°C for 12 h until completely dry. Once dried, the SSs were crushed using a pulverizing machine to obtain fine powder particles, as shown in Fig. 1(b). The crushed material was then sieved using a $450\ \mu\text{m}$ sieve to isolate smaller-sized particles.

2.3. Epoxy-snail shell composites production

The epoxy-snail shell (ESS)-based composites were prepared using epoxy resin and hardener (mixing ratio 2:1) and varying amounts of SSP within 5–20 wt%, as shown in Table 1. The mould was then prepared and coated with a mould release agent to facilitate the easy release of the solidified samples. The production started with measuring 100 g of epoxy resin and 50 g of hardener, which were poured into a 500 mL beaker. The SSP were added to the beaker in the right proportion to prepare the composite samples. The mixture was manually stirred using a glass rod for 5 min to ensure a uniform mixture and was poured into the mould till it was filled. The samples were allowed to cure at an ambient temperature of $24 \pm 1^\circ\text{C}$ and were carefully extracted from the moulds. The developed samples were then categorized into two groups: Group A, representing untreated samples, and Group B, representing thermally treated samples.

Group B samples have the same composition as Group A but were subjected to cyclic thermal post-curing treatment in an oven to induce the samples. The cyclic thermal treatment was carried out by heating at 100°C for 3 h, followed by cooling within the oven and repeated for 7 days, with the same procedure applied daily to maintain uniformity. The oven set-up and schematic of the cyclic thermal treatment were as

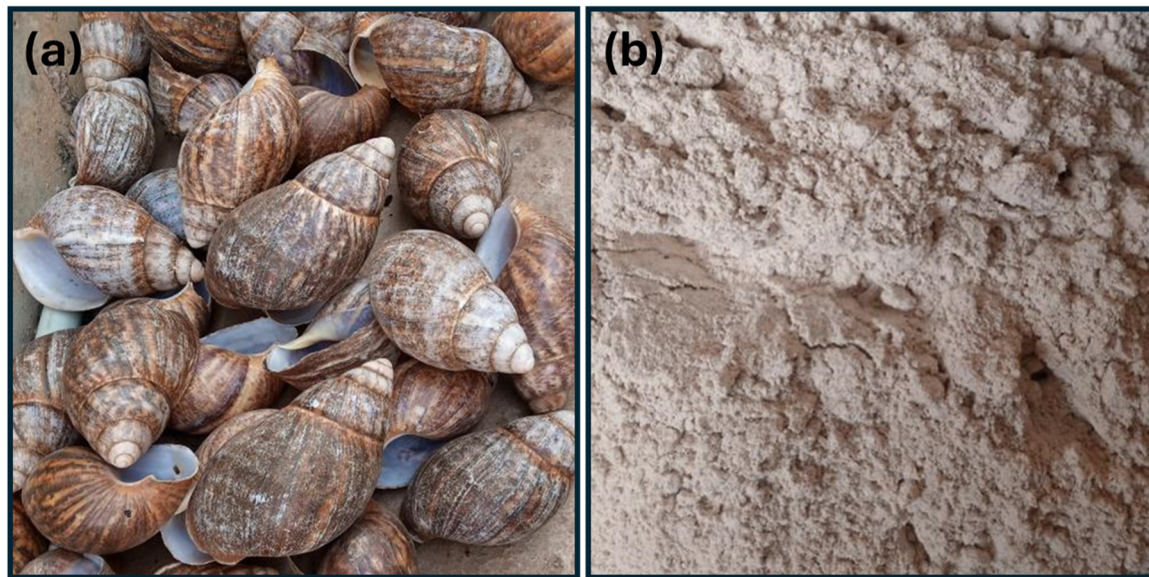


Fig. 1. (a) As-received SSs (b) Cleaned and Crushed SSs.

Table 1
Formulation of Epoxy-Snail Shell Composites.

Samples	Epoxy resin and Hardener (wt%)	Snail Shell (wt%)
ESS0	100	0
ESS5	95	5
ESS10	90	10
ESS15	85	15
ESS20	80	20

shown in Fig. 2(a-b). The thermal cycling regime procedure was selected to simulate accelerated thermal aging conditions that epoxy composites may encounter during service, particularly in sectors such as automotive, marine, and structural applications, where materials are frequently exposed to fluctuating but sub-glass-transition temperatures, potentially leading to stress, fractures, cracks, and abrasion, to represent service conditions as noted in the literature [63]. In contrast, treatments like chemical functionalization, alkaline treatment, and plasma treatment are designed to improve adhesion by modifying the filler surface to promote better bonding with the matrix [64]. Unlike these methods, thermal cycling may induce microcracks and interfacial stresses, making it less effective for improving filler-matrix bonding but valuable for evaluating long-term performance.

2.4. Test and characterization

2.4.1. Mechanical tests

The mechanical tests were conducted at the approved Center for Energy Research and Development, Obafemi Awolowo University of Technology, Ile-Ife, Osun State, Nigeria. The tensile properties of the composites were determined using a Universal Testing Machine (Instron Series 3369) in accordance with the ASTM C1557 standard. Dumbbell-shaped specimens with dimensions of $115 \times 30 \times 3$ mm were prepared for the test, while a 10 kg load cell and a crosshead speed of 5 mm/min were utilized. Three specimens were tested for each classification, and the average values were used.

The flexural properties of the composites were evaluated using a three-point bending test on a Universal Testing Machine according to the ASTM D790 standard. The specimens were positioned in a three-point bending fixture and tested at a displacement rate of 10 mm/min. Three samples were tested for each classification, and the mean values were used.

