



Evaluation of the Mechanical Integrity of Bovine Bone-Based Hydroxyapatite Pellets Across Various Solution pH Levels for Filtration Media Applications

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Abstract. Bovine bone-derived hydroxyapatite (HAp) is a promising porous bioceramic material for filtration applications, but its stability under different pH conditions remains a concern. This study evaluated the chemical stability and mechanical integrity of HAp pellets after immersion in buffer solutions at pH 3.92, 6.77, and 9.12 for 1 and 7 days. Chemical stability was evaluated from mass loss and pH evolution, while mechanical integrity was assessed by compressive strength. After 7 days, mass loss reached 5.30% at pH 3.92, compared with 2.41–2.43% at pH 6.77 and 9.12. Compressive strength decreased from 15.12 MPa initially to 9.09 MPa at pH 3.92, while 11.22 and 10.96 MPa were retained at pH 6.77 and 9.12, respectively. The acidic condition also increased the solution pH from 3.92 to 4.97, indicating progressive interaction between HAp and the surrounding solution. These results indicate that acidic conditions accelerate HAp dissolution and mechanical degradation, whereas neutral and alkaline conditions provide better stability.

Keywords: Bovine Bone, Filter Pellets, Hydroxyapatite, Mechanical Integrity, pH Variation.

1 Introduction

Water pollution in Bali remains a challenge for domestic and industrial water use, with industrial activities contributing to wastewater contamination and limitations in wastewater treatment and reuse (Chapagain et al., 2022; Widianingtiyas et al., 2023). Bali also generates a considerable amount of bovine bone waste from the food processing sector, most of which remains underutilized. According to the Department of Agriculture and Food Security of Bali Province, Bali had 550,350 Bali cattle in 2020, with an average live weight of 300–400 kg and a carcass proportion of approximately 57%, indicating a substantial potential source of bovine bone biomass for hydroxyapatite production (Dinas Pertanian dan Ketahanan Pangan Provinsi Bali, 2020). Bovine bone is a rich natural source of hydroxyapatite (HAp, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), a biocompatible and environmentally friendly ceramic material with excellent adsorption properties (Aissa & Othmani, 2023; Sembiring et al., 2025). Owing to its high affinity toward heavy metals and rapid adsorption kinetics, HAp has attracted

significant attention as a promising adsorbent for wastewater treatment applications (Nayak & Bhushan, 2021).

Despite these advantages, the practical application of HAp powder in continuous filtration systems is limited by particle agglomeration and clogging, which reduce filtration efficiency and complicate material recovery (Sorrentino et al., 2025). Converting HAp powder into pellet form offers an attractive engineering solution because pellets are easier to handle, recover, and regenerate during filtration processes. However, hydroxyapatite is inherently brittle and exhibits relatively low mechanical strength. Moreover, its chemical stability is strongly influenced by the surrounding pH. Under acidic conditions, HAp undergoes progressive dissolution, and the dissolution rate depends on the solution pH and the chemical species present in the medium (Margolis & Moreno, 1992). Continuous dissolution may increase pellet porosity, reduce the load-bearing capacity, and eventually cause structural failure during filtration operation.

Numerous studies have focused on enhancing the adsorption performance of bovine bone-derived hydroxyapatite in powder form. Various modification methods have been reported to improve its affinity toward heavy metals and other pollutants (Ahmed Zim et al., 2026; Jian et al., 2025; Li et al., 2025; Neolaka et al., 2023; Wan et al., 2025). Furthermore, previous investigations have demonstrated that calcination temperature and particle size significantly influence the adsorption efficiency of bovine bone-derived HAp (Widantha et al., 2024). The dissolution behavior of compacted HAp under different pH conditions has also been examined, revealing that lower porosity improves structural integrity, while acidic environments produce the highest mass dissolution rates (Widantha, 2024). These findings indicate that although the adsorption behavior and dissolution characteristics of hydroxyapatite have been extensively investigated, the mechanical durability of pelletized HAp under different chemical environments remains insufficiently understood. In particular, the relationship between solution pH, chemical stability, and mechanical integrity has not been comprehensively evaluated for filtration applications.

Therefore, the novelty of this study lies in the integrated evaluation of the chemical stability and mechanical integrity of pelletized bovine bone-derived hydroxyapatite under different pH conditions, by linking mass loss and solution pH evolution with changes in compressive strength. This study aims to evaluate the chemical stability and mechanical integrity of bovine bone-derived hydroxyapatite pellets after exposure to acidic, neutral, and alkaline environments. The effects of solution pH and immersion time on mass loss and compressive strength were investigated to determine the operational durability of HAp pellets. The findings are expected to provide fundamental information for the development of durable and sustainable hydroxyapatite-based filtration media for industrial wastewater treatment.

