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Research article

Sintering effect on mechanical properties of composites of bovine hydroxyapatite (BHA) – 5% (Al_2O_3 – TİO_2)

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Abstract

Hydroxyapatite (HA) use in biomedicine one as one of the promising biomaterials. Apatite-based ceramics, derived from fine powder of calcinated bovine-bone (BHA), were successfully reinforced with some materials like titanium, zirconia, lithium oxide, bio inert glass and yttria which contained biocompatible elements, via sintering at different temperatures. In this study, hydroxyapatite (HA) material, obtained from calcinated bovine bone (BHA), was mixed with 5 wt.% Al_2O_3-TiO2. In the Al_2O_3-TiO_2 compound, according to rate of the 3 wt.%, 8, 13, 20 and 40 for TiO_2 has been changed. The pressed samples were sintered at various sintering temperatures between 1000°C and 1300°C. Measurements of compression strength, micro hardness, and density, and SEM observation were performed. Experimental results showed that the samples with 13 wt.% TiO_2 reached a maximum of densification and the highest values of mechanical properties were achieved after sintering at 1300°C.

Keywords: *Bovine hydroxyapatite, TiO_2, Al_2O_3, mechanical properties*

1. Introduction

Hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$, is an inorganic material (HA) and related calcium phosphate ceramics have been widely used as bone fillers and implant materials for many years due to their close resemblance with the mineral phase of natural bone[1]. HA is an important biomedical applications, because HA is biocompatibility and bioactivity agent [2]. HA have taken much attention due to the simultaneous achievements of good mechanical properties and excellent biocompatibility [3].

Besides being bioactive, a proper bone substitute should also be able to guide bone regeneration into the defect. Many in vivo experiments confirm that HA promotes osteoconduction [1-3] and therefore HA has found extensive applications as bone defect filler, tissue engineering scaffolds, eye ball prosthesis, bone graft substitutes and as bioactive coating on metallic implants [4].

The use of HA is limited in load-bearing applications. This can lead to instability and unsatisfactory duration of the implant or scaffold in the presence of body fluids and

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under local loading. Furthermore, the mechanical strength of HA structures is further reduced, considerably, by fatigue loading [5].
The development of biocompatible materials that have improved and ultimately bone-8 like mechanical properties is thus a continuous task in the bio ceramics research field. The results of this paper may provide some insight and scientific data for biomedical applications [6-9].

In this study, BHA-5% (Al_2O_3-TiO_2) ternary mixtures were obtained to be 5 different groups and their mechanical properties investigated.

2. Experimental

2.1. Sample Preparation

The BHA powder used in this study was obtained from calcinated fresh bovine femurs, according a method described early study [10]. Firstly, heads of the fresh bone femurs were cut off and then cleaned to remove visible tissues and substances on the bone's surface. The cleaned bovine bone samples were defatted in boiling water followed with sun drying to remove the organic substances and avoid soot formation in the material during the heating treatment. The obtained shafts were deproteinized with NaOH, washed and then calcinated at 850°C for 4 h in air. The resulting white solid specimens were first ground and crushed with a mortar and pestle to produce between -63 µm to +45 µm powders. High purity fine powders of Al_2O_3 (Merck Company) and TiO_2 (Degussa) were used as doping materials. BHA/Al_2O_3/TiO_2 mixtures were ball milled in a zirconia coated container with zirconia balls and ethanol in Restch PM 100 ball milling device at 150 rpm for 4 h. until fine powder was obtained as shown in Table 1 and well homogenized mixture dried at room temperature for 1 day. Pellets (6 mm diameter, 12 mm height) were prepared by uniaxial cold pressing in hardened steel dies, according to British Standards (No. 7253). Finally, the pellets were sintered at 1000, 1100, 1200 and 1300°C for 2 and 4 h in air at the heating and the cooling rates of 5°C/min.

Table 1
The nominal compositions of the BHA/Al_2O_3/TiO_2 composites

Group	BHA(%)	Al_2O_3(%)	TiO_2(%)
1	95	4.85	0.15
2	95	4.60	0.40
3	95	4.35	0.65
4	95	4.00	1.00
5	95	3.00	2.00

2.2. Mechanical Properties of Composites

The compression strength of the sintered samples was measured with a Devotrans Universal Testing device at 2 mm/min speeds. For the measurements of Vickers hardness, a Shimadzu HV2 micro hardness tester was used (200 g load was applied for 20 s). The apparent density (gr/cm^3) and porosity rates (%) of the samples was measured by the Archimedes method.

3. Results and Discussion

Fig. 1-3 show the micro hardness, compression strength and density values of BHA – Al_2O_3-TiO_2 composites with changing sintering temperatures.

