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Study on the degradation behavior and mechanical properties of Diopside in simulated body fluid at pH 7.4

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ABSTRACT

This study explored the impact of immersion time in simulated body fluid on the degradation behavior and mechanical properties of a diopside composite. The selected immersion durations were 6 h and 2, 3, 7, 14, and 30 days. The composite was synthesized using dolomite (CaMg(CO₃)₂) as the raw material, with experimental results analyzed using X-ray diffraction.
The initial results indicated a weight reduction of 0.93% after 6 h of immersion, which shifted to a slight weight increase of 0.08% after 30 days. Porosity rose by 7.67% in the first 6 h due to dissolution but diminished to 3.54% after 30 days following apatite deposition. Mechanical testing revealed that microhardness declined from 4.7 GPa to 4 GPa within 2 days and further decreased to 3.2 GPa by day 14, where it stabilized. Notably, this stabilized value is close to the hardness of tooth enamel. A decrease in flexural strength was also observed, dropping from 167.4 to 129.6 MPa by day 14, aligning with values typically found in cortical bone. X-ray diffraction analysis after 14 days of immersion confirmed the formation of a hydroxyapatite layer during dissolution, highlighting diopside's pronounced bioactivity. Its robust mechanical properties combined with an acceptable level of bioactivity make it a promising candidate for load-bearing biomedical applications, offering durability and resistance to degradation in simulated body fluid environments.

Keywords: Simulated Body Fluid; Diopside; Dolomite; Microhardness; Flexural strength; Hydroxyapatite.

Graphical abstract
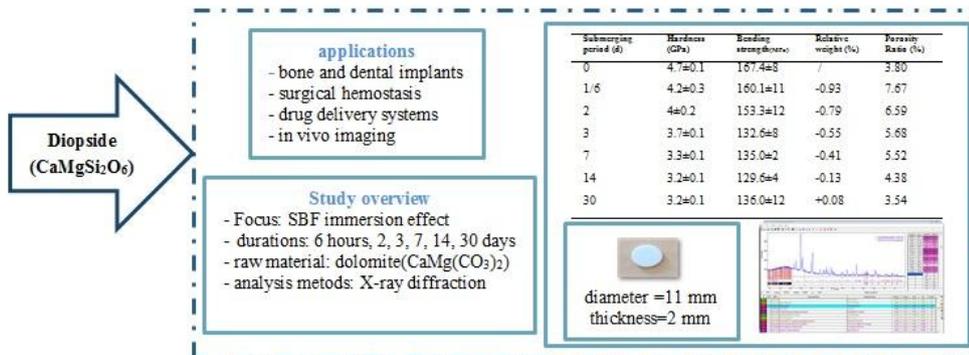
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1. Introduction

Many people around the world suffer from bone-related diseases, regardless of their gender or age. These diseases may result from infections within the skeletal system, injuries caused by accidents, or congenital abnormalities. In such cases, reconstructive surgery becomes essential to improve the quality of life as much as possible. Prosthetic implants replace the damaged part of the body, aiming to perform its function effectively while also providing additional support to surrounding tissues and enhancing the patient's external appearance [1].

These replacements must exhibit biocompatibility to ensure they do not trigger toxic reactions when implanted in the body. Additionally, they need to possess mechanical properties comparable to those of natural bone. Despite notable advancements in this field, current bone substitutes still face limitations, such as inadequate structural load-bearing capacity and insufficient stimulation of effective bone regeneration. The solution may lie in bio-ceramics, which combine superior mechanical efficiency with high bioactivity, offering a promising path forward [2].

In the early 20th century, research began to focus on bioceramics as potential alternatives in orthopedic surgery due to their unique physicochemical properties, which are not found in other organic or metallic materials. Among the most notable bioceramics are the calcium phosphate family, including hydroxyapatite, biphasic calcium phosphate (BCP), and beta-tricalcium phosphate (β-TCP), as well as bioactive glasses and calcium silicate compounds [3-6]. Within this group lies diopside, which serves as the primary subject of this study.