2 Methodology

2.1 Preparation of Hydroxyapatite Pellets

This study employed an experimental approach to evaluate the mechanical integrity of bovine bone-derived hydroxyapatite pellets under different pH environments. The overall experimental procedure is presented in Figure 1.



Figure 1. Experimental workflow illustrating the preparation of bovine bone-derived hydroxyapatite pellets, chemical exposure in different pH environments, and mechanical integrity evaluation.

Fresh bovine bones, consisting of the major hard bone portions obtained from cattle slaughtering in Gianyar, Bali, Indonesia, were collected immediately after cutting. Bones were cleaned to remove residual soft tissues, boiled, and immersed in a sodium hydroxide (NaOH) solution to remove organic impurities. The cleaned bones were dried and calcined at 800 °C using a heating rate of approximately 5°C min⁻¹ and held for 2 hours. The calcined product was subsequently ground and sieved to produce a homogeneous powder. The HAp powder was compacted into cylindrical pellets with a diameter of 20 mm and a thickness of 7 mm using a hydraulic press with an applied load of 20 kN, which was maintained for 60 s. The green pellets were then sintered at 800 °C. The density of the sintered HAp pellets was determined from their measured mass and geometric volume. The porosity was then estimated from the measured density relative to the theoretical density of hydroxyapatite.

2.2 Chemical Exposure Test

The sintered HAp pellets were immersed individually in 100 mL of three standard buffer solutions, with nominal pH values of 4.01, 6.86, and 9.18, with measured initial pH values of 3.92, 6.77, and 9.12, respectively. Each pellet was immersed in 100 mL of buffer solution at room temperature under static conditions, and the buffer solution was not replaced during the immersion period. The pellets were immersed for 1 day (24 h) and 7 days (168 h). For each pH condition and immersion duration, three independent pellet specimens ($n = 3$) were used. The initial mass of each pellet was measured before immersion using an analytical balance. After each immersion period, the pellets were

rinsed with distilled water, dried at room temperature, and weighed again to determine the percentage of mass loss.

The percentage of mass loss was calculated using Eq. (1):

$$\text{Mass Loss (\%)} = \frac{m_0 - m_t}{m_0} \times 100\% \quad (1)$$

where m_0 is the initial mass before immersion and m_t is the mass after immersion.

2.3 Mechanical Integrity Evaluation

Following chemical exposure, the mechanical integrity of the HAp pellets was evaluated using a Universal Testing Machine (UTM, LABTEK) equipped with a 100 kN load cell. The cylindrical pellets were subjected to uniaxial compression in the axial direction between flat compression platens. The test was conducted at a crosshead speed of 5 mm min⁻¹ until fracture, and the maximum load was recorded.

The compressive strength was calculated using Eq. (2):

$$\sigma = \frac{F}{A} \quad (2)$$

where σ is the compressive strength (MPa), F is the maximum compressive load (N), and A is the cross-sectional area of the pellet (mm²).

3 Results and Discussion

3.1 Initial Characteristics of Bovine Bone-Derived Hydroxyapatite Pellets

The bovine bone-derived hydroxyapatite (HAp) pellets were successfully fabricated through calcination, hydraulic pressing, and sintering, producing cylindrical specimens. Visual observation showed that all pellets possessed a relatively smooth surface without visible cracks or edge defects, indicating that the compaction and sintering processes produced structurally intact specimens suitable for subsequent immersion tests.

Table 1. Initial Physical and Mechanical Characteristics of the Sintered Bovine Bone-Derived Hydroxyapatite Pellets.

Property	Mean $\pm$ SD
Density (g cm ⁻³)	2.31 $\pm$ 0.04
Porosity (%)	26.7 $\pm$ 1.3
Compressive strength (MPa)	15.12 $\pm$ 0.23

The initial physical and mechanical properties of the fabricated hydroxyapatite pellets are presented in Table 1. The pellets exhibited an average density of 2.31 $\pm$ 0.04 g cm⁻³, a porosity of 26.7 $\pm$ 1.3%, and a compressive strength of 15.12 $\pm$ 0.23 MPa. These results indicate that the fabrication process produced porous ceramic pellets with adequate mechanical integrity for subsequent filtration applications. The relatively high porosity is beneficial for filtration because it provides interconnected pathways for fluid

transport and increases the available surface area for adsorption (Surya et al., 2024; Xin et al., 2021). However, increasing porosity generally decreases the compressive strength of hydroxyapatite ceramic. Therefore, an appropriate balance between porosity and mechanical integrity is essential to ensure both filtration efficiency and structural stability.