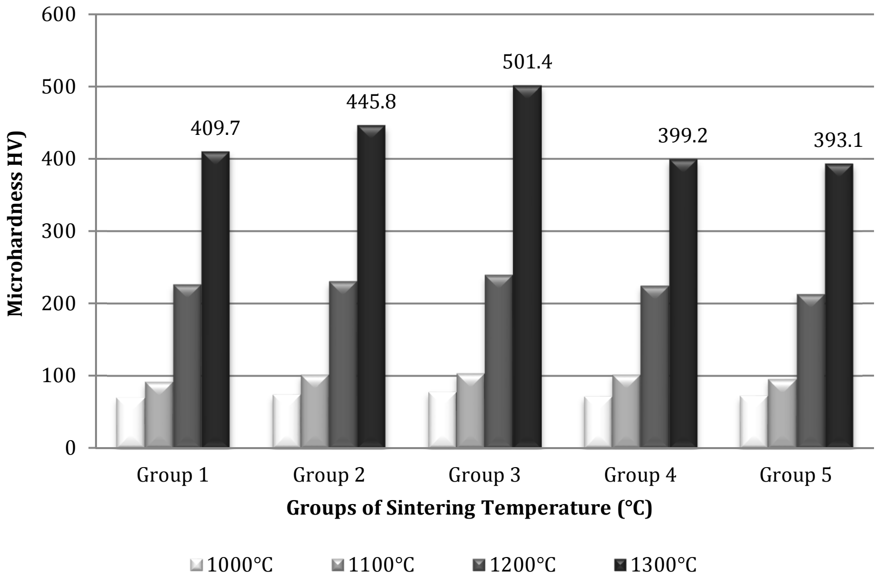

Fig. 1 Influence of sintering temperature on the Vickers micro hardness of groups

The effect of sintering temperature on the micro hardness of the produced samples is shown in Fig. 1. Depend on increasing sintering temperatures (1200 and 1300°C) and rate of Al_2O_3 in ternary mixtures, micro hardness values of all groups increases and optimum results of were obtained for group 3.

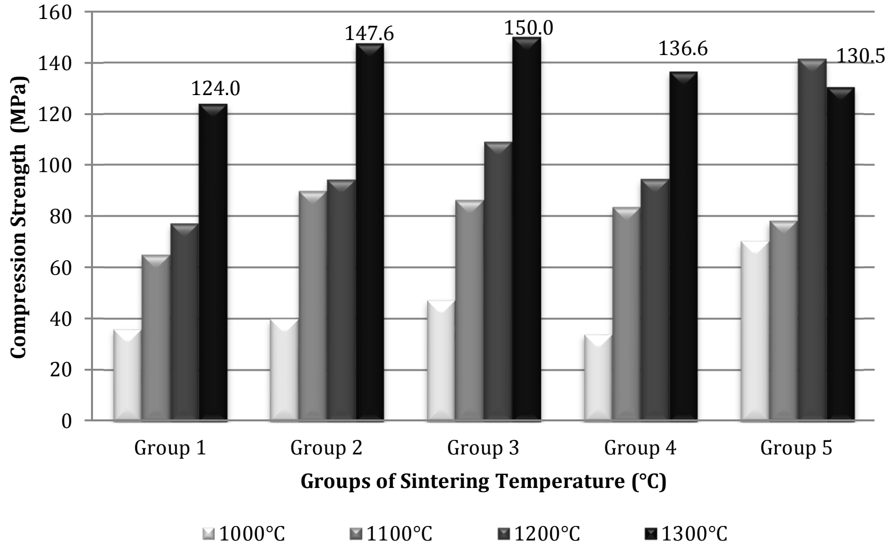

Fig. 2 Influence of sintering temperature on the compression strength of groups.

Depend on both elevated sintering temperatures and rate of Al_2O_3 in composites, compression strength values of five groups' increases and optimum results of were obtained from group 3. We obtained same results on the effect of sintering temperature on compression strength of Bovine Hydroxyapatite based on blends but our compression strength values are much higher than (nearly 200%) early studies [11].

Fig. 3 correlates the mechanical properties with the degree of the densification, which was used at increasing sintering temperatures. This result can be explained by increasing sintering temperature diffusion rate of reinforced materials atoms (Al_2O_3 /TiO_2).

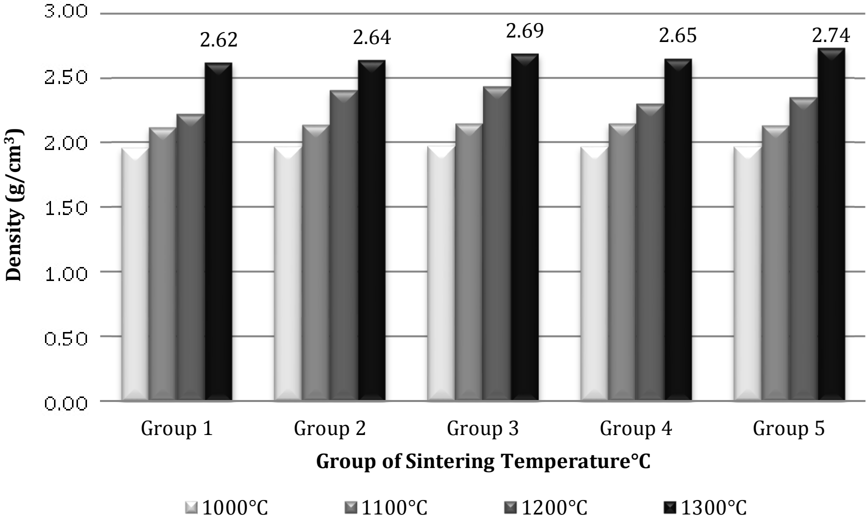

Fig. 3 Influence of sintering temperature on the density (g/cm³) of groups.

4. Conclusion

This work summaries the effect of sintering temperatures on the mechanical properties of Bovine Hydroxyapatite/Al_2O_3/TiO_2 ternary mixtures. The best mechanical properties were obtained when the samples sintered at 1300°C. In all groups highest properties were measured from group 3. Compared binary composites (Bovine Hydroxyapatite/Al_2O_3, Bovine Hydroxyapatite / TiO_2, Bovine Hydroxyapatite / NbO_2, Bovine Hydroxyapatite / MgO etc.) Bovine Hydroxyapatite/ Al_2O_3 /TiO_2 ternary mixtures have higher mechanical properties.

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