Effects of Commercial Inert Glass (CIG) Addition on Mechanical and Microstructural Properties of Chicken Hydroxyapatite (CHA)

Nermin Demirkol¹,a, Ahmet Yavuz Oral²,b, Faik Nuzhet Oktar³,c and Eyup Sabri Kayali⁴,d

¹Ceramic, Glass&Tile Dept., Vocational School of Degirmendere Ali Ozbay, Kocaeli University, Kocaeli, Turkey
²Department of Material Science and Engineering, Gebze Institute of Technology, P.O.Box 141, (41400) Gebze, Kocaeli, Turkey
³Bioengineering Dept., Faculty of Engineering, Marmara University, Istanbul, Turkey
⁴Metallurgical and Materials Engineering Dept., Istanbul Technical University, Istanbul, Turkey

a nermin.demirkol@kocaeli.edu.tr (corresponding author)
b aoral@gyte.edu.tr, c foktar@marmara.edu.tr
d kayali@itu.edu.tr

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Abstract

Hydroxyapatite (HA) can be obtained by both synthetic and natural methods. The synthetic hydroxyapatite is the most commonly used type of HA and it is highly reliable. However fabrication of synthetic hydroxyapatite is complex and expensive. The production of natural hydroxyapatite is easy and inexpensive. In spite of being a biocompatible and bioactive material, hydroxyapatite has a limited usage as an implant material because of its weak mechanical properties. For this reason, HA based composites are required to supply improvement of strength and toughness of the implant materials without losing biocompatibility. In this study, HA composites were synthesized by using natural chicken hydroxyapatite (CHA) reinforced with 5 and 10wt.% commercial inert glass (CIG) powders. Then their physical, mechanical, microstructural properties were characterized. Finally, the most suitable CIG containing CHA composite for orthopedical applications was determined.

Introduction

Biomaterials, which are used as an improvement or replacement of a living tissue, are the biocompatible, reliable and effective materials. Nowadays, the application areas and importance of biomaterials have been gradually increased [1]. Hydroxyapatite (HA; Ca₁₀(PO₄)₆(OH)₂) is one of the best known ceramic biomaterials which is regarded as one of the strongest candidate materials. It easily bonds chemically to bone. The biocompatibility of HA is mainly attributed to the compositional and structural similarities to the bone and tooth minerals [1,2].

It is well known that HA has poor mechanical properties. It has a brittle character. There are various approaches to minimize this drawback [3]. HA material must be reinforced with other ceramics or metals to form a durable and more load resistable composites [4]. There are some recent studies about the production of HA composites reinforced with different additives. Oktar and Goller [5] had produced human derived dentine HA (DHA) composite with glass addition. They observed that sodium free glasses could be better conciliated with the HA structure at high temperatures. Goller et al. [6] had reinforced DHA with 5 and 10 wt. % bioglass (45S5). Lee et al. [1] obtained completely densified body with the addition of small amounts of the glass (up to 5wt. %) to the fluoridated apatite (FHA), after sintering at 1250ºC. The addition of the glass to the FHA also significantly improved the viability and functional activity of osteoblastic cells. Demirkol et. al. [4] had used
commercial inert glass (CIG) for reinforcement of HA composites and determined that a very remarkable glassy phase formed with 10 wt.% CIG addition at 1300°C sintering temperature which decreased strength and ductility.

HA ceramics are produced in the first hand from reagent grade chemicals with complex, tedious and time consuming methods. Natural HAs are very popular because they are containing some trace elements which are playing an important role on osseointegration. Calcination method is one of the economic way to produce HA from various bone wastes (bovine derived HA-BHA [7], sheep derived HA-SHA [8,9], fish bones [10], enamel derived HA [11]) which are collected from clinics. Chicken bones are usually wasted materials. It will be a good idea to use them as a bone source. HA production from these bones seems very promising.

The aim of the present study is to produce chicken derived HA composite by using CIG and study their mechanical and microstructural properties.

