



Porosity-Controlled Degradation Behavior of Hydroxyapatite Synthesized from Type III Dental Gypsum Waste

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Abstract

Type III of dental gypsum waste is commonly discarded as medical waste despite containing high amounts of calcium-based minerals with potential for biomaterial synthesis. This study aimed to develop porous hydroxyapatite (HAp) scaffolds derived from dental gypsum waste and evaluate their physicochemical characteristics and degradation behavior for bone tissue engineering applications. Hydroxyapatite was synthesized through a hydrothermal reaction using dental gypsum waste as the calcium precursor and diammonium hydrogen phosphate as the phosphate source. Porous scaffolds were fabricated using sucrose as a porogen with Dental Gypsum Hydroxyapatite (DGHAp) type III : sucrose ratios of 35:65, 45:55, and 55:45, followed by sintering at 950–1150°C. FTIR analysis confirmed the formation of characteristic phosphate and hydroxyl groups of hydroxyapatites, along with carbonate groups indicating carbonated hydroxyapatite formation. SEM observations revealed interconnected porous structures in all scaffold groups. Higher sucrose content increased pore size and porosity, whereas higher DGHAp content produced denser scaffold structures. Degradation testing demonstrated that higher porosity increased scaffold degradation, while higher sintering temperatures reduced degradation due to increased crystallinity and densification. Among all groups, DGHAp 45% showed the most balanced characteristics regarding porosity and structural stability. These findings demonstrate the potential of dental gypsum waste as a sustainable precursor for porous hydroxyapatite scaffolds with controllable degradation behavior.

Keywords: Dental Gypsum type III; Dental Gypsum Hydroxyapatite; Hydroxyapatite

Background

Type III dental gypsum waste is one of the major solid wastes generated in dental laboratories, particularly during the fabrication of working casts, study models, and prosthodontic procedures. After clinical use, gypsum materials are generally discarded as medical waste and may contribute to environmental pollution if not properly managed. However, this waste still contains high amounts of calcium-based minerals, making it a potential raw material

for sustainable biomaterial synthesis [1].

Chemically, type III dental gypsum mainly consists of calcium sulfate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$). During clinical manipulation, when mixed with water, the material undergoes a hydration reaction and transforms into calcium sulfate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). This reaction is reversible, allowing hardened gypsum to be reconverted into the hemihydrate phase through crushing and calcination at elevated temperatures [1,2]. This reversible characteristic

creates an opportunity to reutilize dental gypsum waste as a calcium precursor for calcium phosphate-based biomaterials.

Among calcium phosphate biomaterials, hydroxyapatite (HAp) $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$ has been widely used in bone tissue engineering because of its osteoconductive properties, biocompatibility, and chemical similarity to the mineral phase of natural bone [3-5]. However, dense and highly crystalline hydroxyapatite is known to exhibit slow biodegradation and low resorption rates under physiological conditions, which may limit the remodeling process during bone regeneration [3,6]. Therefore, controlling the degradation behavior of hydroxyapatite scaffolds becomes an important consideration in scaffold design.

One strategy to enhance hydroxyapatite biodegradation is porosity engineering. Increasing scaffold porosity can enlarge surface area and improve fluid penetration into the scaffold structure, thereby accelerating dissolution and degradation behavior [7,8]. In addition, interconnected porous structures facilitate nutrient transport and tissue infiltration. Pore sizes ranging from approximately 200-500 μm are considered favorable for osteoblast infiltration and vascularization in bone tissue engineering applications [7].

In this study, sucrose was introduced as a porogen agent to generate porous structures within hydroxyapatite scaffolds synthesized from type III Dental Gypsum Hydroxyapatite (DGHAp) waste. The sucrose-containing mixtures were compacted and sintered at high temperatures, allowing sucrose to thermally decompose and leave interconnected pores within the scaffold matrix.

Hydroxyapatite synthesis from gypsum-derived materials has previously been reported, the feasibility of converting gypsum waste into porous hydroxyapatite monoliths through hydrothermal treatment using diammonium hydrogen phosphate [1]. Their findings showed that gypsum waste could be transformed into functional porous hydroxyapatite materials suitable for biomaterial applications.

