



Original article

Sustainable and Eco-friendly Based Hydroxyapatite Particles-Wool Fiber Hybrid Reinforced Epoxy Bio-composites for Biomedical Applications

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ABSTRACT

The rapid growth of the world population and industrialization has led to an unparalleled rise in the generation of waste from animals in the agricultural and food processing industries among other sectors. Besides this, the concerns for the future create an urgent requirement for sustainable materials that can substitute conventional, non-renewable composites in many applications. Hence, this study investigates the development of bio-composites using animal wastes as reinforcement materials. The research offers an environmentally acceptable process that aid waste management and addition to enhancing the performance of materials for modern day applications. Shells and wools were processed to obtain hydroxyapatite particles (HAp) of 18.52 μm sizes and 10 mm long fibers, respectively that were used as reinforcements in epoxy. Stir cast molding in an open mold was used to fabricate the epoxy-based bio-composites with varying proportions of the constituents. Mechanical, wear and microstructural images evaluations revealed and support the use of these bio-reinforcement based materials. The results indicated that wear, hardness and impact strength properties were enhanced with 18 mass. % reinforced composites providing the optimum properties when compared to other weight fractions. It showed optimum values in wear (0.123), hardness (78.4 HS), impact strength (83.1 kJ/m²) and maximum flexural strength (85.2 MPa) supporting the recommendation for use as adhesive biomedical materials in biomedical applications such as orthopaedic implants, dental implants, scaffolds, bone repair, wound healing, and periodontal regeneration.

Key words: Green composites; sustainable; animal shell; hydroxyapatite; wool fiber; biomaterials.

1. INTRODUCTION

Owing to the rapid advancement of novel technologies, numerous studies have shown that composite materials are

among the most important engineering materials, successfully integrated into all areas of technological development [1–2]. Composite materials are known for their exceptional characteristics, primarily due to their

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ability to combine the most desirable properties of each constituent material [3]. The unique properties of composite materials are largely influenced by the type, class, and physical and mechanical characteristics of the reinforcements embedded within the matrix [2]. In recent years, the development of novel biopolymer composites derived from animal waste has gained increasing attention among researchers [4]. Synthetic polymeric materials have been shown to possess high resistance to biodegradation and poor biocompatibility. Consequently, there has been a significant global push to develop low-cost, biodegradable polymers derived from natural sources, especially for biomedical applications [5–6]. Natural and bio-derived polymer composite scaffolds for tissue engineering have been widely explored for bone tissue replacement [7–8]. Studies have confirmed that bio-derived polymer materials offer excellent biodegradability, high corrosion resistance, good wear and shear strength, and outstanding biocompatibility [9]. Generally, polymeric composite materials have proven to be exceptional and essential in the biomedical industry compared to other engineering materials, due to their biocompatibility, mechanical properties, microstructure, and degradation rate [10–12]. HAp and HAp-based materials has been successfully applied in the fabrication of biodegradable polymer-based composite biomaterials due to the similarity of its chemical structure to that of the natural human bone $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ with a calcium to phosphorous ratio (C/P) between 1.5 to 1.7 [13–14]. The incorporation of HAp into a polymer matrix also improves the biocompatibility, degradation rate of the composites and the chemical bonding between the implant and host bone tissues [15–17]. These biopolymer properties are majorly attributed to the percentage of HAp and enhancement of the bioactivity in them [15]. The dire need for developing unique biomedical materials at low cost cannot be overemphasized. Due to this fact, extensive research and development on the use of farm animal wastes are exponentially increasing globally and most research works have revealed the potentials that are imbedded in the eggshells and wool fiber from Nigerian farms which are readily available as waste [16]. The production of HAp from agro-wastes is evolving as an alternative source for bone grafting and reinforcements for the fabrication of biomaterials in developing countries due to its availability and low cost [17]. While several researchers keep working on the use of natural reinforcements for the development of bio-composites, the use of HAp with wool fiber in epoxy has not been given adequate attention. This research is to develop a sustainable and biodegradable hybrid composite for biomedical applications using HAp as well as wool fibers sourced from agro-wastes in Nigerian with epoxy resin as matrix. The choice of these waste materials is determined by their availability, physical characteristics, and capacity to break down naturally. More so, new research has focused on the applicability of biodegradable HAp-based polymers in bioengineering and orthopedic industry, generating a feasible long-run solution to mitigate environmental hazards generated by dumping of synthetic polymers in the environment.

