

Synthesis and Mechanical Properties of HAp/SiO₂/PLA Composite Derived from Goat Jawbone

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ABSTRACT

Biomaterials like nano-hydroxyapatite (nHAp) closely mimic the mineral makeup of real bones. This research is centered around the synthesis of nHAp (Ca₁₀(PO₄)₆(OH)₂) using goat jawbone. After being ball-milled for 4 hr at 150 rpm, the bone underwent a 10 °C/min controlled rate heat treatment to 300 °C, 600 °C, 900 °C, 1000 °C, and 1200 °C. With a lattice strain ranging from $\epsilon = 91.49$ to 12.16, the nHAp crystallite size varied between 3.61 nm and 21.87 nm when measured using the Scherrer method and 1.41 nm and 14.16 nm when measured using the Williamson-Hall method while a porosity range of 81.5% to 84.5% of the nHAp derived from goat jawbone was observed. The composite's mechanical properties were enhanced by adding bio-inert ceramics SiO₂, which helped overcome the nHAp's poor fracture toughness and lack of flexibility. Composites of HAp, SiO₂, and PLA with different weight percentages were created using the co-precipitation technique. In order to characterize the material, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were used to analyze its structure and morphology. The made composites had a compressive strength of 43.26 MPa and porosity ranging from 78.65% to 85.25%.

Keywords-goat jawbone; nano hydroxyapatite; PLA; SiO₂; biocomposite

I. INTRODUCTION

Addressing issues such as bone defects resulting from trauma, osteoporosis, and degenerative diseases requires the development of effective biomaterials that can promote bone regeneration and provide structural support in orthopedic applications [1]. Among the various materials explored, hydroxyapatite (HAp), a bioceramic composed of calcium phosphate, has emerged as a leading material due to its close resemblance to the natural bone. This material is frequently utilized in bone tissue engineering [2]. Although pure HAp exhibits excellent biocompatibility, its mechanical properties, such as strength and fracture toughness, are insufficient for load-bearing applications [3]. To address these challenges, researchers have developed composite materials that combine HAp with ceramics and polymers, enhancing its mechanical properties while retaining its biocompatibility. [4]. By combining HAp with bio-inert ceramics, such as silicon dioxide (SiO_2), and biodegradable polymers, such as polylactic acid (PLA), researchers have created materials with improved mechanical strength, wear resistance, and processability. These composites are particularly well-suited for orthopedic implants and bone scaffolds [5]. These materials enhance bioactivity and structural integrity. Alkaline hydrolysis is a widely used method for synthesizing HAp from natural sources, such as animal bones. This method involves treating the raw material with an alkaline solution, which promotes the formation of intrinsic HAp. Alkaline hydrolysis is preferred for its ability to produce HAp with high bioactivity, making it suitable for bone tissue engineering applications. Calcination is another common method for synthesizing HAp, particularly from natural sources. This process involves heating the raw material to high temperatures, which eliminates organic contaminants and produces a highly crystalline form of HAp. While calcination enhances the material's mechanical strength, it also reduces its bioactivity due to the increased crystallinity [6]. Co-precipitation is a versatile method for synthesizing HAp-based composites, particularly those containing multiple components, such as HAp, SiO_2 , and chitosan. This method involves the simultaneous precipitation of the composite's components from a solution, resulting in a homogeneous material with enhanced properties. Co-precipitation allows for precise control over the composite's composition and structure, making it ideal for producing materials with tailored properties.

The synthesis of HAp from natural sources, such as waste animal bones, has gained attention due to its cost-effectiveness and sustainability. Various animal bones, including those from cows and goats, have been studied for their potential to produce high-quality HAp. The synthesis process typically involves heat treatment and mechanical milling to extract nano-hydroxyapatite (nHAp), which is then used to fabricate composite materials. Goat jawbone has been explored as a source of HAp for the synthesis of composite materials. Using goat jawbone as a source of HAp offers several advantages over bovine bone and synthetic HAp, particularly in terms of ethics, availability, biocompatibility, and structural properties. The extraction process involves ball milling and heat treatment to produce nHAp, which is then combined with SiO_2 and PLA to create composites with enhanced mechanical and tribological properties. These composites have shown promise

for bone replacement applications, offering a balance of biocompatibility and mechanical performance.

In addition to bulk materials, calcium phosphate (CaPO_4) coatings have been developed to enhance the biocompatibility and bioactivity of various substrates. These coatings are typically applied to metallic implants to improve their integration with surrounding bone tissue. Various deposition methods, such as plasma spraying, electrochemical deposition, and sol-gel coating, have been explored for creating CaPO_4 coatings. Each method offers unique advantages in terms of coating thickness, Ca/P ratio, phase composition, porosity, surface topography, and roughness. However, further research is needed to optimize these parameters and achieve coatings with superior performance [7].