Materials and Methods
The CHA powder used in this study was obtained from calcinated chicken bones, according a method described earlier studies [4,8]. Window glass is used to produce commercial inert glass (CIG) which obtained with the average particle size of 68 µm. CHA powder was ball milled with (separately) 5 and 10 wt% CIG powder with ethanol for 4 h. The samples were prepared according to a British Standard for compression tests (BS 7253) [12]. The green samples were sintered at different temperatures between 1000°C and 1300°C for 4 h. Density, Vickers microhardness and compression strength were measured. SEM and EDS analysis were done after etching with 3 vol% HF solution for 1 minute. The crystalline structure of the samples was determined with X-ray diffraction analysis. The compression tests were done with an universal testing machine (Shimadzu) at the crosshead speed of 3 mm/min. Microhardness values were determined under 200 g load. Scanning Electron Microscope (Hitachi TM-1000) was used for microstructural examinations and XRD analysis were conducted on a Bruker D8-Advanced X-ray diffractometer.

Results and Discussion
XRD diagrams of CHA-CIG composites sintered at 1000°C and 1300°C are given in Fig.1. CHA-5 wt.% CIG and CHA-10 wt.% CIG composites sintered at 1000°C includes hydroxyapatite (HA) and calcium magnesium silicate (CMS) phases. CHA-CIG composites sintered at 1300°C contain hydroxyapatite (HA), calcium phosphate (CP), calcium magnesium silicate (CMS), whitlockite (W) and sodium calcium silicate (SCS) phases as seen in Fig.1.

Fig.2 shows the microstructures of CHA-CIG composites sintered at 1000°C and 1300°C. Similar microstructures were observed in composites containing 5 and 10 wt.% CIG sintered at 1000°C (Fig.2a and 2c). However, the composites sintered at 1300°C showed evident sodium calcium silicate (SCS) phase (Fig.2b and 2d).

![Fig.1. XRD diagrams of 5 and 10 wt% CIG added CSHA composites, sintered at 1000°C and 1300 °C.](image1)

![Fig.2. Microstructures of CHA-CIG composites sintered at 1000°C and 1300°C.](image2)
Fig. 2. Microstructures of CHA-CIG (5 and 10 wt%) composites sintered at different temperatures.  
(a) CHA-5 wt% CIG - 1000°C, (b) CHA-5 wt% CIG - 1300°C, (c) CHA-10 wt% CIG - 1000°C, (d)  
CHA-10 wt% CIG - 1300°C.

Fig.3 shows the microstructure and EDS analysis of SCS phase in CHA-10 wt.% CIG composite  
sintered at 1300°C. This phase contains high amount of Si which is the basic element of glassy  
phase.

In earlier studies, Karacayli et. al. [13] studied the mechanical and microstructural properties of  
CHA. Table 1 shows the experimental results of density, compression strength and Vickers  
microhardness of the CHA sintered at different temperatures.
Table 1. Density, compression strength and Vickers microhardness values of CHA sintered at different temperatures [13].

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>Density (g/cm³)</th>
<th>Compression Strength (MPa)</th>
<th>Vickers Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>2,22</td>
<td>40,2</td>
<td>101,4</td>
</tr>
<tr>
<td>1100</td>
<td>2,38</td>
<td>63,2</td>
<td>171</td>
</tr>
<tr>
<td>1200</td>
<td>2,70</td>
<td>66,9</td>
<td>222,8</td>
</tr>
<tr>
<td>1300</td>
<td>2,83</td>
<td>78,8</td>
<td>360</td>
</tr>
</tbody>
</table>

Density and mechanical properties of CHA increased with increasing sintering temperature and the best properties were obtained with samples sintered at 1300ºC.

Table 2 shows the experimental results of density, compression strength and Vickers microhardness of the CHA-CIG composites sintered at different sintering temperature with different amounts of CIG content. Density, hardness and compression strength increased with increasing sintering temperature except composites sintered at 1300ºC. Decreases in density and mechanical properties of these composites are due to the glassy phase of SCS which formed at this temperature.