Diopside, with the chemical formula $CaMgSi_2O_6$, is recognized as a bio-ceramic material belonging to the group of bioactive silicates. These bioactive silicates have the ability to release silicate ions in varying concentrations, which stimulate osteoblast growth a critical factor for bone calcification metabolism. This not only promotes improved bone density but also helps prevent osteoporosis [7,8]. Diopside has also been found to form an apatite layer outside the body, bond with bone tissue inside the body, and exhibit excellent mechanical properties. Furthermore, its composition resembles that of wollastonite and akermanite, but it has a relatively slower degradation rate, making it highly favorable for medical applications [9].

Due to its scarcity in nature and the growing demand for diopside, it has become essential to explore cost-effective methods for its synthesis while maintaining optimal sintering performance and preserving its key properties. Like other ceramic materials, diopside requires specific preparation techniques. Common methods include the sol-gel technique [10] and precipitation methods [11], which yield homogeneous samples at low temperatures. However, these methods often involve hazardous chemicals that can negatively impact both human health and the environment. Additionally, they are relatively expensive. An alternative approach involves preparing diopside using solid raw materials, a simpler, more cost-effective, and environmentally friendly method. However, this method typically produces samples with a relatively large grain size and requires high sintering temperatures. Factors such as densification rate, grain size, porosity, and other microstructural features which are influenced by processing parameters such as milling and sintering temperature can affect the resulting mechanical properties [12].

These challenges have been addressed in the preparation of hydroxyapatite. For instance, a specific tabletop wet milling setup has been successfully employed to produce sub-micron-sized NHA powders [13]. Utilizing this approach, we successfully synthesized nano-sized diopside (average crystalline size of 660 nm) using locally sourced dolomite ($CaCO_3 \cdot MgCO_3$) [14], achieving a theoretical sintering density of 97% for diopside sintered at 1300°C for two hours. The resulting material demonstrated excellent mechanical properties, including a microhardness value of 4.7 GPa and a three-point flexural strength of approximately 200 MPa [15] nearly five times higher than those reported for materials like S545 Bioglass and HA [16].

Bio-ceramics generally interact with living tissues and must maintain the strength and stability of their mechanical properties. To evaluate the impact of the physiological environment on the properties of bio-ceramics, Yoshimi et al., for instance, studied the effect of immersion time in simulated body fluid on HAp/poly-L-lactide (3%). Their findings showed a rapid decline in bending strength from 13 MPa to 2 MPa, along with a relative weight loss percentage of 0.15% and a sintering rate of 30% after 28 days of immersion. However, apatite-like bone formation was observed on the surface of the samples after just 4 days of immersion [17]. Additionally, both in vitro and in vivo studies demonstrated that magnesium significantly enhances the mechanical properties and degradation rate of $CaSiO_3$, which are critical considerations in designing scaffolds for bone regeneration [18].

In the present work, we report on the investigate the degradation behavior and mechanical properties of easily

fabricated diopside in a simulated body fluid at a pH of 7.4, with the goal of evaluating its potential use in bone tissue regeneration applications.

2. Materials and Methods

2.1. Preparation of specimens

Samples of $CaMgSi_2O_6$ were prepared using the solid-state reaction method in accordance with our previous study [15]. This involved mixing high-purity SiO_2 with doloma ($CaO·MgO$) extracted from locally sourced dolomite ($CaMg(CO_3)_2$). The dolomite was calcined at 950°C for 4 hours, resulting in doloma with a purity of approximately 99.6%. The doloma was then mixed with 55.5 wt.% SiO_2, and the resulting mixtures were ball-milled using a custom-designed milling system [15] in an aqueous medium for 4 hours. Notably, a comprehensive study detailing the fundamental properties and key advantages of this modified milling system has been published, where its effectiveness was also demonstrated in the sintering of natural hydroxyapatite [13].

It is worth emphasizing the critical importance of this initial processing step, which was a focal point of our earlier investigations [14,15,19,20]. After milling, the resulting mixture was dried and calcined at 700°C for 2 hours. The calcined powder was then uniaxially pressed at a pressure of 112 MPa to form 13 mm diameter disks. Finally, these disks were sintered at 1300°C for 2 hours to complete the preparation process. X-ray diffraction (XRD) analysis was carried out to identify the crystal phases of the prepared samples. The analysis utilized the BRUKER D8 ADVANCE instrument located in Karlsruhe, Germany, with filtered CuKα radiation and a nickel filter. The measurements were conducted using a step size of 0.03 and a step duration of 50 seconds.

