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Evaluation of mechanical properties of hydroxyapatite-silver nanocomposite for dental engineering applications

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Abstract

Hydroxyapatite bioceramic has low bactericidal and mechanical properties, especially fracture toughness, which may limit its common use in medicine. This study aims to prepare composites of nanoparticles from hydroxyapatite and silver nanoparticles as reinforcements. The composites were fabricated at several concentrations of silver (3, 6, 9, and 12 vol%) by the powder metallurgy method, with specimens compacted under uniaxial pressing and a conventional powder sintering process. The mechanical properties of composites were investigated in this work, such as the compression strength, Vickers hardness, and fracture toughness; also, the structural properties of the material were examined by X-ray diffraction (XRD) technique. The results indicated that adding Ag led to improved fracture toughness, compression strength, and Vickers hardness of the HA/Ag composites, and reached the maximum values at 9vol%, then the values dropped. The better ratio for adding Ag was 9%; after this ratio, the properties of the prepared composites will decrease in their values.

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Keywords: Hydroxyapatite-silver nanocomposites, Fracture toughness, Compression strength, Vickers hardness, Dental applications

1. Introduction

Calcium hydroxyapatite is the major constituent of teeth and bones, so it is widely used in composites for medical applications, such as in orthopedics, in the repair and implants of bone, or as substitute materials in dentistry [1, 2]. In dentistry, hydroxyapatite, a naturally occurring component of tooth enamel and dentin, has therefore been extensively used in scaffolds, implant materials, and coating materials for dental implants [3]. HAp has high importance for both orthopedic applications and dental due to its interesting biological properties such as biocompatibility, bioactivity, non-toxicity, and ability to make direct chemical bonding with natural bone tissue [4].

However, there are some problems related to the manufacture of hydroxyapatite, such as the presence of cracks and high brittleness, which may limit its applications [5]. Many researchers work on the mechanical properties of hydroxyapatite aimed at reducing its brittleness and improving its properties to increase its use as

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a biomaterial. Silver particles are also broadly used as active antibacterial agents due to their ability to reduce the populations of bacteria in dental composites and help in maintaining oral health; therefore, they are mostly used in bone prostheses in implantology, endodontics, dental prostheses, and restorative dentistry [6].

Recently, the combination of HAp with silver particles as reinforcements can improve its properties due to the ductility of silver and produce acceptable composite materials used as antibacterial materials [7]. A number of research studies have studied the preparation of composite materials from HAp with Ag nanoparticles and focused on enhancing the biological and mechanical properties of these biocomposites [8, 9]. The present work aimed to overcome the problems of the two materials and provide better mechanical properties of HAp by preparing an HAp/Ag nanocomposite to evaluate their mechanical strength, that were characterized by compression strength, Vickers hardness, and fracture toughness.

2. Materials and methods

2.1. Preparation of hydroxyapatite - silver composite

The commercial hydroxyapatite and silver powders were used in this work, from Hongwu International Group Ltd, China, in particle sizes (75 and 25nm), respectively, are mixed with a binder material of 2%wt polyvinyl alcohol (PVA) in different quantities of silver nanoparticles 3, 6, 9, and 12 vol% Ag, as shown in Table 1. The mixture was obtained by a ball mill mixer with alumina balls for 5 hours in the presence of water to reduce friction and improve the grinding efficiency. The mixture was dried in an oven to remove water at 70 Co for 48 h, then sieved to be ready to compact into ceramic specimens.

Table 1. Concentrations of materials

Sample ID	Composition (vol. %)	
	Ag	HA
A	0	100
B	3	97
C	6	94
D	9	91
E	12	88

The compacting done by the uniaxial semi-dry pressing technique using a rectangular stainless steel mold with appropriate pressure was 150 MPa, and sintered at a temperature of 1250^{o}C for a 2-hour soaking time with a heating rate of 10^{o}C/min, then cooled inside the furnace.