2. MATERIALS AND METHODS

2.1 Materials

Egg shell (Fig. 1) wastes were sourced from the eggs of 24-week-old black noiler chicken (*Gallus domesticus*) of frizzled feather and fluffed neck structure with an average body weight of 0.9 kg while West African Zebu breed (Azaouak) wool fiber (Fig. 2) was sourced from Owo abattoir market in Akure, Nigeria. Epoxy Resin and Hardener (for epoxy resin), orthophosphoric acid, NaOH and distilled water, were sourced from Pascal Scientific, Akure.



Fig. 1 Egg shell



Fig. 2 Wool fiber

2.2 Preparation of the eggshell based HAp

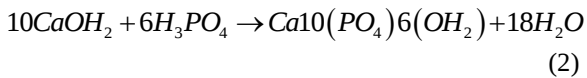
The sourced eggshells were washed and boiled to remove surface impurities, followed by a second washing with distilled water and open-air drying for 5 days. Further drying was performed in an oven (model KX350A; KENXIN International Co. Ltd., China) at 80 °C to completely remove residual moisture. Pulverization and grinding of the eggshells were then carried out to achieve a sieve size of 65 µm. The conversion of the 65 µm eggshell powder into CaO was conducted following the procedure described by [13].

In the first stage of the calcination process, a heating rate of 7 °C/min was used to heat the eggshells from room temperature to 550 °C, where they were held for 2 hours.

In the second stage, a heating rate of 5 °C/min was used to heat them to 700 °C, with a holding time of 2 hours. In the third stage, a heating rate of 4 °C/min was applied to heat the shells to 1000 °C, where they were held for 1 hour. At this stage, the eggshells were transformed into calcium oxide (CaO) through the evolution of carbon dioxide (CO₂). The CO₂ was formed according to Eq.1.



The CaO produced from the calcined eggshell was further converted into HAp using orthophosphoric acid (H₃PO₄), as shown in Eq.2. A stoichiometric amount of the calcined sample was dispersed in a beaker containing 100 mL of distilled water and placed on a hot plate maintained at 90 °C for 2 hours. A 20 mL portion of analytical-grade orthophosphoric acid was then added to the beaker and stirred at 120 rpm. Initially, a suspension was observed, after which the solution became clear after approximately 30 minutes of mixing. The pH value of the solution was maintained below 2 by adding drops of sodium hydroxide solution as needed. The solution was aged for 7 days, then filtered, and the resulting residue was dried at 105 °C for 1 hour in an oven. Subsequently, the dried residue was heated to 1000 °C in a furnace at a heating rate of 150 °C/min for 1 hour. Finally, the lumped HAp solids were manually ground into powders and sieved to obtain particle sizes of approximately 18.52 µm.



2.3 Preparation of wool fiber

The sourced wool fibers were washed several times with warm and cold water mixed with laundry detergent to remove blood and manure. The fibers were then air-dried for 5 days. To eliminate microorganisms present in the fibers, the wool was treated with 0.1 M NaOH. This treatment was performed in a shaker water bath set to a temperature of 50°C and maintained for 4 hours. After treatment, the fibers were washed with tap water and finally rinsed with distilled water to achieve a neutral pH. Finally, the wool fibers were sun-dried for up to 10 days before being cut into 10 mm lengths (Fig. 3).