3.2 Effect of Solution pH on the Chemical Stability of Hydroxyapatite Pellets

The effect of solution pH on the chemical stability of bovine bone-derived hydroxyapatite pellets is presented in Figure 2. The results show that both solution pH and immersion time influenced the mass loss of the pellets. The highest mass loss was observed under acidic conditions, reaching $2.91 \pm 0.33\%$ after 1 day and increasing to $5.30 \pm 0.29\%$ after 7 days. In contrast, the pellets immersed in neutral and alkaline solutions exhibited considerably lower mass losses, ranging from 1.36–1.42% after 1 day and increasing to only 2.41–2.43% after 7 days.

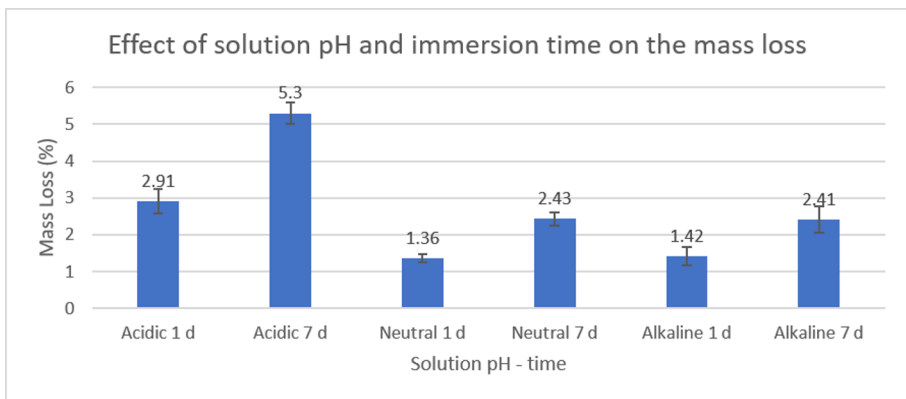


Figure 2. Effect of solution pH and immersion time on the mass loss of bovine bone-derived hydroxyapatite pellets.

The evolution of the solution pH during immersion is shown in Figure 3. The acidic solution exhibited the largest pH increase, rising from 3.92 at the beginning of the test to 4.97 after 7 days. In contrast, only slight pH changes were observed in the neutral and alkaline solutions, increasing from 6.77 to 7.12 and 9.12 to 9.51, respectively. The gradual increase in pH indicates that the chemical interaction between the hydroxyapatite pellets and the surrounding solution altered the solution chemistry throughout the immersion period.

The combined results of mass loss and solution pH evolution indicate that hydroxyapatite was least chemically stable under acidic conditions. The acidic solution produced the highest mass loss and the greatest increase in solution pH throughout the immersion period, whereas only minor changes were observed in the neutral and alkaline solutions. This behavior is consistent with previous studies showing that hydroxyapatite dissolves more rapidly at low pH because excess hydrogen ions attack the apatite crystal lattice, promoting the release of calcium and phosphate ions into the

solution (Margolis & Moreno, 1992; Shellis et al., 2010). The gradual increase in pH under acidic conditions is attributed to the consumption of hydrogen ions during the dissolution process, resulting in partial neutralization of the solution (Fandrich et al., 2026; Pearce, 1988). In contrast, neutral and alkaline environments suppress hydroxyapatite dissolution, resulting in lower mass loss and better chemical stability (Bengtsson et al., 2009; Widantha, 2024).

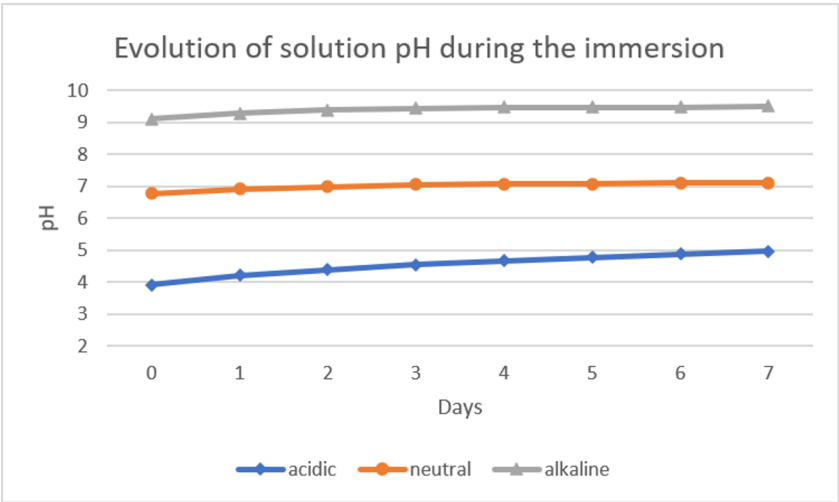


Figure 3. Evolution of solution pH during the immersion of bovine bone-derived hydroxyapatite pellets in acidic, neutral, and alkaline buffer solutions.