2.2. Degradation behaviour of diopside in simulated body fluid

The degradation behavior of diopside was examined in a simulated body fluid (SBF) at pH 7.4. diopside discs were placed into 50 mL Falcon™ tubes, and the buffer solution volume was determined using the formula $V_s=S_a/10$, where V_s is the required solution volume in milliliters, and S_a is the disc's surface area in square millimeters. The average surface area of the sintered discs was calculated to be approximately 260 mm². Based on this, the discs were immersed in 26 mL of solution and stored at 37 °C for durations of 6 hours, as well as for 2, 3, 7, 14, and 30 days.

At each specified time point, the discs were retrieved, rinsed with Milli-Q water, and dried at 100 °C for one day. The change in weight was calculated as a percentage of the initial weight (W%) for diopside samples after immersion in SBF solutions using the following equation:

$$W\% = \frac{m_f - m_i}{m_i} \times 100 \qquad (1$$

m_i represents the weight of diopside prior to immersion, while m_f denotes its weight following immersion. For statistical analysis, three samples were tested for each time interval. Equations (2) and (3) were applied as part of the calculation process to determine the scaffold's porosity, which was derived based on the density retained within the material, as outlined in reference [21].

$$porosity\% = 1 - \frac{\rho\ relative}{\rho\ bulk} \qquad (2$$

$$\rho\ relative = \frac{W_d}{V} \qquad (3$$

Where ρ relative represents the relative density calculated using equation (3), ρ bulk denotes the bulk density measured in grams per cubic centimeter (g/cm³), W_d signifies the dry weight of the diopside scaffold in grams (g), and V refers to the total volume of the diopside scaffold expressed in cubic centimeters (cm³).

2.3. Mechanical properties of diopside

The mechanical properties of diopside, including its tensile strength and hardness, were comprehensively evaluated both before and after exposure to a simulated body fluid (SBF) environment. The SBF was maintained at physiological

conditions with a pH of 7.4 and a temperature of 37 °C, and the diopside samples were soaked for varying durations of 6 hours, as well as 2, 3, 7, 14, and 30 days. To quantify hardness, Vickers hardness values were calculated in accordance with the ASTM C1327 standard. This process involved determining the hardness measurements using a controlled load of 7 N applied over a dwelling time of 15 seconds. For each individual disc-shaped sample (n = 8) with a diameter of 11 mm and a thickness of 2 mm, eight separate indents were produced. The diagonals of these indents were then accurately measured utilizing scanning electron microscopy (SEM) for enhanced precision, with the analyses performed on a Zeiss Ultra device from Germany.

To ensure consistency and reliability in the results, the surfaces of the samples were carefully prepared prior to testing. This preparation involved grinding and polishing the samples using silicon carbide (SiC) grinding paper followed by fine polishing with polycrystalline diamond suspensions of 1 µm in particle size. The final determination of hardness was conducted using the Munz and Fett equation [22], which provided a reliable framework for interpreting the observed data. This rigorous methodology ensured the collection of precise and reproducible measurements to evaluate the material's performance under simulated physiological conditions.

The tensile strength of specimens was determined using a diametral compression test performed with a FORM TEST SEIDNER D 79-40 machine (Germany). A key characteristic of this test is that only a relatively small portion of the specimen's volume experiences peak stress at the point of fracture.

The tensile strength of the specimens was measured using a diametral compression test (FORM TEST SEIDNER D 79-40, Germany). One key characteristic of this test is that only a relatively small portion of the specimen's volume experiences peak stress at fracture.

In its most basic setup, the test involves compressing a right circular cylindrical specimen diametrally between two flat platens. This configuration generates a biaxial stress state within the specimen. Assuming ideal line loading, the vertical plane is subjected to a uniform horizontal tensile stress given by the following equation:

$$\sigma_t = \frac{2P}{\pi d t} \qquad (4$$

Here, σ_t (MPa) represents the maximum tensile stress, P (N) is the applied load at fracture, d (mm) denotes the specimen diameter, and t (mm) indicates the specimen thickness.

