Effect of sintering time on microstructural and mechanical properties of HA compacts

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ABSTRACT

The effects of sintering time on mechanical properties of hydroxyapatite, a principal inorganic component of bones and is an implant material for skeletal reconstruction, have been studied. HA obtained from waste material with 5% organic additive incorporation was cold uniaxially pressed at 2500 psi for 3 minutes isothermal hold. The resulting compacts were sintered at 1150^{o}C for different time periods ranging from 0 to 8 hrs. The samples were characterized by measuring compressive strength, porosity, density and micro hardness. Rapid densification took place during heating and cooling due to particle rearrangement. Compressive strength and micro hardness increased with inverse proportion to porosity as sintering time increased although grain growth did occur. It has been seen that porosity is found to play an important role in determining the properties of sintered hydroxyapatite compacts. These findings suggest that an optimum design of porous HA compacts with a variety of porosities and with sufficient strength is feasible by amending the sintering time.

Key Words: Hydroxyapatite compacts, sintering time, mechanical properties

INTRODUCTION

Hydroxyapatite, one of the calcium phosphate ceramics, has been applied due to its excellent biocompatibility with hard tissues and also with skin and muscle tissues, where bone in growth is intended [**1**]. Due to the close resemblance of its chemical and crystallographic properties with those of bone and tooth minerals, HA is of particular interest for bone grafting, augmentation in maxillofacial surgery and in orthopedics as space filling material **[2]**. However the application of HA are limited to non- load bearing implants such as inner ear bones or filler material because of its poor mechanical properties and resultant lack of reliability. The mechanical behavior of calcium phosphate ceramics strongly influences their applications as implant. The production of HA with improved mechanical properties has led to a number of different studies with regards to HA composites with addition of ceramics [**3**], glass [**4**] or polymer [**5**]. It has also induced research with respect to the specific effects of each variable associated with production of synthetic HA from synthesis to sintering.

It is possible to rectify this non load bearing drawback by means of improving the mechanical properties substantially by sintering. G. Gollar [**6**] and F.P. Cox [**7**] discussed the effects of sintering time on mechanical properties of hydroxyapatite and found that the sintering parameters (temperature, time, atmosphere etc.) are very influential on the mechanical and chemical properties of hydroxyapatite.

This study documents the effect of one sintering variable i.e., time on the mechanical properties including compressive strength, porosity, density and microstructure of HA compacts prepared from egg shell powder. The aim of our investigation was to assess the influence of sintering time on microstructure as well as mechanical properties like density, compressive strength, hardness and porosity of HA Compacts.

EXPERIMENTAL WORK

Hydroxyapatite was prepared using egg shells as calcium precursor and phosphoric acid by precipitation method. The prepared HA powder along with 5% methyl cellulose as a binder was uniaxially pressed in a 17mm steel diameter die using a load of 2500 psi for a period of 3 minutes. These HA Compacts were then sintered at 1150^{o}C at the rate of 5^{0}C / min for the time period of 0, 4, 6 and 8 hrs.

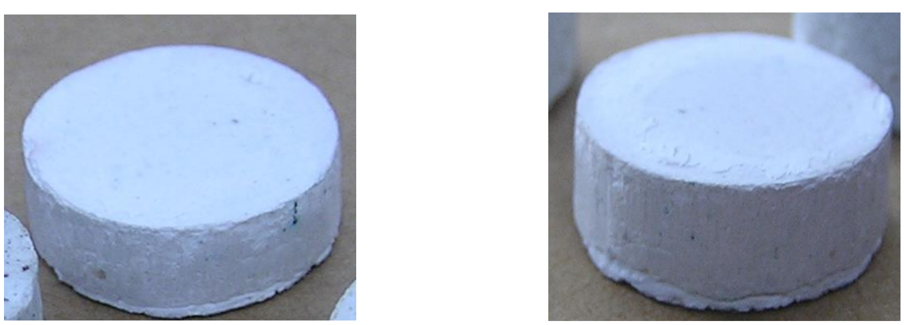


Fig. 1: Hydroxyapatite Compacts after sintering

Micro structural analysis was carried out using image analyzer Leica DM 4000M with a magnification power of 50X to study the densification occurred due to increase in grain size. All samples for microscopic analysis were prepared using alumina paste. Radial shrinkage and green as well as sintered densities were calculated from geometric dimensions. Micro hardness was tested at Vickers's micro hardness tester using a load of 0.5 kg according to ASTM C-1327. Porosity of HA compacts were measured according to ASTM C373-88 for porosity measurement. Compressive strength of the HA compacts was measured using Shimadzu Universal Testing Machine using a load of 5KN and a Ramp/Strain rate of 5mm/min.