3.3 Effect of Solution pH on the Mechanical Integrity of Hydroxyapatite Pellets

The effect of solution pH and immersion time on the compressive strength of bovine bone-derived hydroxyapatite pellets is presented in Figure 4. The untreated pellets exhibited an average compressive strength of 15.12 ± 0.23 MPa. After 1 day of immersion, the compressive strength decreased to 11.47 ± 0.72 MPa in the acidic solution, whereas the neutral and alkaline solutions retained higher strengths of 13.78 ± 0.38 MPa and 13.75 ± 0.29 MPa, respectively. Following 7 days of immersion, the compressive strength further decreased to 9.09 ± 0.36 MPa under acidic conditions, while the neutral and alkaline solutions exhibited compressive strengths of 11.22 ± 0.45 MPa and 10.96 ± 0.43 MPa, respectively. These results indicate that both immersion time and solution pH affected the mechanical integrity of the hydroxyapatite pellets.

The reduction in compressive strength observed after acidic immersion is attributed to the accelerated dissolution of hydroxyapatite under low-pH conditions. Acidic environments promote the release of calcium and phosphate ions from the HAp matrix, resulting in the loss of load-bearing material and the development of a more porous microstructure, which ultimately reduces the mechanical integrity of the bioceramic (Shellis et al., 2010). In contrast, neutral and alkaline environments slow the dissolution

process, thereby better preserving the structural integrity and compressive strength of the pellets. Similar findings have been reported by Yang et al., (2023), who demonstrated that acidic exposure decreased the mechanical properties of hydroxyapatite-based bioceramics through increased porosity and microstructural degradation.

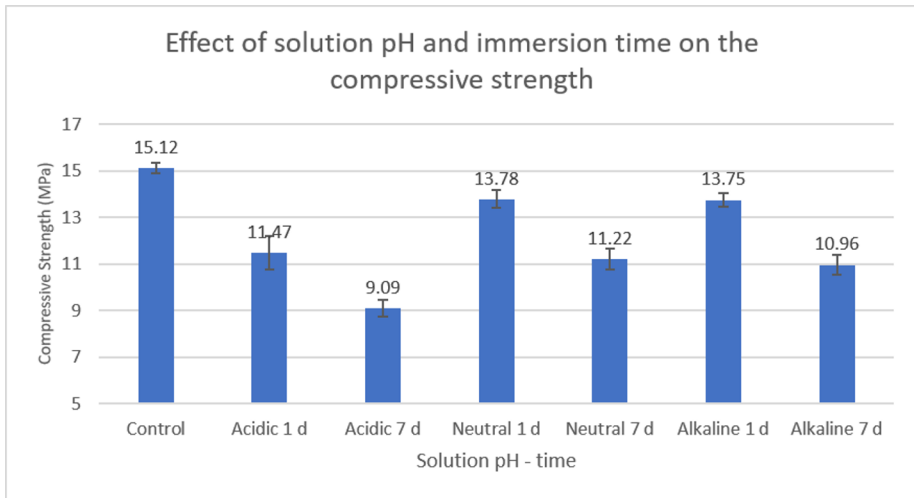


Figure 4. Effect of solution pH and immersion time on the compressive strength of bovine bone-derived hydroxyapatite pellets.

4 Conclusion

Bovine bone-derived hydroxyapatite pellets were successfully fabricated with a density of 2.31 g cm^{-3} , a porosity of 26.7%, and a compressive strength of 15.12 MPa, indicating good initial physical and mechanical characteristics. Solution pH affected both the chemical stability and mechanical integrity of the pellets. Acidic conditions accelerated hydroxyapatite dissolution, resulting in higher mass loss and greater reductions in compressive strength compared with neutral and alkaline environments. These results demonstrate the sensitivity of pelletized hydroxyapatite to chemical exposure and provide a basis for further development of porous HAp materials. However, this study was limited to short-term immersion and mechanical evaluation, without direct assessment of adsorption performance, flow resistance, or long-term filtration durability. Future studies should therefore evaluate pollutant removal performance and long-term performance under realistic wastewater conditions.

Acknowledgment

The authors gratefully acknowledge the financial support provided by Politeknik Negeri Bali through the Regular DIPA Research Scheme under Contract No.

06893/DST/PL8/AL.04/2026. The authors also thank the Mechanical Engineering Department, Politeknik Negeri Bali, for providing laboratory facilities and technical support throughout this research.

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