Table 2. Density, compression strength and Vickers microhardness values of CHA-CIG composites sintered at different temperatures.

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>Density (g/cm³)</th>
<th>Compression Strength (MPa)</th>
<th>Vickers Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5 wt.%</td>
<td>10 wt.%</td>
<td>5 wt.%</td>
</tr>
<tr>
<td>1000</td>
<td>2,28±0,03</td>
<td>2,25±0,05</td>
<td>48± 5,1</td>
</tr>
<tr>
<td>1100</td>
<td>2,53±0,01</td>
<td>2,45±0,05</td>
<td>71± 4,0</td>
</tr>
<tr>
<td>1200</td>
<td>2,78±0,05</td>
<td>2,72±0,04</td>
<td>76± 3,9</td>
</tr>
<tr>
<td>1300</td>
<td>2,35±0,03</td>
<td>2,06±0,06</td>
<td>41±6,1</td>
</tr>
</tbody>
</table>

Fig.4 shows the comparison of density, hardness and compression strength values of CHA and CHA-CIG composites. The density and mechanical properties of composites decreased with increasing CIG content as seen in Table 2 and Fig.4.
In a recent study, Demirkol [8] had studied mechanical and microstructural properties of sheep hydroxyapatite (SHA)-CIG composites sintered at different sintering temperatures. Table 3 presents the experimental density, compression strength and Vickers microhardness values of SHA-CIG composites sintered at different temperatures. Vickers microhardness increased with increasing sintering temperature at all temperatures. However, density and compression strength increased with increasing sintering temperature except SHA-10 wt.% CIG composite sintered at 1300ºC due to formation of a glassy phase (sodium calcium silicate phase) at this temperature.

Table 3. Density, compression strength and Vickers microhardness values of SHA-CIG composites sintered at different temperatures [8].

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>Density (g/cm³)</th>
<th>Compression Strength (MPa)</th>
<th>Vickers Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5 wt.%</td>
<td>10 wt.%</td>
<td>5 wt.%</td>
</tr>
<tr>
<td>1000</td>
<td>2.19±0.06</td>
<td>2.10±0.07</td>
<td>54±5.9</td>
</tr>
<tr>
<td>1100</td>
<td>2.21±0.04</td>
<td>2.13±0.05</td>
<td>64±6.4</td>
</tr>
<tr>
<td>1200</td>
<td>2.48±0.03</td>
<td>2.30±0.04</td>
<td>68±7.6</td>
</tr>
<tr>
<td>1300</td>
<td>2.59±0.04</td>
<td>2.01±0.05</td>
<td>76±7.2</td>
</tr>
</tbody>
</table>

The highest density and mechanical properties were obtained with SHA-5 wt.% CIG composite sintered at 1300ºC. In this composite, there is not sodium calcium silicate (SCS) phase which is cause to vitrification and dramatic reduction of mechanical properties. The highest compression strength values were obtained with CHA-5 wt.% CIG composite sintered at 1200ºC and SHA-5 wt.% CIG composite sintered at 1300ºC as the same value (76 MPa) for both composites.

Summary
In this study, the microstructural and mechanical properties of chicken HA with CIG addition were examined. The findings of this study are concluded as follows:
1. The mechanical properties of composites decreased with increasing CIG content.
2. The Vickers microhardness values of CHA-CIG composites increase with increasing sintering temperature. However, density and compression strength values of CHA-CIG composites increase with increasing sintering temperature, except CHA-CIG composites sintered at 1300ºC.
3. The highest density and mechanical properties were obtained in CHA-5 wt.% CIG composite sintered at 1200ºC.
4. Sodium calcium silicate (SCS) phase causes to decrease compression strength and density of CHA-CIG composites sintered at 1300ºC.
5. Biocompatibility studies are going on.
References