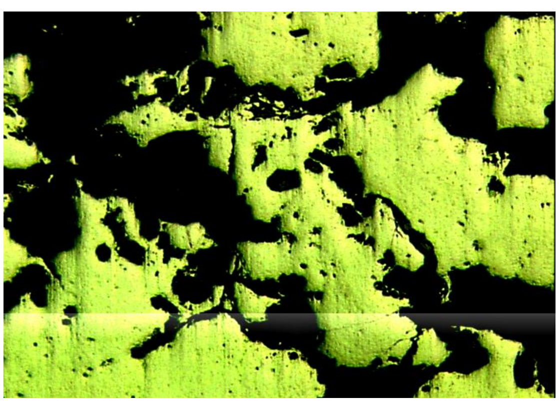
RESULTS AND DISCUSSIONS

MICROSTRUCTURAL ANALYSIS:

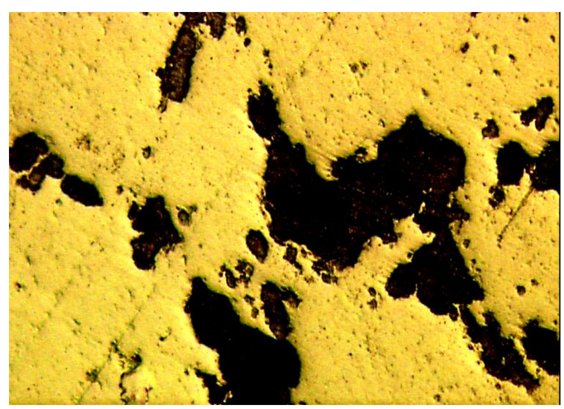
Micro structural analysis reveals a porous structure of hydroxyapatite compacts. The black portion shows the interconnected pores while the colored portion is granular hydroxyapatite phase. The pores developed are interconnected and porous hydroxyapatite with interconnected pores is thought to be a good candidate as scaffold material for bone regeneration and as a synthetic bone substitute material.

From the micro structural analysis of HA compacts sintered at 1150^{o}C for 0, 4, 6 8 hrs respectively (fig 2) it is obvious that densification took place due to increase in grain size with the increasing sintering time and the decrease in porosity can also be observed.

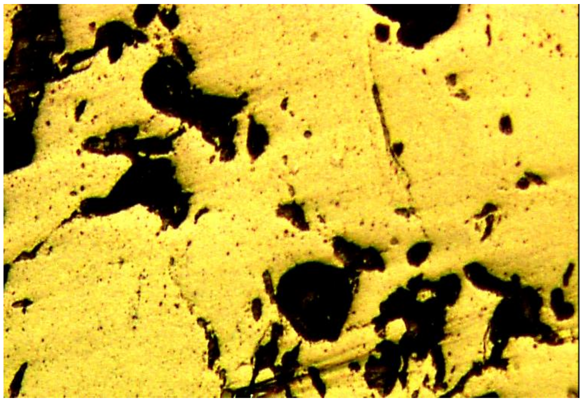
F.P. Cox [**7**] and T.P.Hoepfner [**8**] also discussed that with the increase in sintering time the grain size of hydroxyapatite increased and hence densification took place.



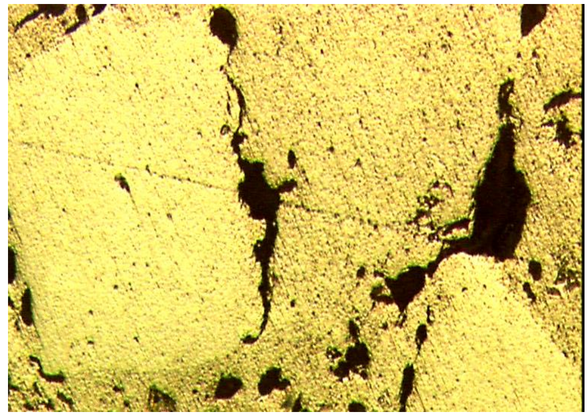
(a) Microstructure of HA compacts sintered for 0 hrs



(b) Microstructure of HA compacts sintered for 4 hrs



(c) Microstructure of HA compacts sintered for 6 hrs



(d) Microstructure of HA Compacts sintered for 8 hrs.