The impact strength was assessed using a Charpy V-notch impact testing machine (Instron CEAST 9050) according to the ISO 179 standard. Specimens with dimensions of $64 \times 11 \times 3$ mm were notched at the center and placed horizontally on the machine, with a 60 mm span between supports. The test involved releasing a suspended pendulum to fracture the samples. The initial and final gauge readings were recorded,

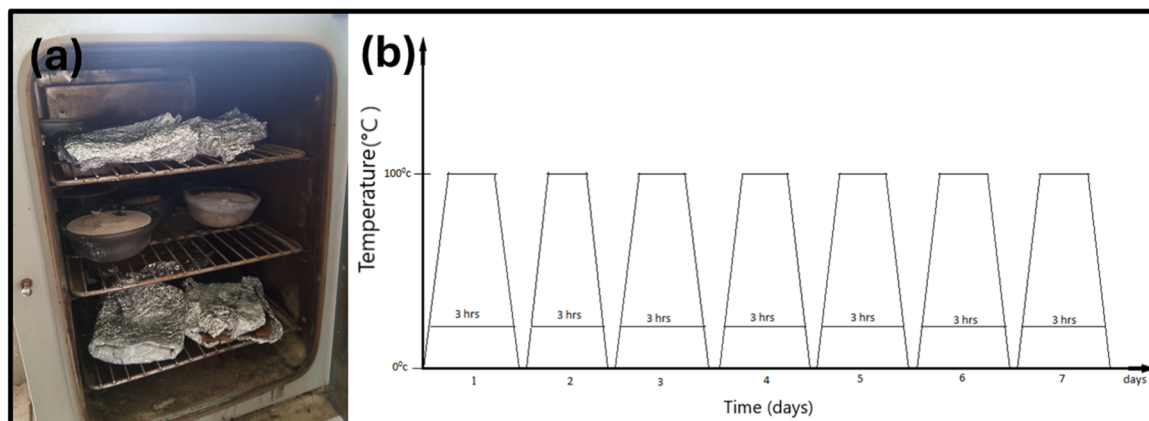


Fig. 2. Thermal Treatment Cycle (a) Oven Set-up (b) Schematic of the Thermal Cycle.

and the impact strength was calculated. Three specimens per classification were tested, and the average values were documented.

The hardness of the specimens was measured using a Shore D hardness tester at the Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Ondo, Nigeria. Each sample was placed on the tester's stand and indented at 15 points. The average of 15 readings was calculated and used for analysis.

2.4.2. Water absorption tests

Water absorption tests were conducted in accordance with the ISO 175 standard. 250 cm³ of water was poured into clean plastic containers for the test. The initial weight of each sample was measured using a chemical weighing balance (FA2104A model, precision: 0.0001 g). The samples were immersed in water, and weight readings were recorded at seven-day intervals over 30 days. Before each measurement, the samples were removed, wiped with a clean cloth, and then weighed. The percentage of water absorption was calculated using the collected data and the standard in Eq. 1.

$$W(\%) = \frac{W_b - W_d}{W_d} \times 100 \quad (1)$$

where W is the water absorption percentage, W_d and W_b are the dry weight and the weight of the specimen after time t, respectively.

2.4.3. Density measurements

The density of the samples was determined by cutting them into pieces with a 4 mm diameter. A Quartz Rock Density Tester (AU-300R) was used to measure density, with the machine automatically determining the weight and volume of each specimen.

2.4.4. X-ray diffraction (XRD) Characterization

The X-ray diffraction (XRD) analysis of the SSp was carried out using an X-ray diffractometer with Cu-K α radiation ($\lambda = 1.544 \text{ \AA}$). The scattered radiation was recorded over a 2θ range of 5° to 40° at a scanning rate of 0.02° per minute. The crystallinity index (CI) was determined using Eq. 1, where CI represents the relative degree of crystallinity, I_{200} is the intensity of the peak corresponding to the (200) plane, and I_{am} is the intensity of the diffracted peak at $2\theta = 18^\circ$. Eq. 2 shows the calculation for the crystallinity index (CI).

$$CI(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (2)$$

2.4.5. Scanning electron microscopy (SEM)

The surface morphology was analysed using a Scanning Electron

Microscope (SEM) model from Oxford Instruments, TESCAN S8000, Kohoutovice, Czech Republic. Before analysis, the samples were affixed to individual holders and subsequently coated with approximately 10 nm thin sputter coating of high-purity gold (AU10) to improve conductivity and prevent charging during SEM imaging using a Quorum Q150T ES from Quorum Technologies Ltd., Laughton, UK. This was based on findings established in prior studies [65]. To minimize chaging effects observed during imaging, the samples were treated with an additional slight silver coating between the samples and the holder to improve electrical charging. Although minor variations in scaling and magnification occurs to obtain clearer and interpretable morphological images.