The relationship between the measured tensile strength (σ_t) and its equivalent three-point flexural (bending) strength (σ_f) is described using the equation:

$$\sigma_f = 2.7\,\sigma_t \qquad (5$$

This correlation has been validated by references [23] and [24]. It is worth noting that this derived flexural strength is commonly used for comparative purposes, as it remains a standard parameter among leading researchers in this field.

3.   Results and Discussion

The X-ray diffraction spectrum depicted in Fig. 1(a) for the diopside samples sintered at 1300°C for 2 h prior to immersion in an SBF solution illustrates that the various diffraction peaks are characteristic of the diopside phase. These peaks are observed at diffraction angles of 13.73°, 26.71°, 27.70°, 29.89°, 30.40°, 31.00°, 35.05°, 35.58°, 39.31°, 41.17°, 42.34°, 43.00°, 44.41°, 52.09°, 56.71°, 65.80°, and 66.46°. These observed peaks align with the ICDD file number 00-101-1057.

The diopside's crystal structure is identified as monoclinic, with lattice parameters a = 9.7100 Å, b = 8.8900 Å, c = 5.2400 Å, and β = 74.160°. XRD analysis confirms the chemical composition as $CaMgSi_2O_6$. The unit cell volume of the monoclinic diopside is V = 435.15 Å^3. Furthermore, the spectrum reveals that all peaks pertain solely to the diopside structure, with no additional impurity peaks detected. There is also no indication of degradation or secondary phases present. A densification of 96.7% was achieved at 1300°C.

Based on this information, diopside can be effectively synthesized using solid-state sintering of raw materials at relatively high temperatures without requiring acidic catalysts. In comparison, traditional inorganic biomaterials such as calcium phosphate and bioactive glass often exhibit thermal instability at elevated temperatures. As a result, careful temperature control is necessary when processing these ceramics to prevent crystalline phase degradation or unwanted glassy phase crystallization, which could compromise their bioactivity and mechanical properties.
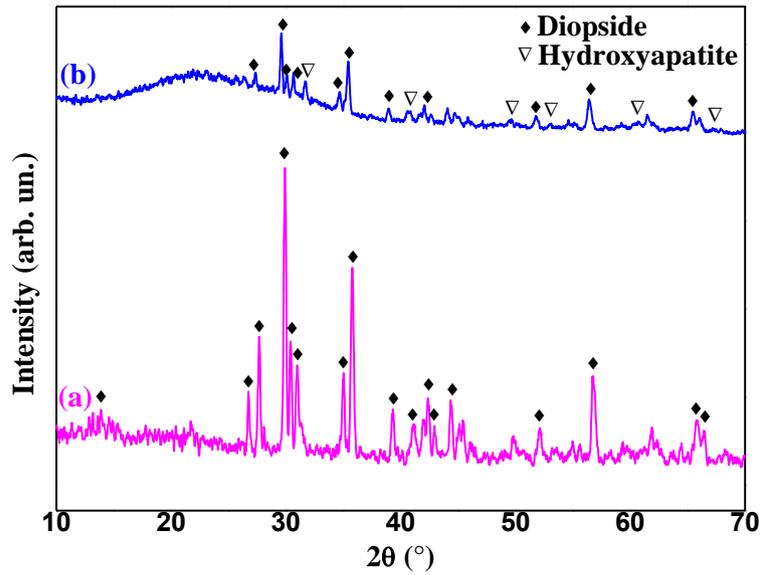
Fig 1. X-ray diffraction pattern of diopside sample sintered at 1300 °C for 2 h (a) before and (b) after immersion in SBF for 14 days.

The Vickers hardness and bending strength of diopside, both before and after immersion in simulated body fluid for up to 30 days, are summarized in Table 1 and illustrated in Fig. 2. The results show that the bending strength and Vickers hardness reach values of 167 ± 8 MPa and 4.7 ± 0.1 GPa, respectively. Notably, no significant changes were observed in the mechanical properties of diopside after the 30-day submersion in SBF. For comparison, commercially available glass-ceramic biomaterials such as CERABONE (apatite-wollastonite glass-ceramic) and BIOVERT (mica-apatite glass-ceramic) display a comparable bending strength, with an average value of 180 MPa [25]. It is significantly higher than the bending strength of hydroxyapatite enhanced with the addition of 0.5% P_2O_5, which reached approximately 57 MPa [26].