2.2. X-ray diffraction

XRD technique offers an identical pattern of diffraction for all phases present in pure or composite material. X-ray diffractometer (XRD 6000, Shimadzo, with Cukα radiation (λ = 1.5405 Å), with an applied power and scanning speed of 40 kv/30 mA.

2.3. Fracture toughness (K_{Ic})

The samples of HA and HA/Ag composite with dimensions 3×4×50 mm were prepared for the fracture toughness test, which was measured with the single-edge notched beam method and according to ASTM C1421. Figure 1 shows the prepared sample.

Figure 1. The fracture toughness specimens

2.4. Compression strength

Compression strength was determined using an electronic universal testing machine (WDW-5E) according to ASTM standard C-1424; the test speed was 0.5 mm/min, using cylinder samples in D=10mm, H=20mm dimensions, as shown in Figure 2.

Figure 2. The compression strength test specimen

2.5. Vickers hardness

The test is conducted according to ASTM C1327-90, by a micro Vickers hardness tester (TH-717) using a 1 kg load and a soaking period of 15 sec. Three disc samples (D=13mm, H=5mm) are prepared in a steel mold and accurately finished to test the hardness under the same conditions; then the average value for the three samples is taken.

3. Results and discussion

3.1. X-ray diffraction

Figure 3 displays the X-ray diffraction pattern of pure hydroxyapatite specimen A. The figure shows that all peaks in the pattern match the hydroxyapatite phase and appear minor phase of β-tricalcium phosphate, identical to JCPDS card NO. 09-0169 [10], indicating that there is no decomposition of hydroxyapatite after the sintering process.

Figure 3 shows the XRD of HA-Ag composites at 3, 6, 9, and 12 vol%. As is clear from the pattern, the silver did not interrupt the apatite structure. Ag/HA composite samples do not lose their crystallinity after the addition of Ag [11]. As the Ag volume fraction was increased from 3 to 12 vol%, the Ag peaks at 38.190o and 44.374o regularly increased, showing that Ag particles were incorporated in the hydroxyapatite phase [12].