3. Results and discussion

3.1. Tensile properties snail shell particle filled epoxy composites

Fig. 3(a) shows the influence of SSp particle content and cyclic thermal treatment on the tensile strength of epoxy composites, while Fig. S1 shows the stress–stress-strain curve of the treated and untreated samples. For untreated samples, tensile strength values increased from 0.63 MPa (ESS0) to 1.44 MPa (ESS5), 1.57 MPa (ESS10), 1.72 MPa (ESS15), and peaked at 2.27 MPa (ESS20), marking a 260.3% increase compared to ESS0. Likewise, thermally treated samples showed a similar trend with higher values of 2.35 MPa (ESS0), 3.61 MPa (ESS5), 1.36 MPa (ESS10), 3.11 MPa (ESS15), and a maximum of 4.56 MPa (ESS20), which culminated in 94.0% improvement over the control (ESS0). The enhancement is attributed to the optimal distribution and critical length of SSp in the matrix [66]. Although this study did not explore SSp content beyond 20 wt%, existing literature suggests that higher filler loading may lead to reduced performance due to particle agglomeration and poor dispersion. Thus, the results showed that 20 wt % is the optimum reinforcement content for enhanced tensile strength both at treated and untreated conditions. By attaining higher tensile strength after the treatment, it shows that this treatment parameter has impacted the structural morphology and formation that aid higher resistance to tensile stress.

Likewise, Fig. 3(b) shows the tensile modulus of epoxy composites with and without thermal treatment at various SSp loadings. For untreated samples, modulus values were 0.71 GPa (ESS0), 0.65 GPa (ESS5), 0.67 GPa (ESS10), 0.71 GPa (ESS15), and 0.52 GPa (ESS20). Treated samples had values of 0.62 GPa (ESS0), 0.55 GPa (ESS5), 0.66 GPa (ESS10), 0.61 GPa (ESS15), and 0.64 GPa (ESS20). In most cases, treated samples showed lower tensile modulus than untreated ones, likely due to filler agglomeration and limited load transfer, which

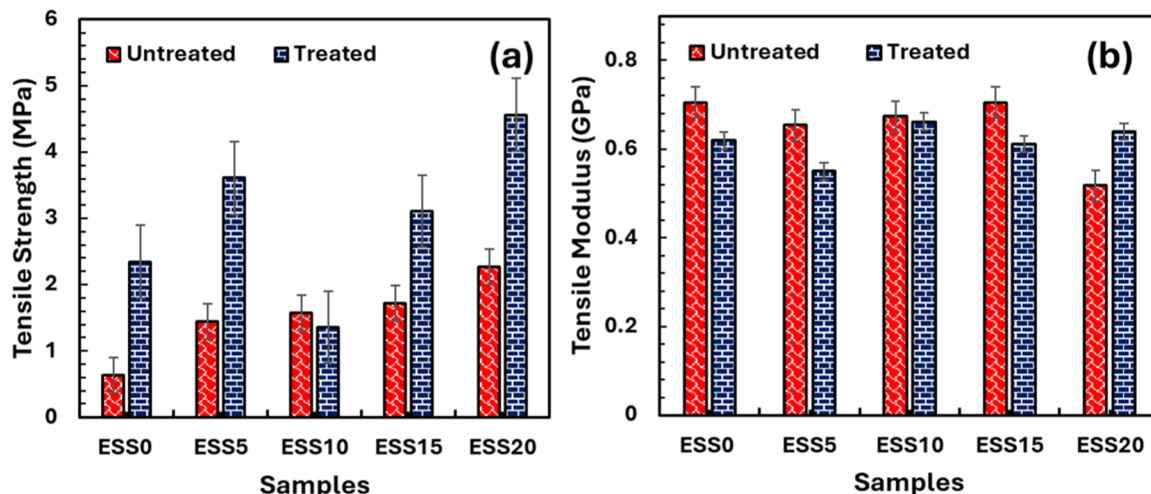


Fig. 3. Tensile (a) Strength (b) Modulus of Treated and Untreated Samples.

can initiate cracks. The reduced modulus in the treated ESS0 is also linked to post-treatment weakening. However, at 20 wt% (ESS20), the treated sample showed a higher tensile modulus than the untreated one, indicating improved filler-matrix interaction at this concentration. Similar to tensile strength results in Fig. 3(a), the treated sample with 20 wt% content was the one with the optimum modulus value compared to the control and the untreated at the same content. Though lesser values were observed compared to untreated samples in some cases, this may be due to structural transitional effects. However, at the optimum value, the treated sample was the best when compared with both the control and untreated samples. Thus, the treatment has positively impacted the tensile properties of the composites. Having discovered this and, based on the roles of tensile strength on materials for structural applications, this method will be suitable for modifying tensile properties of epoxy-based composites and other related polymer-based composites.

3.2. Flexural properties of snail shell particle-filled epoxy composites

Fig. 4(a) shows the flexural strength of epoxy composites with varying SSp content, before and after thermal treatment, while Fig. S2 shows the flexural strain curve from the three-point bending test of the treated and untreated samples. Untreated samples had flexural strengths of 16.8 MPa (ESS0), 18.2 MPa (ESS5), 22.1 MPa (ESS10), 23.8 MPa (ESS15), and 25.3 MPa (ESS20), with the highest strength at 20 wt% SSp. Treated samples showed strengths of 24.2 MPa (ESS0), 17.3 MPa (ESS5), 17.4 MPa (ESS10), 16.2 MPa (ESS15), and 16.2 MPa (ESS20). Except for ESS0, flexural strength decreased after treatment, likely due to reduced stiffness and weaker interfacial bonding caused by the thermal treatment and polymer degradation. Previous studies indicate composites with calcium carbonate fillers show slightly higher flexural strength than those with stainless steel SSp, where the rigid SSp restricts epoxy mobility, increasing cross-link density and improving load transfer between matrix and filler [67,68].