Mechanical testing further revealed that the microhardness of diopside decreased from the initial 4.7 GPa to 4 GPa within the first two days of immersion and continued to drop to 3.2 GPa by day 14, where it stabilized (Fig. 2(a)). Remarkably, this stabilized microhardness is similar to that of tooth enamel (approximately 4 GPa) [27] and significantly higher than hydroxyapatite synthesized at 1300°C (0.63 GPa) [28] and higher than the hydroxyapatite prepared with the addition of 5% P_2O_5 (2.1 GPa) [26]. Considering its favorable mechanical properties, which align closely with those of established bioceramics, diopside presents itself as a strong potential candidate for replacing traditional bioceramics in load-bearing applications.

Table 1. Mechanical properties of diopside before and after soaking in SBF for up to 30 days.

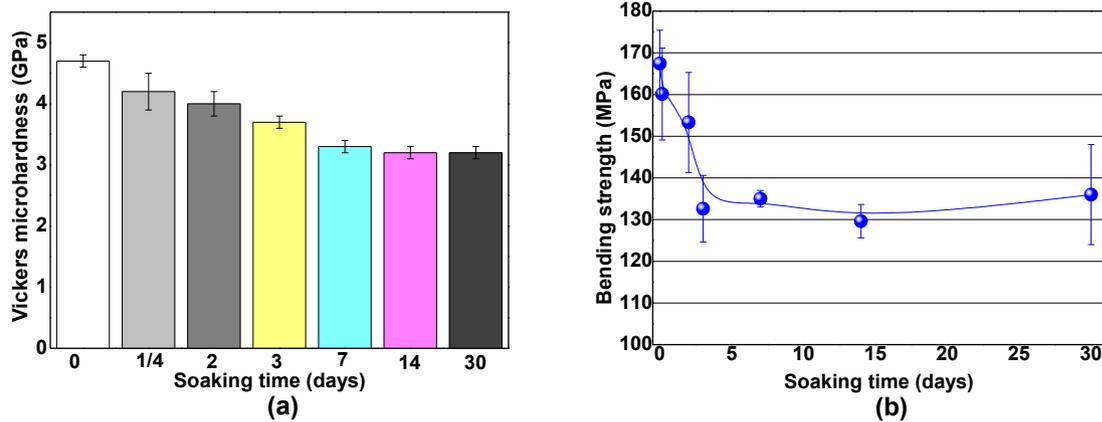| Submerging period (d)	Hardness (GPa)	Bending strength (MPa)
0	4.7±0.1	167.4±8
1/4	4.2±0.3	160.1±11
2	4±0.2	153.3±12
3	3.7±0.1	132.6±8
7	3.3±0.1	135.0±2
14	3.2±0.1	129.6±4
30	3.2±0.1	136.0±12

Fig 2. (a) Vickers microhardness and (b) Bending strength of the diopside discs before and after 6 h and 2, 3, 7, 14, and 30 days of soaking in SBF.