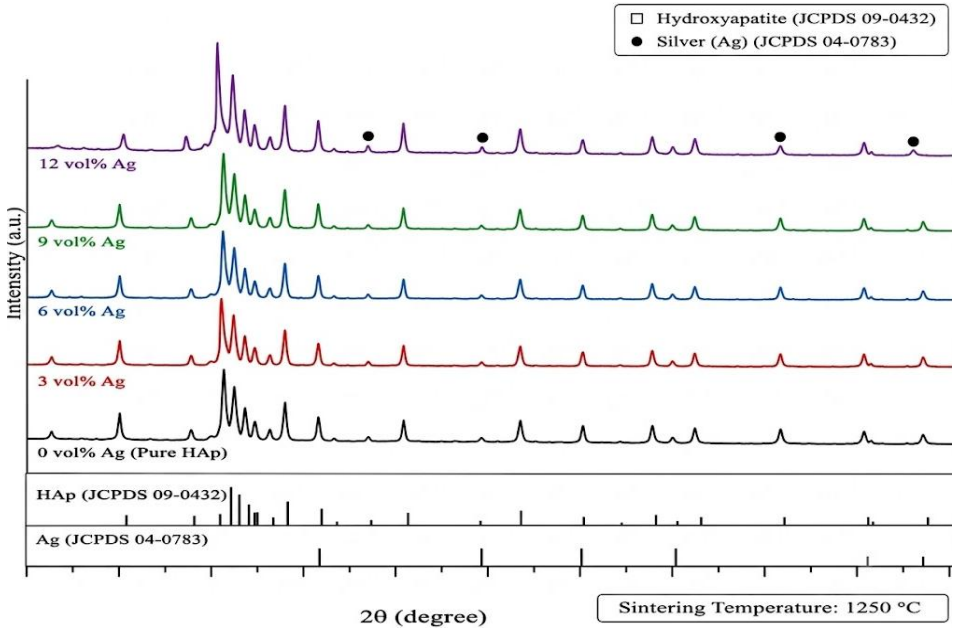

Figure 3. XRD of HA and HA-Ag composite specimens

3.2. Fracture toughness

The result of the fracture toughness test is shown in Figure 4. This result was calculated according to ASTM C 1421 [13]. The addition of silver nanoparticles has a positive effect on the fracture toughness of hydroxyapatite powder, as shown in Figure 4. Sample A (100% hydroxyapatite) exhibited very low toughness (0.89MPa), while with increasing silver content, at 9% Ag particles (Sample D) displayed the highest (1.6MPa), reflecting a 45 % improvement.

Adding Ag to hydroxyapatite, which is very brittle, has constantly improved the toughness due to enhanced densification with better stress distribution. This is attributed to silver particles, which deform plastically and deflect the progression of the crack [14]. The important factor affecting the fracture toughness of composite materials is crack deflection at the silver particle site, where the crack is generally deflected after it meets silver particles [15]. The fracture toughness values display a reduction with increasing silver contents up to 12% vol at sample E; this is due to filler distribution. The scientific fact indicates that strength does not always increase with an increase in filler concentration; this is due to the reason that the high concentration of filler leads to more defects that deteriorate the material [16].

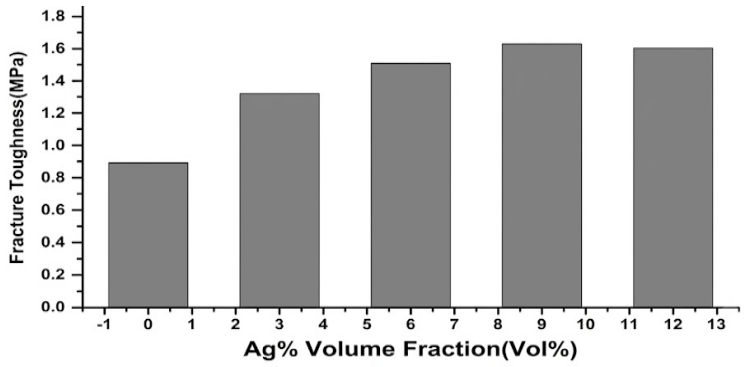

Figure 4. Relation between fracture toughness and Ag volume fraction

3.3. Compression strength

The result of compressive strength for HA and HA -Ag composite is shown in Figure 5, which is calculated according to ASTM standard C-773. Compressive strength is a hidden signal of the adhesion between particles; this property is a quantitative term of a compact amount, which is related to the touched surfaces among the particles of HAp and silver [17]. As seen from Figure 5, the presence of Ag particles has improved the compression strength of the hydroxyapatite particles.

This behavior occurs because the distribution of silver at this concentration can occur without agglomeration and achieve a more homogeneous distribution [18]. Also, it can be seen that the compressive strength values of sample E show a reduction with the increasing silver contents up to 12% vol. This is due to the high risk of agglomeration of silver particles and their amalgamations owing to the diminished average free path among the particles at such high silver concentrations [17].

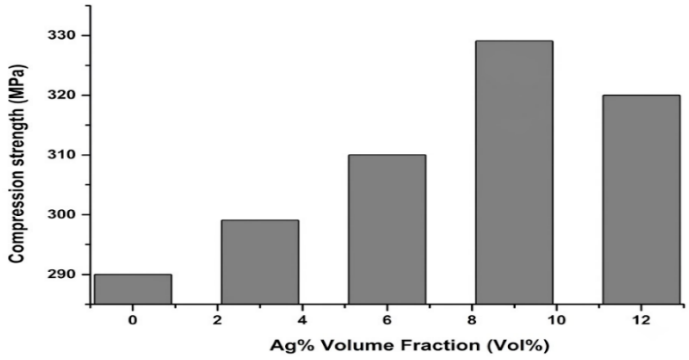

Figure 5. Relation between compression strength and Ag volume fraction

3.4. Vickers hardness

Their results are calculated according to ASTM standard C1327-90. Figure 6 shows that the hardness values were enhanced by adding silver particles, which might be attributed to the low hardness value of hydroxyapatite particles in comparison with Ag nanoparticles [19]. The addition of the hard Ag particles improved the material surface due to good bonding of composite components, causing a high resistance to indentations [20].