Fig. 4(b) presents the flexural modulus of epoxy composites with different SSp contents. For untreated samples, the modulus dropped sharply from 0.54 GPa (ESS0) to 0.34 GPa (ESS5), then gradually rose to 0.43 GPa (ESS10), 0.46 GPa (ESS15), and 0.5 GPa (ESS20), likely due to reduced strength and rigidity. In contrast, treated samples showed an initial increase from 0.57 GPa (ESS0) to 0.64 GPa (ESS5), peaking at 0.75 GPa (ESS10), followed by a decrease to 0.72 GPa (ESS15) and 0.6 GPa (ESS20). The results revealed a positive impact of the treatment compared to the untreated and control samples, where 10–15 wt% was seen to possess higher values. The implication of this was that, there is adequate and effective load transfer between epoxy and fillers, while the

later decline may be attributed to polymer degradation and reduced stiffness as observed in the tensile modulus and flexural strength properties in Figs. 3b and 4(a), respectively. Nonetheless, SSp improve the flexural properties of epoxy composites at moderate loadings due to good interfacial bonding with the matrix and structural compatibility [69,70].

3.3. Impact strength of snail shell particle-filled epoxy composites

Fig. 5(a) shows the impact strength of SSp reinforced epoxy composites for both treated and untreated samples. It was discovered that the treatment gave enhancement to the samples, both at the control and reinforced samples, at a peak value of 20 wt% content compared to the untreated samples. The treated control ESS0 sample had an impact strength of 2.39 kJ/m² while it was 2.09 kJ/m² at ESS20. This improvement is due to strong filler-matrix adhesion, which enhances resistance to crack propagation and energy absorption. The decline in impact strength due to the inclusion of filler contents may be attributed to many factors, such as polymer degradation, poor filler-matrix interaction, micro-crack formation, and weakened matrix cohesion [45].

3.4. Hardness of SSp filled epoxy composites

The hardness of the SSp reinforced epoxy composites is shown in Fig. 5(b), where it was discovered that the hardness tends to increase as the reinforcement content increases. The hardness value of the untreated ESS0 was 62.5 HS while for SSp-filled composites with optimum value was ESS20 (65.6 HS). Likewise, for treated samples, ESS0 was 64.7 HS while the optimum value was achieved at ESS15 (65.4 HS). The result revealed that thermal treatment actually enhanced the hardness of both control and reinforced samples. Optima values were attained at 20 wt% for untreated-based composites, while it was obtained at 15 wt % for treated composites. The increase is potentially due to uniform dispersion of SSp in the epoxy matrix, reduction in the interparticle distance, and good interfacial adhesion that led to an increase in the resistance of the composites to surface indentation.

3.5. Density of snail shell particle-filled epoxy composites

The density of the SSp reinforced epoxy composites is shown in Fig. 6, where the density values are calculated for SSp-filled epoxy composite materials. The bars illustrate a clear trend of an increasing density as the reinforcement content increases. An increase in fiber content leads to a corresponding rise in the composite's density. The highest recorded density value was at ESS20 with a value of 1.27 g/cm³.

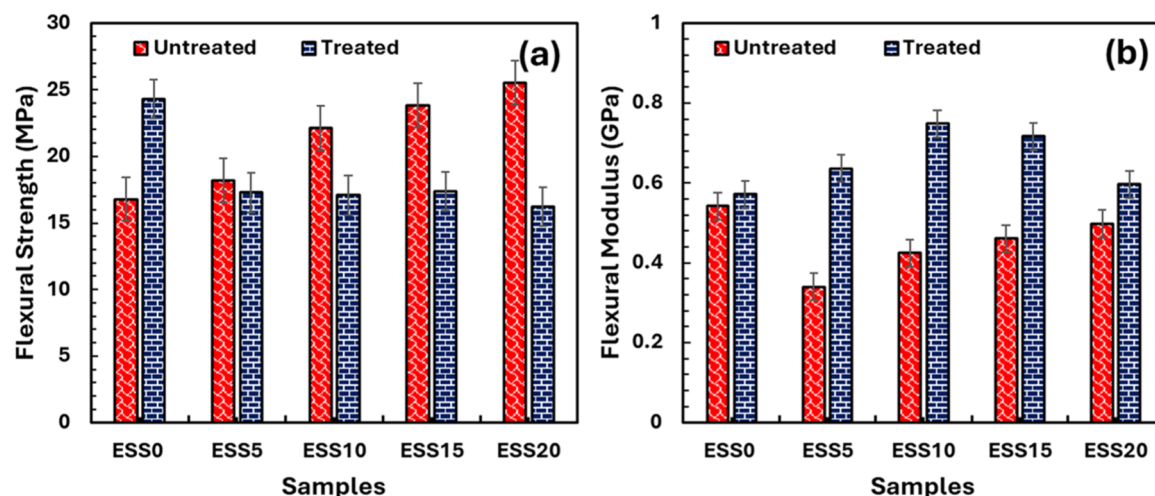


Fig. 4. Flexural (a) Strength (b) Modulus of Treated and Untreated Samples.