In the present study, the synthesized scaffolds were characterized using Fourier Transform Infrared Spectroscopy (FTIR) to identify characteristic hydroxyapatite functional groups and evaluate the possible formation of carbonated apatite resembling natural bone mineral. Surface morphology and pore structures were analyzed using Scanning Electron Microscopy (SEM). Furthermore, degradation testing in simulated physiological and inflammatory environments was performed to evaluate scaffold stability and dissolution behavior.

This study aims not only to recycle dental gypsum waste but also to upcycle medical waste into value-added sustainable porous biomaterials with controllable degradation behavior for bone tissue engineering applications.

Results and Discussion

FTIR Characterization of DGHAp Scaffolds

FTIR characterization confirmed the successful formation of hydroxyapatite from type III dental gypsum waste. All scaffold groups exhibited characteristic hydroxyapatite absorption bands without significant chemical alterations after sintering.

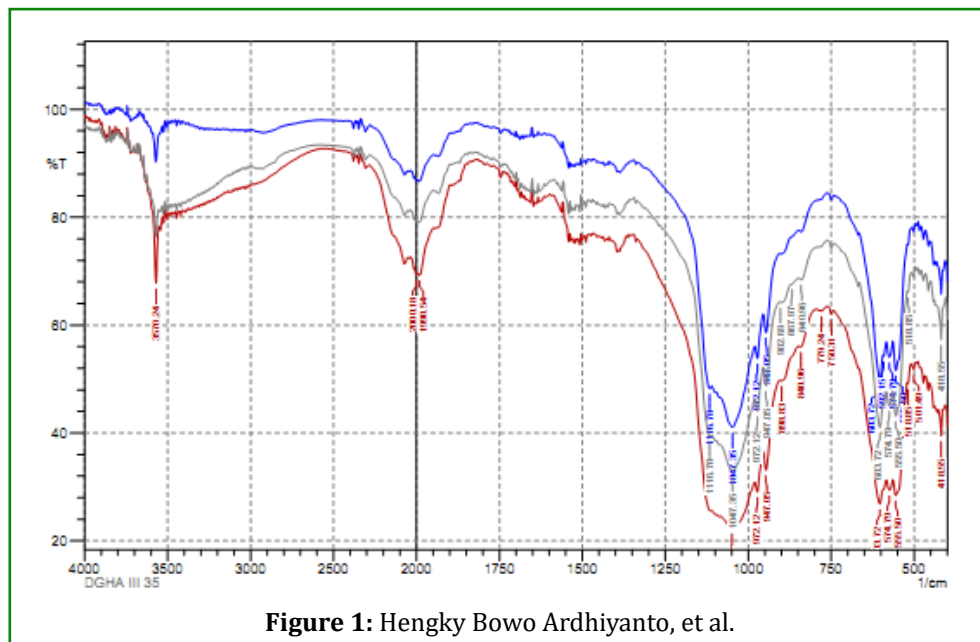


Figure 1: Hengky Bowo Ardhiyanto, et al.

The absorption bands (Figure 1) observed at approximately $1030\text{-}1090\text{ cm}^{-1}$ corresponded to asymmetric stretching vibrations of phosphate groups (PO_4^{3-}), while the peak around 960 cm^{-1} represented symmetric phosphate vibrations. In addition, bands observed between $560\text{-}605\text{ cm}^{-1}$ indicated phosphate bending vibrations characteristic of hydroxyapatite [9,10].

Hydroxyl group (OH^-) absorption was identified at approximately $3570\text{-}3640\text{ cm}^{-1}$ and around 630 cm^{-1} , confirming the preservation of the hydroxyapatite structure after high-temperature sintering [9]. Differences in spectral intensity indicated that DGHAp : Sucrose ratios affected scaffold crystallinity. DGHAp 55% exhibited sharper and more intense absorption bands, indicating higher crystallinity and hydroxyapatite content. In contrast, DGHAp 35% displayed broader and less intense bands due to higher porogen content, resulting in lower structural density and increased porosity.