2.4 Composite fabrication

The eggshell derived HAp/wool fiber hybrid reinforced epoxy-based composites were produced in an open mold. An unreinforced epoxy composite sample (control) was first produced for the basis of comparison. The composites were produced by dispersing 3, 6, 9, 12, 15 and 18 mass% of the HAp-wool fibers as the reinforcements in the ratio of 1:1 while the epoxy and hardener as the matrix were mixed in the ratio of 2:1. The materials were compounded by manual stirring and pour the homogenous mix into an open mold where they were allowed to cure in their

respective test samples molds. Three samples were fabricated for each test and the average values after evaluations were used as representative values.



Fig. 3 Prepared wool fiber

2.5 Characterization of the fabricated composites

2.5.1 Wear test

The wear resistance of the samples was evaluated using a Taber abrasion tester (TABER Rotary Platform Abrasion Tester – Model 5135, USA) in accordance with the ASTM D4060-10 standard. A center hole of 10 mm was made in each sample to fix the test piece onto the machine. The sample was secured to the instrument platform, which is motor-driven at 500 rpm. Each specimen was a flat, round disc approximately 100 mm in diameter and with a standard thickness of about 6.35 mm. The ASTM D4060-10 standard was used to calculate the wear resistance, expressed as the loss in weight after a specified number of abrasion cycles (500). A set of two rotating abrasive wheels, each with a thickness of 12.6 mm and a diameter of 50 mm, was used against the test samples under a contact load of 1000 g. The rotational speed was maintained at 500 rpm for approximately 1000 cycles. The wear indices were calculated based on the sample weight loss, as shown in Eq. 3.

$$\text{Wear Index} = (W_i - W_f) \cdot 100 / C \quad (3)$$

where, W_i , W_f and C are the initial weight, final weight, and number of test cycles, respectively.

2.5.2 Flexural test

Flexural test on the samples were examined on a universal testing machine (UTM FS 300–1023, USA) according to ASTM D-790-03 standard. At least three samples from each lot were tested in accordance with the standard. The test specimen had a dimension of 150 × 50 × 3 mm and was hooked on the grip of the machine. The test speed was 5 mm/min over a span of 65 mm and the obtained results were analyzed afterward.

2.5.3 Impact test

The notched Izod impact test was conducted in accordance with the ASTM D256–10 (2018) standardized test method for determining the Izod Pendulum impact resistance of plastics. The test was performed using a Hounsfield balanced impact testing machine, serial number 3915, model number H10-3. Impact test samples, with dimensions of $64 \times 11 \times 3$ mm, were notched (V-notch type) at the center with a depth of 2.54 mm, an angle of 45° , and a root radius of 0.25 mm. The samples were placed horizontally on the machine, maintaining a 60 mm distance between the support lines. The test samples were positioned in a cantilever arrangement, clamped upright, with the V-notch level with the top of the clamp. The machine's pendulum struck the test piece and was allowed to fall freely from a fixed height of 610 mm.

2.5.4 Hardness test

A digital Shore D Hardness Tester was used to perform the hardness test. The hardness of each material was measured and determined by the hardness tester, expressed as HS. For each hardness measurement, six indentations were made at different points on each specimen. The average value was calculated to provide the mean hardness for each selected material. A load of 15 kg was applied to each specimen with a dwell time of 15 seconds. This test was conducted in accordance with ASTM D2240-00 and was also carried out in previous research by Oladele et al. [18].

2.5.5 Microscopy characterization

The Scanning Electron Microscope (SEM) characterization of the fracture surfaces of the samples was performed using the EVO MA 15 (Zeiss, Germany), a variable-pressure SEM with a tungsten filament electron source, an accelerating voltage range of 0.2–30 kV, and a resolution of up to 3 nm. The samples were gold-coated via sputtering to improve electrical conductivity.