The mechanical properties of HAp-based composites are critical for their success in orthopedic applications. Dynamic Mechanical Analysis (DMA) has been used to evaluate the performance of these materials, particularly in terms of their strength and elasticity. Studies have shown that the addition of SiO_2 nanoparticles to PLA nanocomposites can significantly enhance their mechanical properties. For example, a 10:2 ratio of SiO_2 nanoparticles to PLA has been found to improve material strength, indicating the potential for superior performance in composite materials [8]. Snail shells have also been investigated as a source of HAp for composite materials. The synthesis process involves heat treatment and wet ball milling to extract nHAp, which is then combined with SiO_2 and chitosan to produce hybrid composites. These composites exhibit improved compressive strength and modulus, making them suitable for bone tissue engineering applications.

Grey Relational Analysis (GRA) is a statistical method used to optimize the composition and processing parameters of composite materials. This approach has been applied to HAp-based composites to determine the optimal ratio of components for achieving the desired mechanical properties. By using GRA, researchers can identify the best combination of materials and processing conditions to produce composites with enhanced performance. In [8], SiO_2 hybrid composite was synthesized using the co-precipitation method, with different weight ratios. The porous structure of the nHAp:GEL: SiO_2 was decided using the technique of liquid substitution. The 40% silica added to the nHAp: GEL results in a higher compressive strength and a modulus. The synthesis and characterization of HAp/ SiO_2 /PLA composites from goat jawbone were investigated using ball milling and heat treatment to extract nHAp. These composites were analyzed for morphological, mechanical, and tribological properties, aiming to enhance mechanical performance for potential bone replacement applications. This study introduces goat jawbone-derived HAp as an ethical, abundant alternative to bovine/synthetic sources, offering superior biocompatibility. Optimizing SiO_2 in HAp/ SiO_2 /PLA composites enhances compressive strength (44.26 MPa) and modulus (0.50 GPa), overcoming brittleness for load-bearing use. Improved tribological properties further suit orthopedic implants requiring wear resistance and low friction.

II. MATERIALS AND METHODS

The research materials such as HAp, PLA, and silica reinforced composites and all the analytical chemical reagents were bought. The chemical process was carried out in deionized water.

A. HAp Preparation

Goat jawbones, a byproduct of the meat industry, are reused in this study, promoting waste management. Their use does not require ethical approval. Figure 1 shows the goat jawbones in raw, fragmented and powdered form. The goat jawbones were cleaned using forced water then immersed in a saltwater solution for 24 hr to detach any remaining impurities. Then the bones were dried under direct sunlight for 48 hr. Finally, they were broken into smaller pieces and were further dried for 24 hr. Finally, they were pulverized in a ball mill for 1 hr.



Fig. 1. Raw, fragmented and powdered forms of the utilized goat jawbone.

Powdered goat jawbone underwent heat treatment in a muffle furnace at varying temperatures (300 °C, 600 °C, 900 °C, 1000 °C, and 1200 °C) to eliminate unwanted materials and proteins. Subsequently, the powder was dried at 100 °C for 24 hr in a convection oven. Experimental X-Ray Diffraction (XRD) was conducted according to the JCPDS standards, particularly card 09-0432 for nHAp, to identify the phases present. Microstructural analysis of the goat jawbone at different temperatures was conducted using Scanning Electron Microscopy (SEM).

B. Composite Slurry Preparation

nHAp derived from goat jawbone calcined at 1200 °C was utilized to fabricate nHAp/SiO₂/PLA composites. The hybrid composites were prepared via a co-precipitation method [10], adapted from previous work on HAp/SiO₂/PLA synthesis [14]. The slurry preparation for the nHAp/SiO₂/PLA composites involved controlled titration of the reactants into solution using peristaltic pumps, a water bath for temperature maintenance, and a pH controller. A Teflon-coated stainless-steel beaker served as the reaction vessel to prevent corrosion and contamination. The reaction vessel was gradually charged with a uniformly dispersed solution of nHAp and PLA in ethanol. Subsequently, a controlled amount of SiO₂ was slowly added to the reaction vessel using peristaltic pumps. The reaction solution was maintained at 38 °C with a pH of 8.0. The concentration of PLA was kept constant, while the ratio of nHAp to SiO₂ was varied as follows: 100:0:0, 80:8:12, 80:10:10, 80:12:8, and 80:15:5. The resulting nHAp/SiO₂/PLA slurries were left in room temperature for several hours. The composite slurry was then washed five times for 1 hr each via glass filtration to remove any unreacted precursors or byproducts. Finally, the composite powders were dried at room temperature.