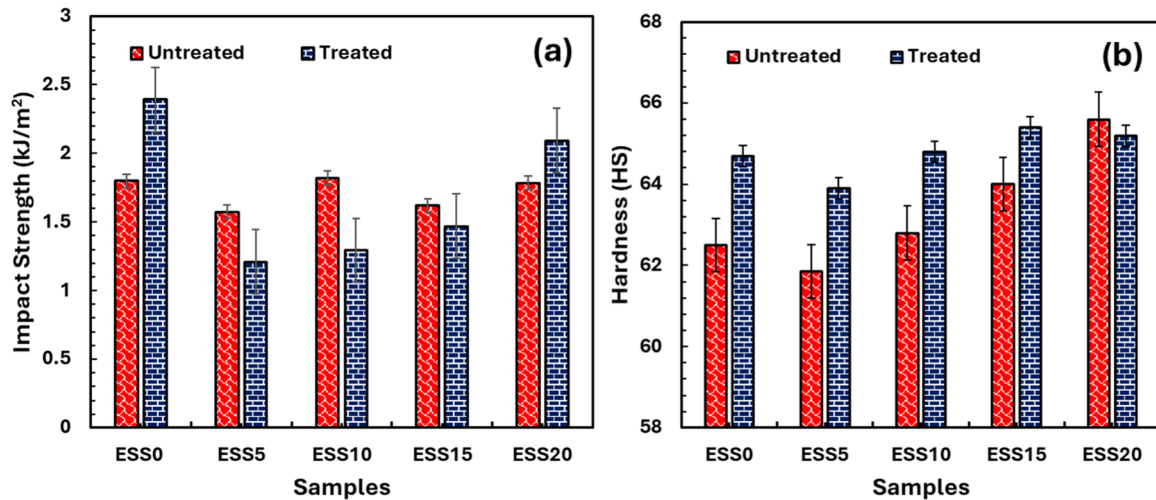


Fig. 5. (a) Impact Strength (b) Hardness of Treated and Untreated Samples.

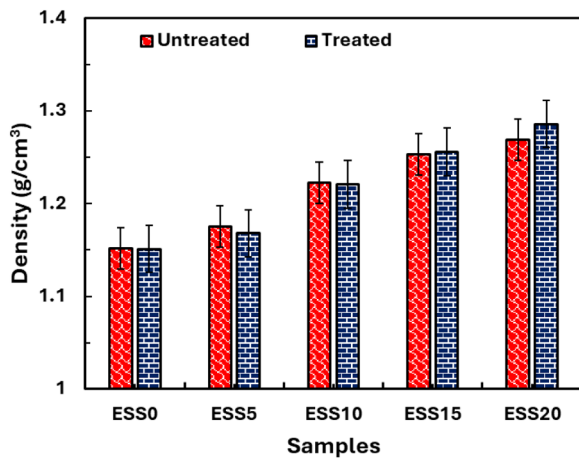


Fig. 6. Density of Treated and Untreated Samples.

(untreated) and ESS20 with a value of 1.29 g/cm³ (treated), while the lowest values were at ESS0 (1.15 g/cm³) for untreated and ESS0 with a value of 1.15 g/cm³ for treated. This increase in composite density with higher fiber content can be attributed to the fact that the fiber material is heavier and denser than the matrix material. As the fiber content increases, the overall density of the composite rises accordingly. Likewise,

the density tends to increase for treated samples as the reinforcement content increases, leading to densification, which might be due to closure or filling of possible pores.

3.6. Water absorption of snail shell particle-filled epoxy composites

Water absorption testing is crucial for evaluating the water absorptivity of composite materials. Fig. 7 illustrates the effect of SSp on the water absorption behavior of treated and untreated SSp-filled epoxy composites as immersion time increases. The data show that the moisture absorption rate increases with an increase in fiber loading and time. This behavior is primarily influenced by the composite's density and void content. Fig. 7(a) showed a wide spread in the absorption at higher values for all the samples for the untreated, while Fig. 7(b) presented a more compacted spread of samples at a low water absorption rate. This showed that the treated samples had fewer pores/voids for water absorption. However, treated composites (ESS20) showed the highest absorptivity and positively correlated with an increase in absorption as particulate content increases. The lowest water absorption rate was observed in ESS0.

3.7. Surface morphology and elemental composition of snail shell particle-filled epoxy composites

The surface morphology and elemental composition of the SSp were investigated using SEM and XRD, respectively. The SEM image shows

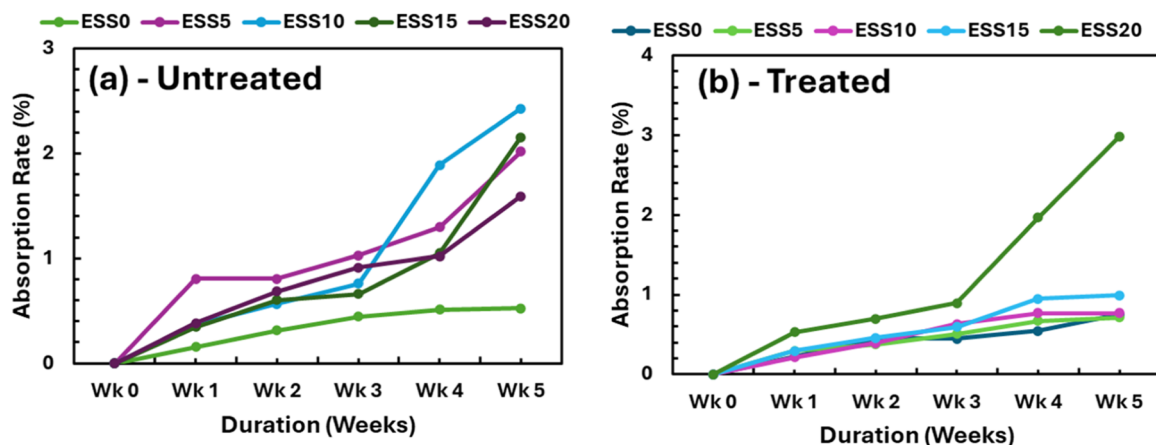


Fig. 7. Water Absorption Rate (a) Untreated (b) Treated Samples.