3. RESULTS AND DISCUSSION

3.1 Wear properties

The effects of eggshell-based HAp/wool fiber reinforcements on the wear properties of epoxy composites are shown in Figure 3. The results presented in Figure 4 indicate that wear property improvements occurred as the weight fraction of reinforcements increased. This enhancement is attributed to the reduced frictional force resulting from a lower coefficient of friction, which is consistent with the findings of [19]. It was also observed that a higher density of HAp/wool fiber in the epoxy matrix led to a reduction in frictional force, thereby diminishing the contact layer between the abrasion disc and the epoxy resin, as reported by [19]. Additionally, the strong covalent bond between the HAp/wool fiber and the epoxy resin contributed to the improvements in wear properties. The optimum wear resistance value was achieved in the sample

with 18 mass % reinforcements, which exhibited a value of 0.123 mg, while the control sample showed the least wear resistance due to the higher frictional force on the unreinforced epoxy matrix. The findings suggest that the hybrid composite with the minimal wear rate demonstrates the lowest specific wear rate and coefficient of friction, as well as a favorable wear mechanism, thus possessing the optimal wear resistance.

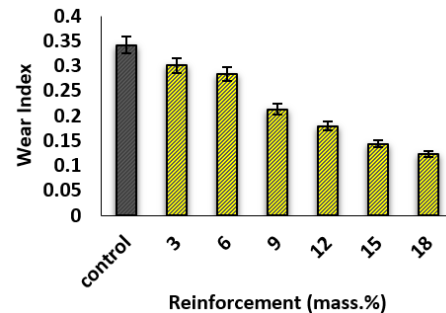


Fig. 4 Variation of wear index properties.

3.2 Hardness properties

The variation in hardness properties is shown in Fig. 5, where enhancements in hardness were observed as the reinforcement weight fraction increased. Strong adhesion occurred as the weight fraction of HAp/wool fiber increased, due to improved wettability between the reinforcements (HAp/wool fiber) and the epoxy matrix, leading to these improvements. These results are consistent with the findings of previous researchers, Mangisetty et al. [20] and Oladele et al. [21].

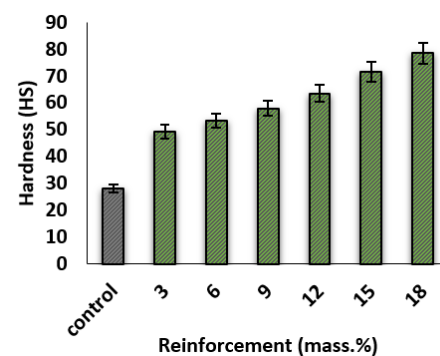


Fig. 5 Variation of hardness properties

In direct correlation with the wear results in Fig. 4, the sample with 18 mass % reinforcement exhibited the optimum hardness value of 78.4 HS, representing a 180% increase compared to the control sample (28 HS). These findings corroborate the observed performance in Figure 3 and confirm previous results showing that higher hardness typically leads to greater wear resistance [21]. The enhanced hardness is attributed to the inherent hardness of eggshell-based HAp, the resilient fibrous structure of the wool fiber, and the synergistic effect of both reinforcements, which together enhance the composite's surface resistance and stiffness.

Overall, these findings indicate that the developed bio-composites are highly suitable for applications requiring high hardness and wear resistance. They are promising sustainable and eco-friendly green composite materials for biomedical applications such as bone repair, wound healing, and periodontal regeneration.

3.3 Impact strength

Fig. 6 shows the variation in impact strength with the addition of HAp/wool fiber hybrid reinforcements. Similar to Fig. 4, it was observed that the impact strength increases as the reinforcement content increases, contrary to what is typically observed in monolithic materials. These findings highlight the benefits achievable in hybrid composites, where each reinforcement showcases its inherent advantages within the developed composites.