C. Composite Pellet Preparation

To prepare HAp/SiO₂/PLA composite specimens, a homogeneous mixture of the materials, proportioned according to a specific weight ratio, was axially compacted in a pellet press. A load of 2 tones was applied using hydraulic compression, with polyethylene glycol (PEG) acting as a binder. The compacted pellets were then dried in a controlled-environment dryer. For compression testing, five cylindrical specimens were prepared with a diameter of 10 mm and a length of 20 mm (twice the diameter), adhering to ASTM D 5024-95 guidelines. Figure 2 illustrates the various HAp/SiO₂/PLA compositions used in these compression tests. Similarly, for wear testing, five cylindrical specimens were fabricated, each measuring 10 mm in diameter and 30 mm in length.



Fig. 2. Compression test specimens.

D. Porous Structure Measurement

The pore structure of the composite materials was measured using the standard liquid substitution methodology. Ethanol was used in this method because it easily enters the scaffolds. SEM was used to examine the morphology and quantify pore size [9]. The porosity of composites was determined by the liquid substitution method, using (1):

$$\text{Porosity} = \frac{(W_f - W_i)}{\rho_{\text{ethanol}}} \left(\frac{1}{V_2 - V_1} \right) \quad (1)$$

where W_i and W_f represent the scaffold initial and final weight (after soaking), V_1 is the volume of the ethanol, V_2 is the total volume, and $\rho_{\text{ethanol}} = 0.789 \text{ g/cm}^3$.

E. Mechanical Properties of the Composites

The compressive strength and modulus were measured using an testing machine following standard ASTM D 5024-95. The samples were subjected to compression at a crosshead speed of 0.5 mm/min until failure [10]. The ultimate compressive strength and modulus were graphically determined.

F. Wear

Wear test was performed to assess the tribological behavior of the composites using a pin-on-disc tribometer. The test was performed under a 10 N load, with a sliding distance of 60 mm at a speed of 150 rpm for 1200 s. Composites wear property was measured by calculating weight loss and wear rate.

III. RESULTS AND DISCUSSION

A. Characterization of Goat Jawbone

The heating of goat jawbone to different temperatures effectively removed protein and impurities. Figure 3 illustrates the goat jawbone powder treated at 300 °C, 600 °C, 900 °C, 1000 °C, and 1200 °C. The heat treatment temperatures were chosen based on HAp's thermal decomposition. Water has been evaporated at 300 °C, organic matter combusts at 600 °C, and crystallization occurs at 900–1000 °C. At 1200 °C, full recrystallization stabilizes HAp. Intermediate temperatures were excluded from the analysis as they lack significant phase transitions, ensuring optimal porosity and mechanical integrity for bone tissue engineering.

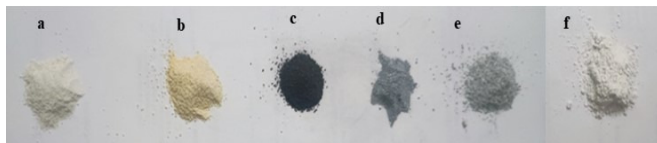


Fig. 3. Goat jawbone powder: (a) Initial stage, (b) 300 °C, (c) 600 °C, (d) 900 °C, (e) 1000 °C, (f) 1200 °C.