the particle size of the SS_p, which shows random particle distribution as shown in Fig. 8(a), while Fig. 8(b) illustrates the plot of intensity versus angle (2 θ) for the XRD analysis of SS_p. The SS_p exhibits numerous peaks, indicating the presence of multiple phases and one predominantly detectable phase that signifies a more crystalline structure. In the SS_p, the primary phase corresponds to aragonite, with diffraction peaks observed at 26.5°, 33.2°, 36.5°, 38.2°, 46.0°, 48.2°, 52.5°, and 76.8°, as referenced by the JCPDS card 00–041–1475. These findings indicate that the SS_p consists of calcium carbonate (CaCO₃). The high concentration of the ceramic CaCO₃ phase in SS particles suggests that the African giant land snail shell could serve as a cost-effective alternative source of CaCO₃ material for composites development.

The dispersion of SS_p in the epoxy matrix was examined using SEM analysis of the fractured surfaces of the cured epoxy/SS_p composites. The SEM images of ESS5 and ESS10 (treated and untreated) are shown in Fig. 9, and ESS15 and ESS20 (treated and untreated) in Fig. 10. From the literature [66]. It was noted that the pristine ESS0 sample exhibited a smooth surface. However, the SEM analysis observed that its brittle nature resulted in visible cracks; as studies show that some biodegradable composites often exhibit complex imaging phenomena arising from conditions such as surface heterogeneity and charging effects; this case was also observed in our developed composites [71–73]. All the samples are observed to contain voids, which can serve as water traps, resulting in more water absorptivity and reduced impact strength.

ESS5 denotes a low content of SS_p with high voids in the untreated sample, which was reduced after treatment. The presence of voids in the untreated sample may be due to the reactions that are taking place between the SS_p and epoxy matrix, as well as entrapped air during production. At higher SS_p reinforcement, such as ESS10, ESS15, and ESS20, the fractured surfaces of the composites appeared rougher. This roughness and surface damage could be attributed to good interaction between the SS_p and the epoxy matrix, leading to higher resistance to the applied stress. A large amount of SS_p was noticed in the untreated ESS15, which might be part of the reasons for enhanced properties as the reinforcement content increases. Comparing the treated with the untreated composites, it was noticed that the treated composite possessed a smoother surface. A detailed inspection revealed the presence of microcracks. Additionally, at higher SS_p loadings, particularly at ESS20, the presence of SS_p was more pronounced and had a noticeable impact on the fracture morphology observed. Likewise, untreated samples exhibited relatively smoother fracture surfaces with more uniformly distributed particles, suggesting better interfacial bonding and reduced agglomeration. Also, in the treated ESS20 sample, the SEM images revealed rougher fractured surfaces, irregular crack propagation paths, and localized areas with large SS_p clusters. These features aid good stress distribution tendencies and ensure delayed fracture mechanisms, hence, revealing ductile fracture tendency. Thus, these features contributed to the enhanced mechanical and physical properties of the

materials at this content and treatment condition used. Hence, higher filler content directly influenced the fracture morphology by promoting interfacial debonding, reducing void formation, and brittle failure characteristics. Notably, all the samples exhibited fine dispersion of SS_p incorporated into the epoxy matrix, while the pores/voids were reduced after the thermal treatment. These findings suggest strong interfacial bonding between the epoxy matrix and SS_p. Effective interfacial bonding ensures uniform dispersion of load from matrix to fillers, contributing to improved composite performance.

3.8. Benefits of the research

Beyond the demonstrated mechanical and morphological enhancement shown in this work. The incorporation of SS_p as a reinforcing filler also presents significant environmental and economic, and practical benefits. SS_ps are widely available agro-waste byproducts, especially prevalent in regions with high snail consumption, such as parts of Africa and Asia. Their valorization as functional fillers also aligns with current trends in circular economy frameworks, which emphasize waste-resource strategies and material sustainability across. From an environmental perspective, the use of SS_p contributes to landfill reduction and supports resource-efficient composite designs. This approach is consistent with the broader movement in materials science towards bio-based and renewable fillers, as seen in the growing interest in agricultural waste-derived reinforcements like eggshells, seashells, and bone ash. These biogenic fillers are increasingly being used as a sustainable alternative to mined mineral fillers, such as calcium carbonate and talc, which are energy-intensive to extract and process.

Economically, SS_p provides a low-cost, locally sourced alternative, reducing dependency on imported materials and enhancing the material sovereignty of low-middle-income regions. The relatively simple and energy-efficient processing methods, such as washing, drying, grinding, and sieving, make it feasible for scalable production, even in resource-constrained environments. These attributes make SS_p reinforced composites promising for cost-sensitive sectors, industries such as construction, packaging, and automotive interiors, where affordability, lightweighting, and moderate performance are critical.

While this study focuses primarily on the materials characterization and property-performance relationship, future work should include a comprehensive life cycle assessment (LCA) and techno-economic analyses to quantify the environmental footprint and economic viability of SS_p composites across their lifecycle. Incorporating sustainability metrics such as embodied energy, carbon footprint, and end-of-life scenarios is becoming a key requirement in the development of next-generation composites.