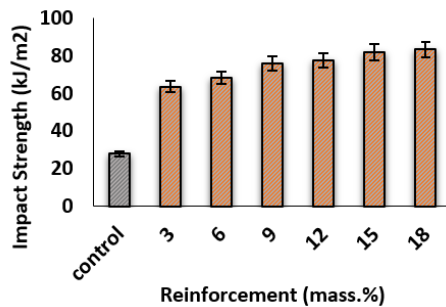


Fig. 6 Variation of impact strength

The high impact strength provided by the wool fiber, along with proper wetting between the HAp and wool fibers, contributed to these improvements. These observations are also in agreement with the findings reported in [20] and [21]. Furthermore, efficient dispersion of the HAp/wool fibers within the matrix may have contributed to the enhanced properties of the reinforced composites, as supported by [13]. An optimum impact strength value of 83.1 kJ/m² was observed in the sample with 18 wt.% reinforcement, indicating a 192% enhancement compared to the control sample, which exhibited a value of 28 kJ/m². The enhanced impact strength was attributed to the crack-deflecting capability of HAp and the energy absorption mechanisms of the wool fibers. The synergistic effects of both reinforcements result in enhanced toughness and greater potential for impact energy absorption, rendering these bio-composites suitable for applications such as bone repair, wound healing, and periodontal regeneration, where exceptional impact strength and durability are required.

3.4 Flexural properties

Fig. 7 shows the effects of HAp/wool fiber hybrid reinforcements on the maximum flexural strength, where a steady increase was observed. The results revealed that an enhancement in flexural strength occurred as the reinforcement content increased from 9 mass% to 18 mass%. This observation highlights the importance of studying the effect of weight fractions on the properties of

reinforced composites. The low flexural strengths observed in the 3 mass% and 6 mass% reinforced composites could be attributed to the insufficient amount of reinforcement, which was not adequate to achieve significant improvement. The optimum value was obtained with 18 wt.% reinforcement, achieving the highest flexural strength of 85.2 MPa, representing a 105% increase compared to the control (unreinforced) sample, which had a flexural strength of 41.6 MPa. This improvement can be attributed to the presence of wool fibers, which help to distribute stress more uniformly throughout the composite, thereby preventing localized failure and enhancing flexural strength. Uniform stress distribution inhibits the initiation and propagation of cracks, further improving the composite's flexural strength [22], [23].

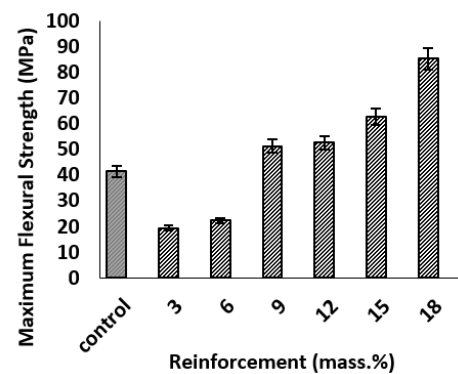


Fig. 7 Influence of HAp/wool fiber on maximum flexural strength

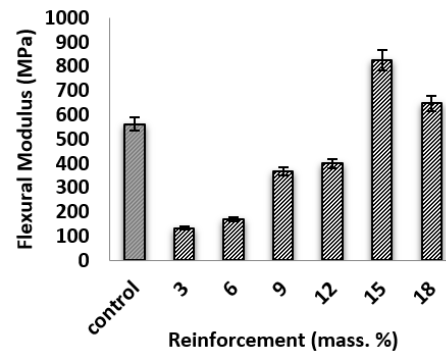


Fig. 8 Variation of flexural modulus properties

Fig. 8 shows the variation in flexural modulus properties, revealing trends similar to those observed in Fig. 7, with some exceptions. While the flexural modulus generally increases with reinforcement content, enhancement was observed only between 15 and 18 mass %, with a slight reduction at 18 mass %. These observations contrast with the results shown in Fig. 7. The sample with 15 mass % exhibited the optimum flexural modulus, with a value of 823.7 MPa, indicating a 46% improvement compared to the control sample (561.1 MPa). The reasons for these trends are similar to those discussed for Fig. 7. However, beyond the 15 mass % threshold, the addition of more reinforcement may lead to agglomeration, where the particles cluster rather than distribute uniformly. This agglomeration creates stress concentration points, which

weaken the composite's flexural modulus, as observed in the sample with 18 mass %.