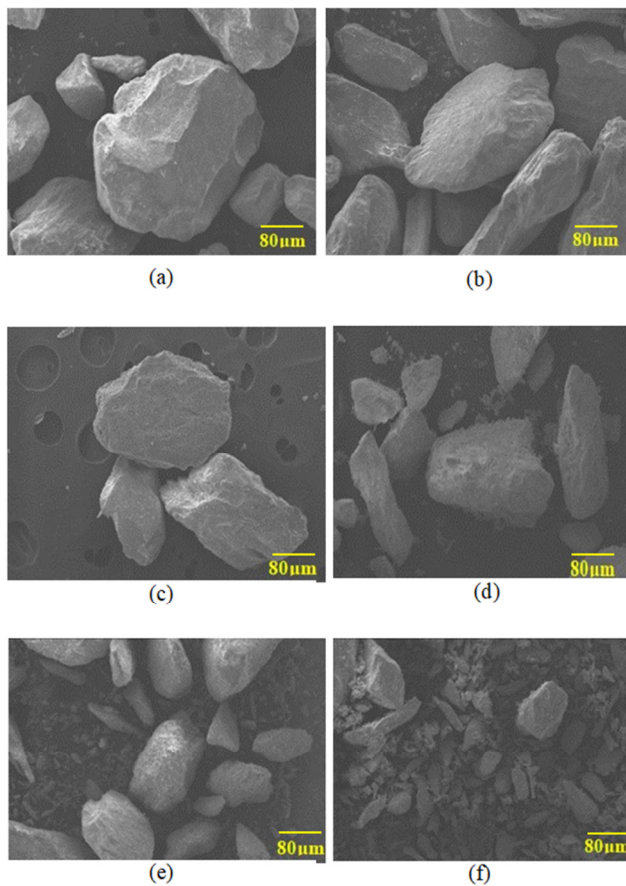


Fig. 4. SEM images of goat jawbone powder: (a) Raw goat jawbone, (b) at 300 °C, (c) at 600 °C, (d) at 900 °C, (e) at 1000 °C, (f) at 1200 °C.

The thermal changes in the bone's organic and inorganic components primarily influence its color transformation. At

300 °C, water evaporates, collagen begins to degrade, and the color shifts from white to light brown. At 600 °C, the combustion of organic compounds leads to carbonization, darkening the powder. At 900 °C and 1000 °C, the hydroxyapatite phase begins to develop, giving the material a bluish-grey hue. At 1200 °C, the powder turns white due to the complete removal of organic matter and the occurrence of recrystallization. Figure 4 illustrates the SEM images of goat jawbone powders at different temperatures.

Figure 5 presents the XRD generated graph of the HAp composites at different temperatures and the comparison with the JCPDS 09-0432 standard. It can be noted that intensity varies with increasing temperature, with the maximum intensity recorded at 1200 °C. The major diffraction peaks, corresponding to 2θ values of 22.82, 25.62, 28.98, 32.64, 32.82, 33.32, 35.35, 39.5, 46.5, 46.56, 47.98, and 50.16, align with the JCPDS 09-0432 reference for hydroxyapatite. Table I summarizes the porosity of goat jawbone scaffolds at different temperatures, showing an increasing trend in porosity from 300 °C to 1200 °C. From the XRD characterization results, the presence of HAp in goat jawbone treated at 1200 °C is confirmed. Figure 6 shows the SEM pattern of goat jawbone powder heated at 1200 °C. The image indicates that the powder is interrelated by macro level and micro level pores within it. These pore surfaces present in the sample promoted significant increase in the surface area of adsorption for protein molecules. The porosity of the scaffolds was measured as 84.51 % with the liquid substitution method.

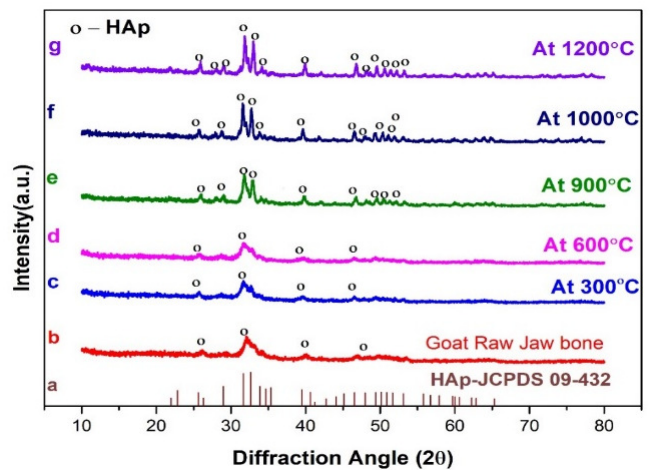


Fig. 5. XRD graph of goat jawbone. (a) JCPDS 09-432, (b) goat raw jawbone, (c) 300 °C, (d) 600 °C, (e) 900 °C, (f) 1000 °C, (g) 1200 °C.

TABLE I. POROSITY OF GOAT JAWBONE USING LIQUID SUBSTITUTION METHOD AT DIFFERENT TEMPERATURES

Temperature (°C)	Porosity (%)
300	76.25
600	78.65
900	80.25
1000	82.60
1200	84.51

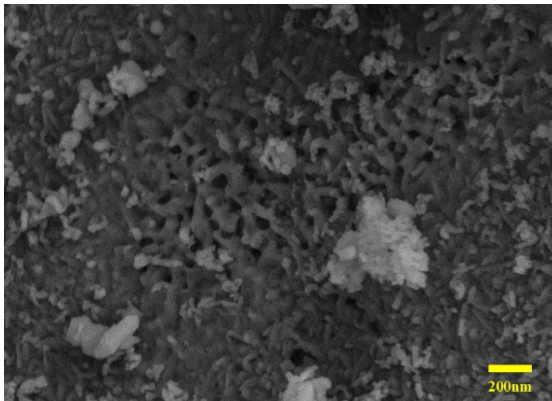


Fig. 6. SEM pattern of goat jawbone powder at 1200 °C.