Furthermore, the use of animal-derived materials such as snail shells in composite manufacturing raises important considerations around ethical sourcing and biosafety. From an environmental standpoint, snail

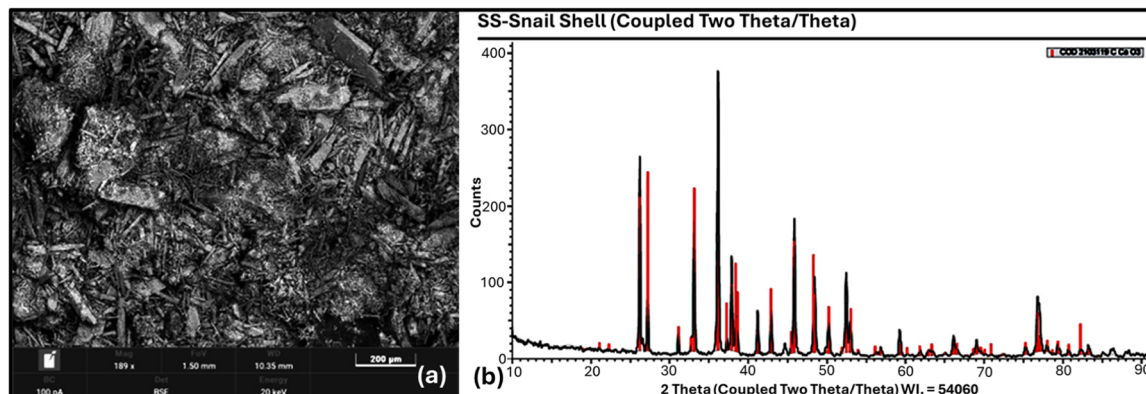


Fig. 8. SS_p (a) SEM (b) XRD Characterization.

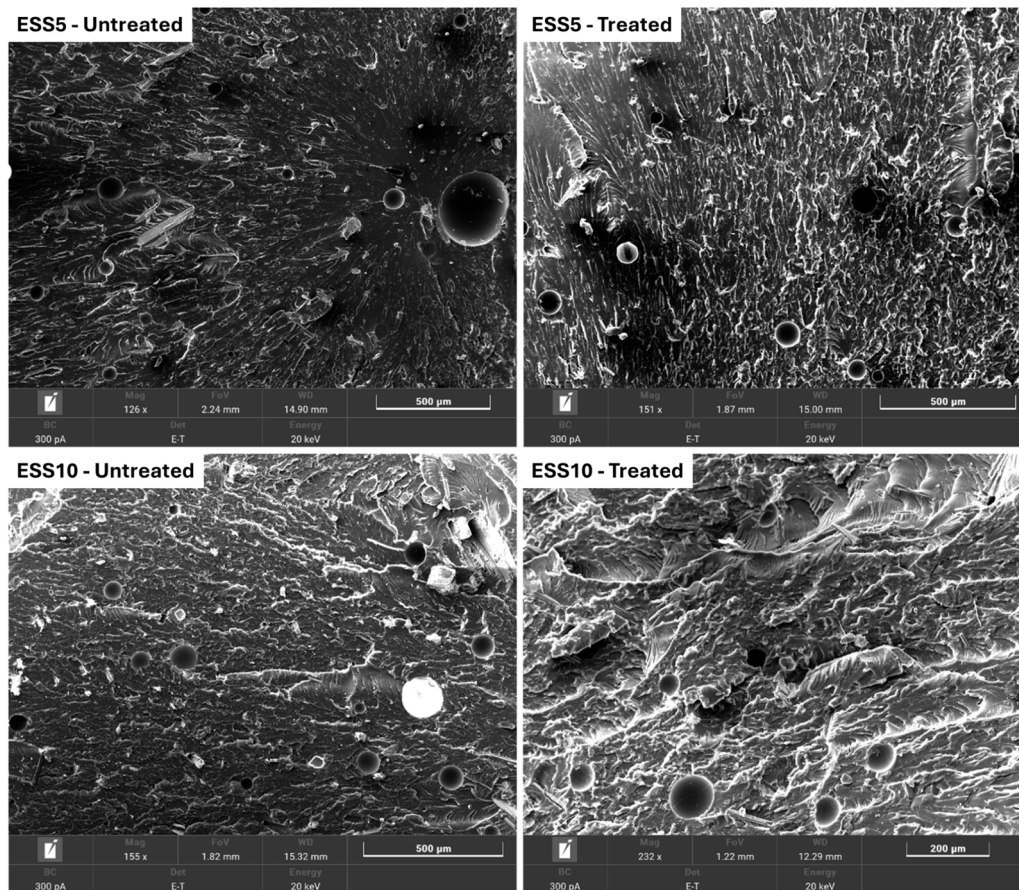


Fig. 9. SEM of Treated and Untreated ESS5 and ESS10.

shells are typically discarded as agro-waste and pose minimal ecological risk when responsibly collected and processed. Their reuse in composites promotes waste valorization and contributes to sustainable material sourcing. However, it is important to ensure that collection does not encourage overharvesting of snails or disrupt local ecosystems. In terms of health and safety, proper cleaning, drying, and sterilization, such as ethanol washing and oven-drying used in this study, are crucial to eliminate residual organic matter and reduce the risk of microbial contamination or odor development. Once processed into powder and embedded in a cured epoxy matrix, snail shell particles are fully encapsulated and pose no known health hazard during end-use. Nevertheless, occupational safety measures (e.g., dust control during grinding) should be observed during material preparation.

3.8.1. Limitations and future perspectives

The study provides important insights into the effects of bioderived calcium carbonate and thermal cycling on the morphology and mechanical behavior of reinforced epoxy biocomposites; however, it is not without limitations. The investigation is primarily based on the morphological analysis and mechanical performance of the composites. However, other critical thermal and chemical characterization techniques, such as thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), and Fourier-transform infrared spectroscopy (FTIR), were not explored. These techniques are part of our ongoing work to better understand the composite's thermal stability, glass transition behaviour, viscoelastic properties, and interfacial interactions, thereby providing a more holistic view of the structure and property relationships.