These findings demonstrated that type III dental gypsum waste could be successfully converted into hydroxyapatite with chemical characteristics comparable to conventional synthetic hydroxyapatite.

SEM Morphological Analysis of DGHAp Scaffolds

SEM observations (Figure 2) demonstrated that all scaffold groups exhibited interconnected porous structures, which are essential for bone tissue engineering applications [7].

DGHAp 35% scaffolds exhibited the highest porosity with pore sizes ranging from $300\text{-}600\text{ }\mu\text{m}$. The large and irregular pore structures resulted from the high sucrose content used as the porogen. The increased porosity enlarged scaffold surface area and enhanced fluid penetration, which potentially accelerated degradation behavior. However, excessive pore formation reduced structural compactness and mechanical stability [8]. DGHAp 45% scaffolds exhibited more homogeneous pore distribution with pore sizes ranging from $250\text{-}500\text{ }\mu\text{m}$. The scaffold walls appeared more compact compared to the DGHAp 35% group. This composition provided a more balanced combination of porosity and structural integrity. The pore size range remained favorable for osteoblast infiltration and nutrient transport while maintaining improved scaffold stability [7]. DGHAp 55% scaffolds exhibited the densest structures with smaller pore sizes of approximately $200\text{-}300\text{ }\mu\text{m}$. Reduced sucrose content resulted in decreased pore formation and increased scaffold compactness. Although this structure provided greater mechanical stability, the lower porosity may reduce fluid penetration and slow scaffold degradation behavior.

Overall, SEM analysis demonstrated that increasing hydroxyapatite content produced denser and more stable scaffold structures, whereas increasing sucrose content enhanced porosity and potentially increased degradation behavior.

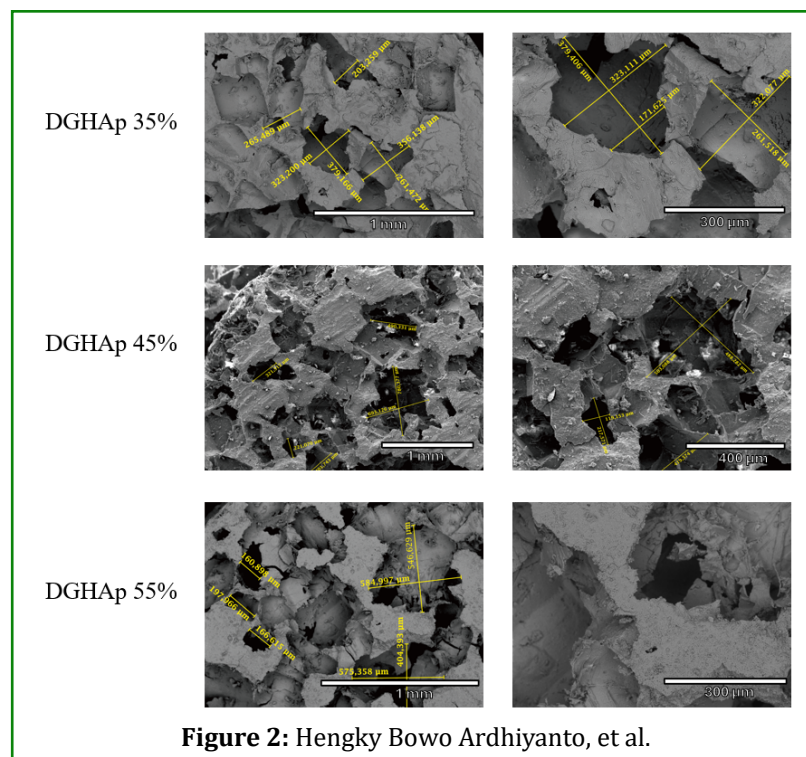


Figure 2: Hengky Bowo Ardhiyanto, et al.

Degradation Behavior of DGHAp Scaffolds

The degradation test results in table 1 demonstrated that all scaffolds exhibited higher degradation rates under acidic conditions (pH 3) than under physiological conditions (pH 7.4). This result indicated that hydroxyapatite dissolution was strongly influenced by environmental pH.

$$D = \frac{W_0 - W_1}{W_0} \times 100\%$$

D = degradation

W_0 = weight of the scaffold before the experiment

W_1 = weight of the post-experimental scaffold.