B. Crystalline Size and Strain of Goat Jawbone Powder

XRD can be employed to evaluate peak broadening in terms by determining the crystalline size and strain. The Scherrer equation was employed to determine the particle size of the HAp. The Scherrer formula and the modified WH

method were applied to measure the crystalline size and strain in ZnO-NPs composites, respectively [11, 12]:

$$D = \frac{k\lambda}{\beta \cos \theta} \tag{2}$$

where D represents the particle size in nm, λ is 1.54056 Å, k is a constant equal to 0.9, β is the full width at half maximum, and θ is the peak position. The Williamson-Hall (W-H) method [13] was employed to ascertain the crystal imperfection and distortion of the powder as a result of strain. The particle's stain formula is:

$$\varepsilon = \frac{\beta}{4 \tan \theta} \tag{3}$$

In (3), it is assumed that the contributions of strain and particle size to line broadening are independent. From (2) and (3) we get:

$$\beta = \frac{K\lambda}{D \cos \theta} + 4\varepsilon \tan \theta \tag{4}$$

$$\beta \cos \theta = \frac{K\lambda}{D} + 4\varepsilon \sin \theta \tag{5}$$

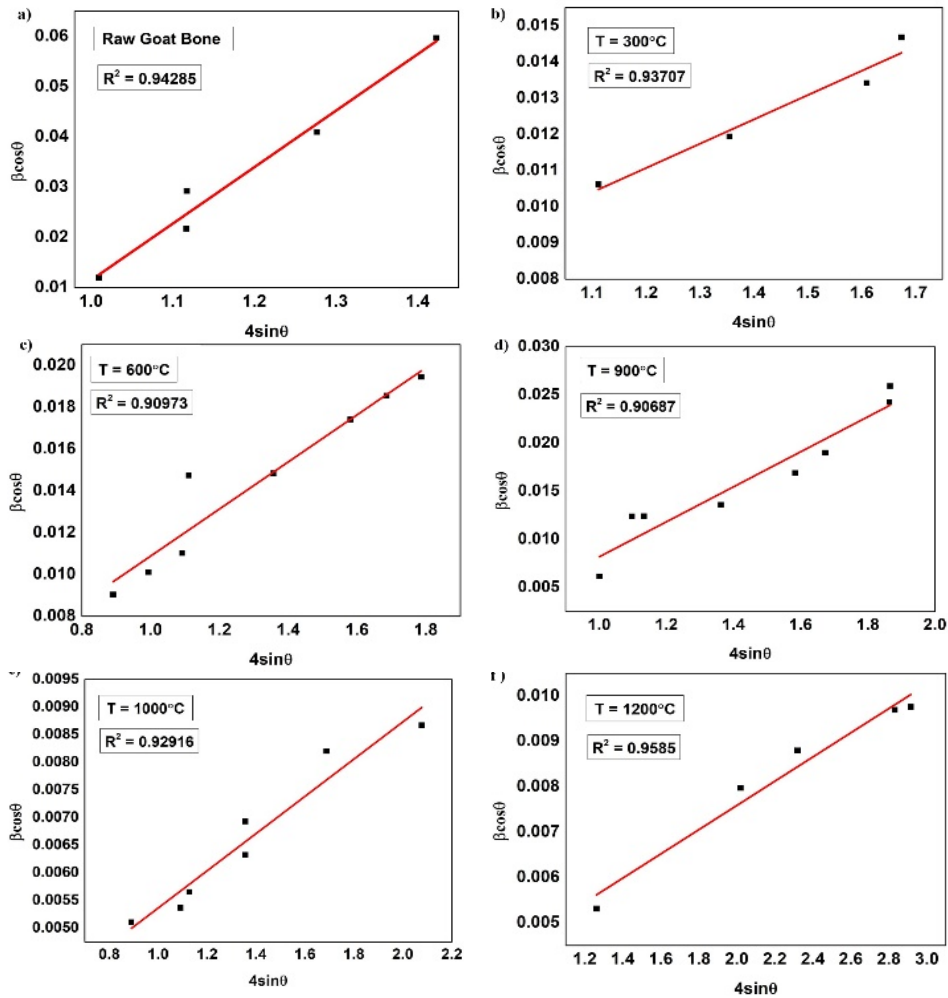


Fig. 7. W-H plots of $4\sin\theta$ vs $\beta\cos\theta$ of goat jawbone powder heated at different temperatures.