3.5 SEM images

The utilization of a scanning electron microscope (SEM) enabled the examination of the fractured surface morphology of the developed bio-composites. Fig. 9 (a–f) presents SEM images of the unreinforced sample (a) and composites reinforced with 3, 6, 9, 12, and 18 mass % of hybrid fillers (b–f). These samples were selected as representatives of low, medium, and high weight fractions, with 18 wt% corresponding to the sample showing optimal properties among the high-weight fraction group.

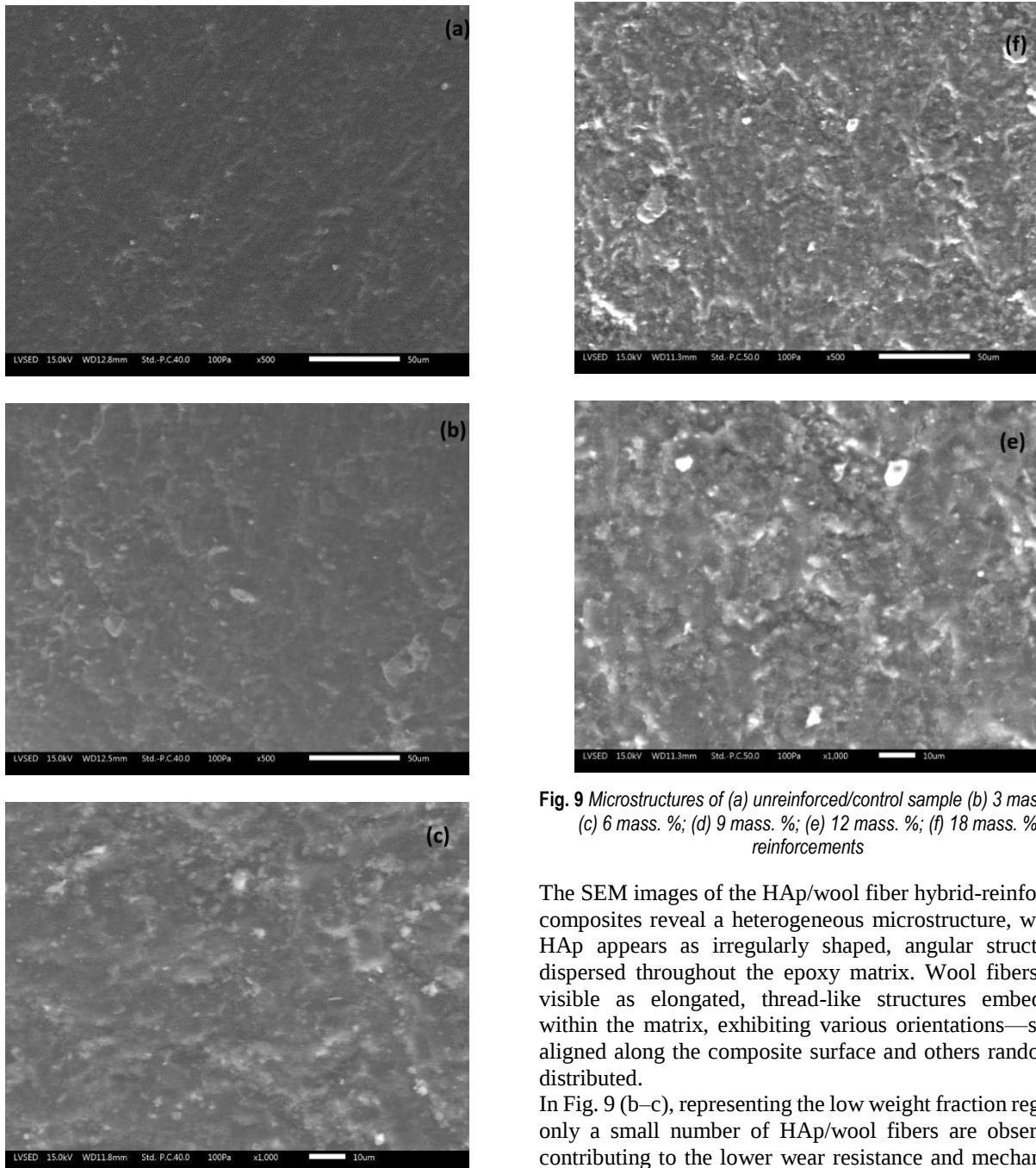


Fig. 9 Microstructures of (a) unreinforced/control sample (b) 3 mass. %; (c) 6 mass. %; (d) 9 mass. %; (e) 12 mass. %; (f) 18 mass. % reinforcements

The SEM images of the HAp/wool fiber hybrid-reinforced composites reveal a heterogeneous microstructure, where HAp appears as irregularly shaped, angular structures dispersed throughout the epoxy matrix. Wool fibers are visible as elongated, thread-like structures embedded within the matrix, exhibiting various orientations—some aligned along the composite surface and others randomly distributed.

In Fig. 9 (b–c), representing the low weight fraction region, only a small number of HAp/wool fibers are observed, contributing to the lower wear resistance and mechanical properties compared to the other composite samples and

the control. Figs. 9 (d–e), corresponding to the medium weight fraction region, show a more uniform dispersion of reinforcements within the epoxy matrix, resulting in improved mechanical and wear properties. The high weight fraction sample is shown in Fig. 9 (f), where a higher volume of reinforcements is observed with a more uniform dispersion.

Overall, the SEM analysis revealed a well-dispersed HAp/wool fiber content in the epoxy matrix, indicating good wetting and adhesion, which contributed to the enhancement of mechanical and wear properties as the reinforcement content increased. To maximize the benefits of hybrid reinforcement, careful control of the reinforcement content and dispersion techniques is essential. Optimizing processing methods to minimize agglomeration and defects can further enhance composite performance.

4. CONCLUSION