Other future studies would also focus on optimising the morphological and processing parameters of SS_p and the reinforced epoxy composites to further elucidate the correlation between structure and

properties. Complementarily, statistical tools such as analysis of variance (ANOVA) and confidence interval estimation would be integrated into the experimental designs to rigorously assess the significance of variations in key mechanical and physical properties, thus improving data reliability and reproducibility. In addition, a life cycle assessment (LCA) and cost-benefit analysis would be incorporated in our subsequent research in quantifying the overall sustainability and economic feasibility of scaling this bio-based filler system. Such analyses will not only advance scientific understanding but also determine the broader environmental and industrial impact of SS_p composite adoption in sustainable material development.

4. Conclusion

This study comprehensively evaluated the mechanical and physical properties as well as morphological features of epoxy bio-composites reinforced with snail shell powder SS_p at varying weight percentages for both treated and untreated samples. The investigation revealed that thermal cycling treatment and 10–20 wt% reinforcement content were the most suitable conditions/parameters for the development of snail shell reinforced epoxy composites for structural applications. The incorporation of SS_p into epoxy-based composites significantly improved mechanical properties, with 20 wt% treated SS_p (ESS20) yielding the highest tensile strength (4.56 MPa) that culminated in a 94% increase over the treated control, while in the untreated condition, the ESS20 was 260.3% over the untreated control sample ESS0. Flexural strength was at a peak of 25.3 MPa in untreated ESS20, while treated composites showed a maximum flexural modulus of 0.75 GPa at ESS10. Impact strength was highest in ESS10 (1.82 kJ/m²) for untreated and ESS20 (2.09 kJ/m²) for treated composites. Hardness was at maximum at ESS20 (65.6 HS) for untreated and ESS15 (65.4 HS) for treated. Water

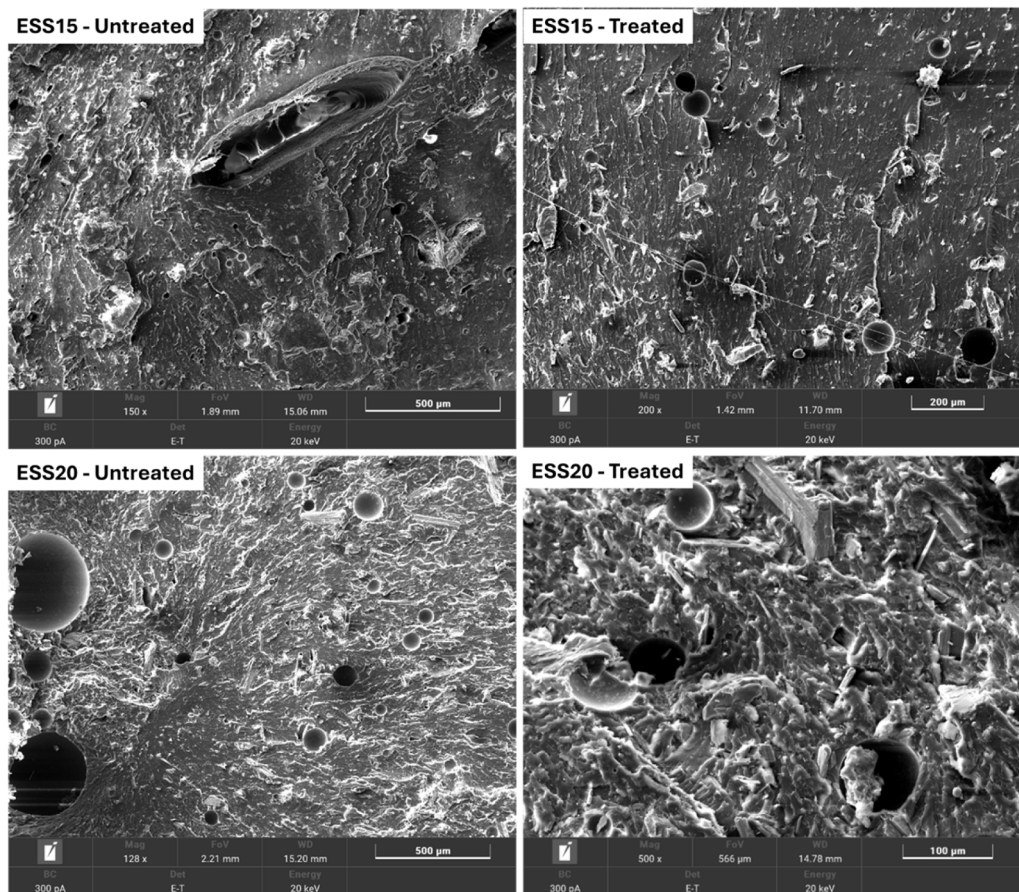


Fig. 10. SEM of Treated and Untreated ESS15 and ESS20.

absorption was limited by the thermal cycling treatment carried out on the samples, while the density was enhanced as the SSp content increased in addition to thermal treatment. SEM and XRD confirmed good dispersion and a dominant CaCO_3 phase, respectively.

CRedit authorship contribution statement

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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.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.nxmte.2026.101964](https://doi.org/10.1016/j.nxmte.2026.101964).

Data availability

The data will be made available on request.

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