The UDM is represented by (5) and the hall plot shows the nHAp composite at various temperatures with the x-axis as $4\sin\theta$ and the y-axis as $\beta\cos\theta$. The y-intercept and slope or strain ϵ were used to determine the crystalline size of HAp in Figure 7. In both the Scherrer and W-H methods, the particle size was calculated using the major peak phase, which had 2θ values of 22.82, 25.62, 28.98, 32.64, 33.32, 35.35, 46.5, 47.98 and 50.16. The crystallite size and strain lattice of goat jawbone at varying temperatures are illustrated in Table II. Table III shows that using the Scherrer equation (1), the particle size of goat jawbone increased from 3.61 nm to 21.87 nm at 1200 °C. Using the W-H equation (4), it was observed that the strain between the particles decreased as the calcination temperature of the goat jawbone increased. The goat jawbone powder, when subjected to 1200 °C, exhibited a crystallite size of $D = 21.87$ nm according to the Scherrer method, while the W-H method indicated a crystallite size of $D = 14.16$ nm and a strain of $\epsilon = 12.16 \times 10^{-3}$.

TABLE II. LATTICE STRAIN AND CRYSTAL SIZE OF GOAT JAWBONE AT VARIOUS TEMPERATURES

Sample	Scherer method	W-H method	
		UDM	
	Crystallite size D (nm)	Crystallite size D (nm)	$\epsilon \times 10^{-3}$
Goat raw jawbone	3.61	1.41	91.49
300 °C	4.14	4.80	67.1
600 °C	5.10	5.60	19.28
900 °C	10.75	6.83	18.18
1000 °C	15.91	12.76	13.37
1200 °C	21.87	14.16	12.16

C. Hybrid Composite Mechanical Properties

Compression testing was conducted on the HAp/SiO₂/PLA composite scaffolds. Composite fractures, shatters, or cracks visibly when subjected to compressive force are compressive strength failure criteria. Table III presents the compressive strength, compressive modulus, and porosity of the composites, while the stress-strain curves for each composition are illustrated in Figure 8. The HAp composites derived from goat jawbone treated at 1200 °C exhibit a compressive strength of 28.58 MPa, as shown in Table III. The addition of silica enhances both the compressive strength and modulus of the composite. When SiO₂ is introduced as a reinforcing element in nHAp/PLA, the compressive modulus increases from 0.30 GPa to 0.50 GPa. A study on co-precipitation synthesized nHAp:GEL: SiO₂ composite scaffolds addressed the issue of low intrinsic fracture toughness. The compressive strength of these scaffolds was attributed to the organic-inorganic interface between the gelatin (GEL) matrix, nHAp, and SiO₂. The GEL component played a critical role in the scaffold's performance and was a limiting factor in the mechanical properties of the composites. At 72.5% porosity, SiO₂-doped nHAp:GEL composites exhibited a maximum compressive strength of 8.01 MPa and a modulus of 0.73 GPa. These mechanical properties varied with changes in silica content [14].

To ensure the reliability of the results, five cylindrical samples ($n = 5$) per composition were tested, and the mean values were recorded. The compression test results of the HAp/SiO₂/PLA composite revealed a compressive strength of

44.26 MPa, comparable to natural bone's. The composite also exhibited a porosity of 79.82%, which plays a crucial role in its overall structural characteristics. The incorporation of SiO₂ into the nHAp/PLA composite at varying weight percentages (5%, 8%, 10%, 12%, and 15%) led to a gradual increase in compressive strength. Among these compositions, the 80:15:5 ratio achieved the highest compressive strength of 44.26 MPa, highlighting the significant role of silica in reinforcing the composite and enhancing its mechanical properties. Silica plays a crucial role in improving the composite's strength and structural integrity. Its presence facilitates the transformation of nHAp, which is inherently brittle, into a more deformable material when integrated into the SiO₂/PLA matrix. This transformation enhances the composite's durability and mechanical performance, making it more suitable for biomedical applications where both strength and porosity are critical factors.

TABLE III. COMPRESSIVE STRENGTH, MODULUS, AND POROSITY OF THE COMPOSITES

Sample	Weight Ratio			Composite compressive strength (MPa)	Composite compressive modulus (GPa)	Porosity
	HAp	SiO ₂	PLA			
1	100	0	0	28.58	0.30	84.5
2	80	8	12	37.45	0.38	83.25
3	80	10	10	40.56	0.41	81.31
4	80	12	8	42.45	0.50	80.45
5	80	15	5	44.26	0.46	79.82

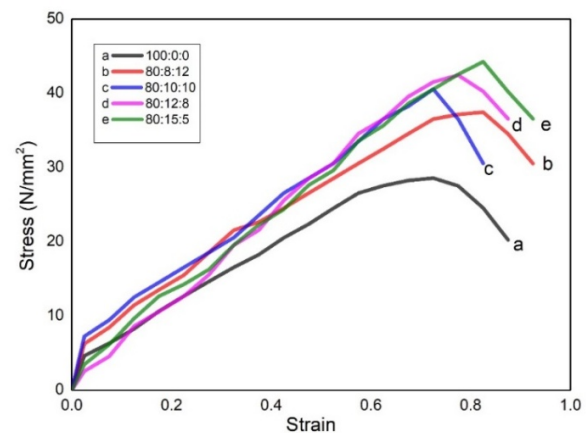


Fig. 8. Compressive strength of composites samples.