Therefore, the prepared composite at 3% concentration of Ag achieved a maximum level of interfacial stress due to enhanced mechanical interlocking caused by silver particles. The achieved result showed that the improvement in Vickers' hardness of the prepared dental composites was compared with the current hardness values [21, 22]. This may be due to either an effect of an appropriate amount of the fillers or due to a good sintering temperature of the composite [23].

An additional increase in the particle concentration (9% and 12%) reduced the hardness of the materials because of the formation of cavities and voids, which worked as nucleation sites and helped the internal crack tips of the filler particles. Also, at a high amount of Ag particles, agglomeration will be found, so the interaction will be reduced, causing a decrease in the mechanical properties of the composites.

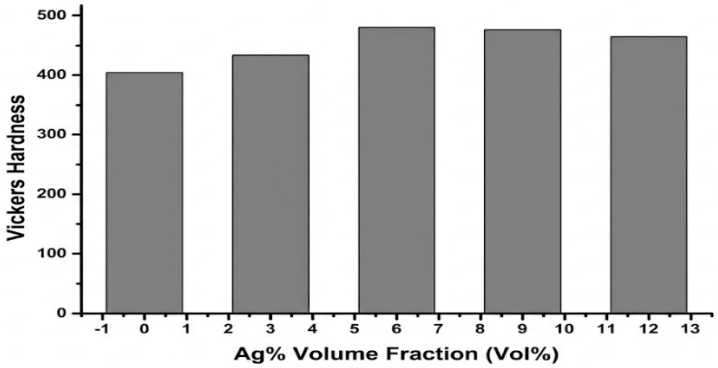

Figure 6. Relation between Vickers hardness and Ag volume fraction

4. Conclusion

HAp-Ag composite has been successfully synthesized by the powder metallurgy method, with different Ag volume fractions (3, 6, 9, and 12% vol), providing an advanced material used in the field of bioapplications.

In this study, the mechanical properties of HA-Ag composites (compression strength, Vickers hardness, and fracture toughness) have been investigated. This shows that the incorporation of silver particles leads to an enhancement in the mechanical behavior of these materials. Fracture toughness increased by about 45% of the HA toughness at a 9% Ag composite, while compressive strength improved approximately 13% and the Vickers hardness by about 19% of the properties of HA at a 6% Ag addition ratio. Moreover, it was further shown that the enhancements of properties do not continue in the same manner and will drop at a high level of addition of silver particles. For any composite material, there is an ideal ratio of reinforcement fillers at which the behavior of the composite will be perfect.

5. Future works

Further studies need to be conducted to consider other factors for evaluating mechanical features of hydroxyapatite-silver nanocomposite for dental engineering applications, like costs associated with electricity, gas, and liquids, financing effects, and transition risks. In addition, artificial intelligence [24] and machine learning to increase the efficiency of evaluation [25, 26], optimization techniques [27, 28], cloud computing technology for saving the physical and chemical data parameters based on specific portal [29], Internet of Thing for automation choice [30], and microwave devices as sensors [31, 32] can support the objective of this study as future trends.

Declaration of competing interest

The authors declare that they have no known financial or non-financial competing interests in any material discussed in this paper.

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Author contribution

Alaa Sabeh Taeh: Conceptualization, methodology design, architectural analysis, data collection, and drafting of the manuscript. Riyam I Jaddan, Israa F. Ghazi: Theoretical framework development, heritage identity analysis, data interpretation, and critical revision of the manuscript. All authors contributed equally to the discussion of results, refinement of arguments, and final approval of the version to be published.

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