The study of changes in relative weight and porosity ratio represents a simple yet crucial method to highlight the alterations occurring in a material after immersion in simulated body fluid. The results of relative weight change measurements as a function of immersion time are presented in Table 2 and illustrated in Fig. 3. It is evident from Fig. 3(a) that the highest percentage of weight loss occurred after six hours of immersion, amounting to 0.93%. This can be attributed to the dissolution of diopside, leading to the release of calcium and magnesium ions. Over time, the weight loss percentage gradually decreased, reaching a minimal level by 14 days of immersion. This reduction is likely due to the continued dissolution of the material alongside the onset of deposition processes. Interestingly, the sample immersed for 30 days exhibited an increase in relative weight compared to its initial state. This is likely due to a significant structural transformation resulting from the growth of an apatite layer, which contributed additional weight to the material. In comparison with previous studies by Yoshimi et al. [17], it was found that their reported weight loss for hydroxyapatite samples immersed for two days was approximately 1.1%, a value higher than the 0.79% obtained in this study under the same immersion period (as shown in Table 2). Furthermore, when the immersion period for the current study's samples was extended to 14 days, the weight loss percentage dropped significantly to 0.13% (as per Table 2), which is substantially lower compared to Yoshimi reported value of 1.13% [17]. Meanwhile, the decomposition of wollastonite reached 3.8% in the SBF solution after being immersed for 14 days as well [29]. This means that the degradation rate of diopside is significantly slower in the SBF solution. This can be attributed to the presence of magnesium ions in the crystal lattice of diopside, which may inhibit the premature degradation of calcium/phosphate phases and subsequently reduce the degradation rate of diopside in simulated body fluids. According to previous studies, it has been shown that the bond energy of Mg-O is higher than that of Ca-O [30], which contributes to greater stability of the crystal structure and prevents the rapid decomposition of diopside. Compared to the previous report by Srinath et al. [31], which indicated that the weight loss of diopside synthesized from eggshells and RHA increased linearly with immersion time, reaching 1.5% after 14 days of soaking, the current study demonstrates that diopside ceramics derived from dolomite exhibit an extremely low degradation rate in SBF. Therefore, diopside ceramics with lower dissolution rates compared to traditional bioceramics could be a promising candidate for load-bearing bone applications.

To gain a deeper understanding of the impact of simulated body fluid on diopside samples, the relative density was calculated using Eq. 3, and porosity was then derived using Eq. 2 for these samples, both before and after immersion. The results obtained are recorded in Table 2 and illustrated in Fig. 3(b). It was observed that the porosity of samples immersed for six hours showed a significant increase, rising from 3.8% before immersion to 7.67% (as shown in Table 2). This increase is likely attributed to the dissolution process. In contrast, samples immersed for periods ranging from 2 to 30 days exhibited a marked decrease in porosity, reaching 3.54% for the sample immersed for 30 days. This reduction can be attributed to pore filling due to apatite nucleation and growth, which induces fundamental structural changes. Referring to the study by Yoshimi et al. [17], the porosity of hydroxyapatite samples increased by approximately 2% within the immersion period of 2 to 14 days. This value is comparable to the results achieved in the current study, where porosity rose by 2.21% over the same immersion duration.

Table 2. Relative weight and porosity ratio of diopside before and after soaking in SBF for up to 30 days.

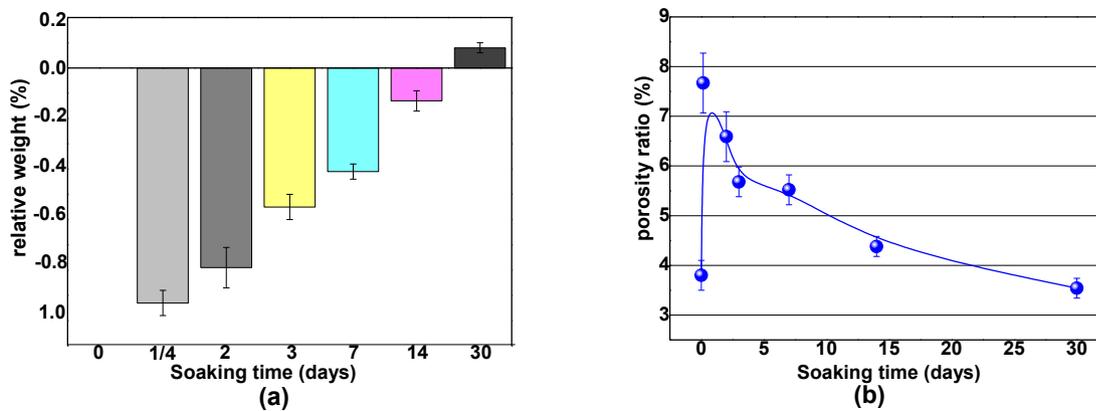| Submerging period (d)	relative weight (%)	porosity ratio (%)
0	/	3.80±0.3
1/4	-0.93±0.05	7.67±0.6
2	-0.79±0.08	6.59±0.5
3	-0.55±0.05	5.68±0.3
7	-0.41±0.03	5.52±0.3
14	-0.13±0.04	4.38±0.2
30	+0.08±0.02	3.54±0.2

Fig 3. (a) Relative weight and (b) porosity ratio of the diopside discs before and after 6 h and 2, 3, 7, 14, and 30 days of soaking in SBF.