D. Wear Property

Goat jawbone powder, heat-treated at 1200 °C, and converted into HAp was used to synthesize the HAp/SiO₂/PLA composite. The composite was prepared in various proportions to create four different samples with a diameter of 10 mm and a length of 30 mm for wear testing. Wear testing was conducted on the HAp/SiO₂/PLA composite scaffolds using a pin-on-disc tribometer. The test parameters included a 10 N load, a sliding distance of 60 m, and a sliding speed of 150 rpm. The weight loss that compromises structural integrity is a failure indicator in wear testing. Table IV presents the weight loss, Coefficient of Friction (CoF), and the wear rate of the composites, while Figure 9 illustrates the CoF variation over time (1200 s).

TABLE IV. WEIGHT LOSS, COF, AND WEAR RATE

Sample	Weight Ratio			Weight loss (g)	CoF	Wear rate (mm ³ /Nm)
	HAp	SiO ₂	PLA			
1	100	0	0	0.0104	0.31062	0.82691
2	80	8	12	0.0226	0.37521	1.637203
3	80	10	10	0.0057	0.16863	1.353993
4	80	12	8	0.0015	0.14111	0.86743
5	80	15	5	0.0007	0.08944	0.639468

The incorporation of silica significantly enhances the composite's tribological performance. As the SiO₂ content increases, the CoF decreases from 0.31062 to 0.08944, while the wear rate decreases from 1.6372 mm³/Nm to 0.6394 mm³/Nm. Silica plays a crucial role in improving the wear resistance of the composite, making it more durable for potential applications. The incorporation of SiO₂ enhances wear resistance by improving interfacial bonding, grain refinement, and hardness. SiO₂ reduces friction, prevents abrasion, and forms a protective tribofilm, minimizing material loss. Its reinforcing properties distribute loads, fill voids, and resist crack propagation, making the composite durable for biomedical applications.

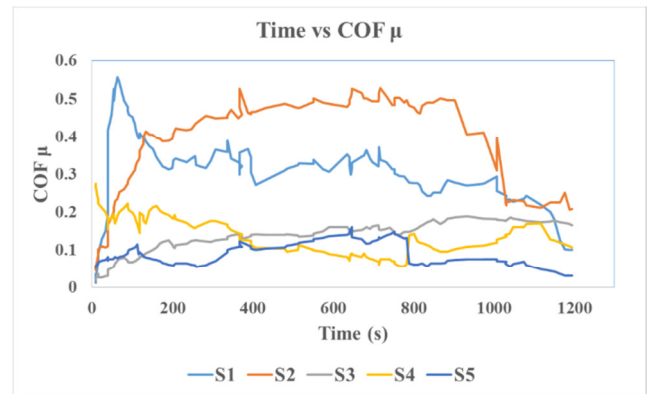


Fig. 9. CoF vs time graph of the composite samples.

E. Surface Morphology

The SEM images of the worn surfaces of nHAp/PLA composites with 5%, 8%, 10%, and 12% SiO₂ are presented in Figure 10.