The growing demand for sustainable and eco-friendly composite materials in biomedical applications has driven research toward the use of renewable and bio-based reinforcements. This study successfully developed and characterized hydroxyapatite (HAp) derived from eggshells and wool fibers from the West African Zebu breed (Azaouak) as hybrid reinforcements in an epoxy matrix for potential biomedical use. The mechanical and tribological performance of the composites was systematically evaluated through wear, hardness, impact strength, flexural strength, and flexural modulus tests. The results demonstrated that incorporating 18 wt.% of the hybrid reinforcement yielded optimal performance, showing improved mechanical properties and wear resistance. The wear test (0.123 mg) confirmed enhanced surface durability, while the hardness measurement (78.4 HS) indicated increased material toughness due to the presence of hydroxyapatite. Impact strength (83.1 kJ/m²) and flexural strength (85.2 MPa) results revealed that the composites maintained sufficient structural integrity, making them suitable for biomedical applications where mechanical reliability is essential. However, the study also found that surface treatment of wool fibers is recommended prior to incorporation into the epoxy matrix to enhance biocompatibility, especially for implant-related applications.

The scientific contribution of this work lies in demonstrating the feasibility of using waste-derived hydroxyapatite and natural wool fibers as sustainable reinforcements in epoxy-based biomedical composites. This approach offers an eco-friendly alternative to conventional synthetic reinforcements, aligning with global efforts to promote sustainability in biomedical material development.

To further validate the suitability of these composites for biomedical use, future research will focus on biocompatibility testing, in vitro cytotoxicity assessments, and long-term degradation studies in simulated body environments. These investigations will provide deeper

insight into the potential applications of these materials in bone scaffolds, dental implants, and orthopedic devices.

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