The analysis results obtained through X-ray diffraction of diopside samples immersed for 14 days in simulated body fluid, as shown in Fig. 1(b), reveal a decrease in the intensity of diffraction peaks corresponding to the diopside compound. This decrease is particularly evident at diffraction angles 26.71° and 30.40°, supporting the hypothesis of partial dissolution suggested by the analysis of the changes in relative weight, porosity, and mechanical properties of the diopside samples under the influence of the simulated body fluid.

Additionally, the diffraction spectrum exhibits an amorphous phase attributed to the growth of a calcium phosphate layer. This layer forms as a result of the deposition process, involving the interaction of calcium and phosphate with the silica layer on the interface of the diopside samples [32, 33]. In this initial phase, an ion exchange process occurs between Ca^{2+} and Mg^{2+} ions on the diopside surface and hydrogen ions (H^+ or H_3O^+) present in the simulated body fluid [20]. This exchange leads to a reduction in calcium and magnesium content on the diopside surface, resulting in the collapse of the silica network. Consequently, there is a decrease in relative weight and mechanical properties, accompanied by an increase in porosity to form silanol groups with a negatively charged surface. This surface charge plays a critical role in the formation of the hydroxyapatite layer on the soaked surface. Subsequently, a silica-rich layer is produced on the surface due to the repolymerization of silanol groups. These silica-rich layers serve as suitable sites for attracting Ca^{2+} and PO_4^{3-} ions. The attracted ions can form calcium phosphate films (CaO-P_2O_5), and over time, the accumulation of these ions results in the formation of a calcium phosphate layer on the soaked surface. This process ultimately leads to the development of an amorphous, apatite layer. Moreover, new diffraction peaks were observed at angles 31.68°, 40.79°, 49.59°, 53.17°, 60.70°, and 67.29, which correspond to hydroxyapatite. These peaks align with data card 00-230-0273, confirming the crystallization of an apatite layer on the surface of the diopside samples. The hydroxyapatite crystal structure is classified as hexagonal, with lattice parameters a = 9.4210 Å and c = 6.8800 Å. XRD analysis verifies its chemical composition as $Ca_{10}(PO_4)_6(OH)_2$. The volume of the hexagonal unit cell was determined to be V = 528.83 Å³.

These findings highlight the bioreactivity of diopside as a material capable of forming an apatite layer similar to bone tissue. This observation aligns with previous results reported by Srinath et al [31] and Sobhani [34].

Conclusion

This study examines the relationship between immersion time and the mechanical properties and degradation behavior of diopside prepared at 1300°C from local dolomite in simulated body fluid. Hardness declined slightly from 4.7 GPa to 3.2 GPa, and bending strength fell from 167.4 GPa to 129 GPa after 14 days in SBF. Beyond this point, hardness stabilized, while bending strength rose to 136 GPa. These values are comparable to established bioceramics. The degradation of diopside was also assessed by monitoring changes in relative weight and porosity. After six hours of immersion, relative weight dropped by 0.93%, and porosity increased to 7.67% due to dissolution. Over 30 days, relative weight increased by 0.08%, and porosity decreased to 3.54%, driven by apatite growth and pore crystallization. These results indicate that diopside decomposes slowly. Bioactivity was evaluated through X-ray diffraction after 14 days in SBF, confirming the formation and crystallization of an apatite layer on the surface following initial dissolution. The results indicate that diopside synthesized from local dolomite using the solid-state method shows promising potential for bone regeneration applications. This is attributed to its remarkable mechanical properties combined with resistance to degradation in SBF environments. Ongoing research is focused on further investigations through cultured cells to validate its effectiveness.

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflict of Interest

The authors declare that they have no conflict of interest.

Data Availability Statement

Not applicable.

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