Scaffolds sintered at 950°C showed the highest degradation values, reaching 24.23% at pH 3 and 10.07% at pH 7.4. In contrast, scaffolds sintered at 1150°C exhibited the lowest degradation rates, namely 16.17% at pH 3 and 7.32% at pH 7.4.

The higher degradation observed in acidic conditions was attributed to the increased solubility of calcium phosphate materials in low-pH environments. Hydrogen ions (H⁺) accelerated hydroxyapatite demineralization by increasing calcium phosphate dissolution [4,11].

The degradation behavior was strongly influenced by scaffold porosity and sintering temperature. Lower sintering temperatures produced scaffolds with higher porosity and lower densification, thereby facilitating fluid penetration and accelerating degradation. In contrast, higher sintering temperatures increased crystallinity and densification, reducing pore volume and limiting liquid diffusion into the scaffold structure [10,11].

Sintering Temperature	Degradation	
	pH 3 (%)	pH 7,4 (%)
950°C	24,23 ± 1,70	10,07 ± 0,38
1050°C	18,10 ± 0,57	8,37 ± 1,18
1150°C	16,17 ± 1,28	7,32 ± 1,22

Table 1: Degradation percentage of DGHA scaffolds sintered at different temperatures after immersion in citric acid buffer (pH 3) and phosphate-buffered saline (PBS, pH 7.4). Higher degradation was observed under acidic conditions, while increasing sintering temperature reduced scaffold degradation due to increased densification and crystallinity. Values are presented as mean ± standard deviation.

These findings demonstrated that porosity engineering played a critical role in controlling the degradation behavior of highly crystalline hydroxyapatite scaffolds. By increasing

scaffold porosity through sucrose incorporation, surface area and scaffold–fluid interaction were enhanced, resulting in improved biodegradation behavior.

Among all scaffold groups, DGHA 45% exhibited the most balanced characteristics between porosity, structural integrity, and degradation behavior. This composition provided sufficient pore interconnectivity to facilitate scaffold dissolution while maintaining adequate structural compactness. Overall, the results indicate that hydroxyapatite derived from type III dental gypsum waste possesses tunable degradation behavior and favorable porous morphology, supporting its potential application as a sustainable biomaterial for bone tissue engineering.

Conclusion

This study successfully developed porous hydroxyapatite scaffolds derived from type III dental gypsum waste through hydrothermal synthesis and porosity engineering approaches. The results demonstrated that dental gypsum waste can be effectively upcycled into value-added sustainable biomaterials for bone tissue engineering applications.

FTIR characterization confirmed the successful formation of hydroxyapatite, as indicated by the presence of characteristic phosphate (PO₄³⁻) and hydroxyl (OH⁻) functional groups.

SEM observations revealed that the addition of sucrose successfully generated interconnected porous structures within the scaffold matrix. Increased sucrose content produced higher porosity and larger pore sizes, whereas increased DGHAp content resulted in denser and more compact scaffold structures. Among all groups, DGHAp 45% demonstrated the most balanced characteristics in terms of pore distribution, porosity, and structural stability.

The degradation study showed that scaffold degradation behavior was strongly influenced by porosity and sintering temperature. Scaffolds with higher porosity exhibited greater degradation due to increased surface area and enhanced fluid penetration. In contrast, higher sintering temperatures increased scaffold crystallinity and densification, thereby reducing degradation rates. These findings confirmed that porosity engineering plays an important role in controlling the biodegradation behavior of highly crystalline hydroxyapatite scaffolds.

Overall, this study demonstrated that type III dental gypsum waste have a potential as a calcium precursor for the fabrication of porous hydroxyapatite scaffolds with controllable degradation behavior. The developed scaffolds exhibited favorable chemical characteristics, interconnected porous morphology, and tunable biodegradation properties,

supporting their potential application in bone tissue engineering and hard tissue regeneration.

Declaration of Interest

The authors declare no potential conflict of interest.

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