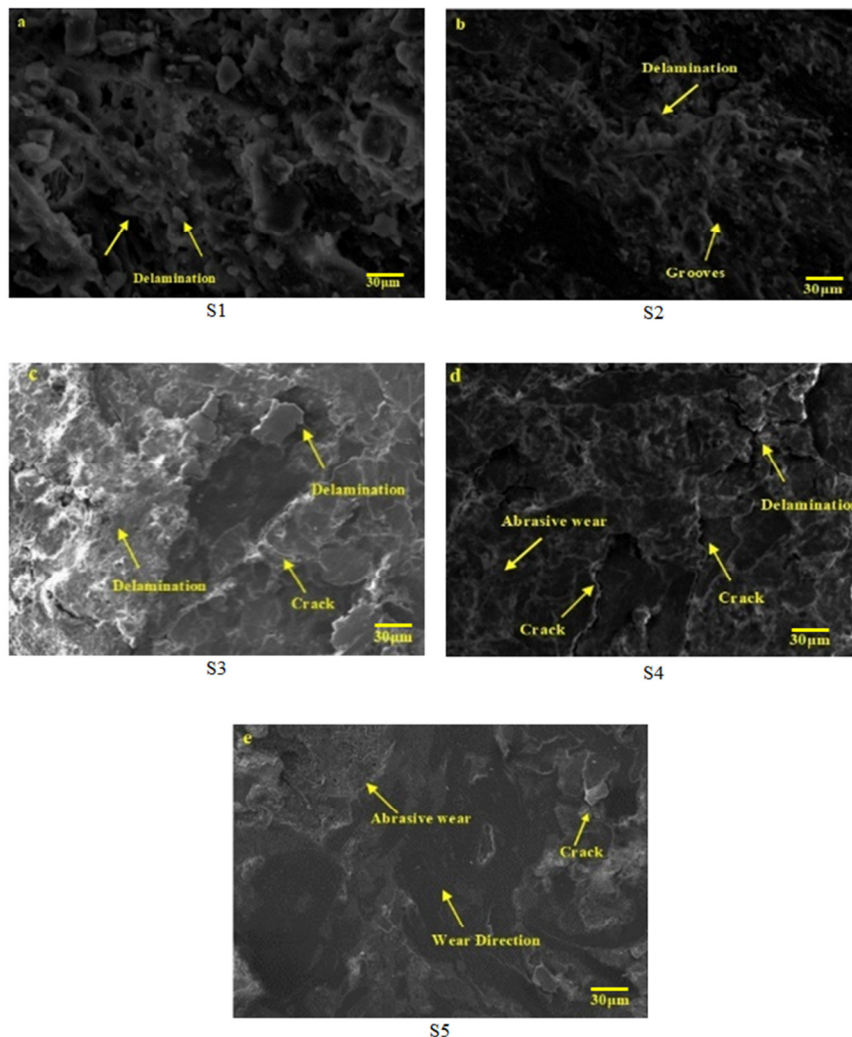


Fig. 10. SEM image micrographs of the worn surfaces of nHAp/SiO₂/PLA composites.

The composites samples were evaluated using a pin-on-disc tribometer under a 10 N load at 150 rpm to analyze their wear behavior. Post-wear compression testing would provide direct evidence, however, the current data strongly suggest durability without severe structural degradation. Combining tribological data, SEM analysis, literature review, and biomedical application requirements allows a reasonable inference that the composite maintains sufficient mechanical integrity after wear testing. Figure 10(a) shows significant delamination in the S1 composite after being subjected to a 10 N sliding load at 150 rpm for 1200 s, with a sliding distance of 60 mm. In contrast, Figure 10 (c) reveals that in the S3 composite HAp/SiO₂/PLA at 80:10:10 ratio, both delamination and wear grooves are present. The S1, S2, and S3 composites exhibit substantial delamination, cracks and grooves, as observed in Figures 10(a), (b), and (c). However, the S4 and S5 composites, which contain 12% and 15% SiO₂, show significantly fewer cracks, as seen in Figures 10(d) and (e), although some delamination is still evident. The addition of SiO₂ has notably improved the wear resistance of the composites under load conditions. In S1 and S2, the applied load has contributed to increased wear delamination. However, Figures 10(c), (d), and (e) demonstrate a reduction in abrasive wear, with material delamination occurring at controlled levels. Notably, Figure 10(e) highlights a significant reduction in abrasive wear, with the smallest amount of cracking observed. These findings confirm that the incremental addition of SiO₂ in the nHAp/PLA composite enhances its tribological properties, leading to better wear resistance and durability.

IV. CONCLUSION

This study successfully synthesized and characterized HAp/SiO₂/PLA composites derived from goat jawbone. The addition of SiO₂ significantly enhanced mechanical strength, reduced porosity, and improved tribological performance. Among the tested compositions, Sample 5 (80:15:5 HAp/SiO₂/PLA) demonstrated optimal properties, making it a promising candidate for bone tissue engineering applications. PLA is biodegradable, and HAp is bioactive, but SiO₂ incorporation may alter the composite's degradation rate. Long-term degradation effects of SiO₂ in physiological environments need further investigation to ensure safe use in implants. SEM and XRD analyses confirmed the composite's improved structural integrity and wear resistance. Additionally, its properties closely resemble those of human cancellous bone, highlighting its potential as a biomaterial for implants.

For future research, investigations could focus on in vitro and in vivo studies to assess biocompatibility and osteoconductivity, optimizing SiO₂ content to further enhance mechanical properties, and functionalizing the composite for drug delivery applications in bone regeneration.

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