Fig.2: Microstructures of HA Compacts sintered at different temperatures

MECHANICAL PROPERTIES:

Fig. 3 shows that after heating samples to 1150°C and cooling without holding at 1150°C, a radial shrinkage of 4.14% occurred. 6.39% shrinkage occurred after 4 hrs isothermal hold. This increase in radial shrinkage also effects other mechanical properties like density, compressive strength and hardness For longer sintering times greater than 4 hrs, shrinkage rates are extremely slow and would seem to show pore closure resulting in decreased porosity. Porosity and pore size were found to play an important role in determining the properties of sintered hydroxyapatite compacts. **(9).**

Sintering Time (hrs)	**Radial Shrinkage (%age)**
0	4.14
4	6.39
6	6.49
8	6.52

Table 1: Relationship between radial shrinkage and sintering time

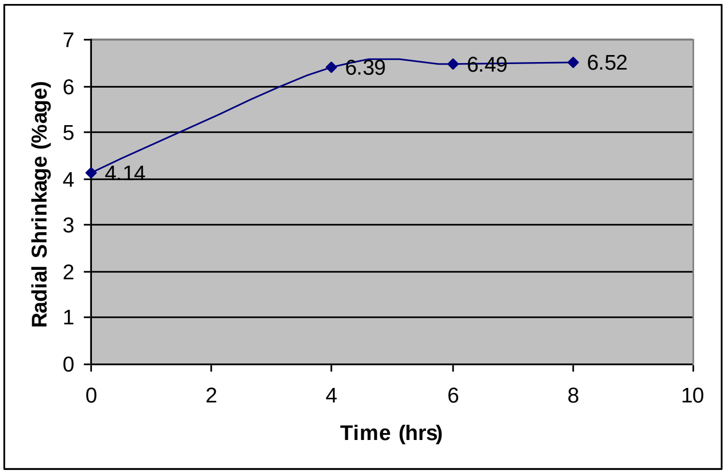


Figure 3: Effect of sintering time on radial shrinkage

Density of HA compacts increases with the increase in sintering time. This effect may be due to increase in radial shrinkage and decrease in porosity. Figure.4 shows the effects of sintering time on porosity and figure 5 shows the relationship between sintering time and density. Table 2 depicts the comparison between porosity and density and shows that with the increase in sintering time porosity decreases and as a result of the decreased porosity increase in density occurs.T.P. Hoepfner and E.D. Case [**8**] also discussed the effects of sintering time on density of HA compacts and concluded that densification of HA particles take place with the increase in sintering time.

Sintering Time (hrs)	**Porosity (%age)**	**Density (g/cm^3)**
0	26.17	2.21
4	11.68	2.22
6	11.54	2.30
8	9.27	2.33

Table2: Relationship between porosity and density with increasing sintering time

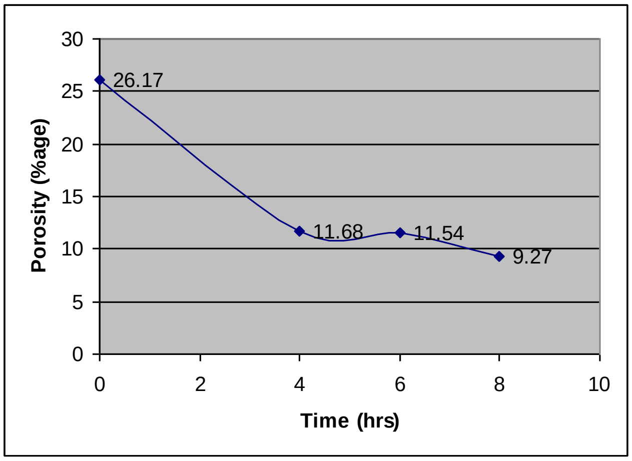


Fig. 4: Effect of sintering time on porosity

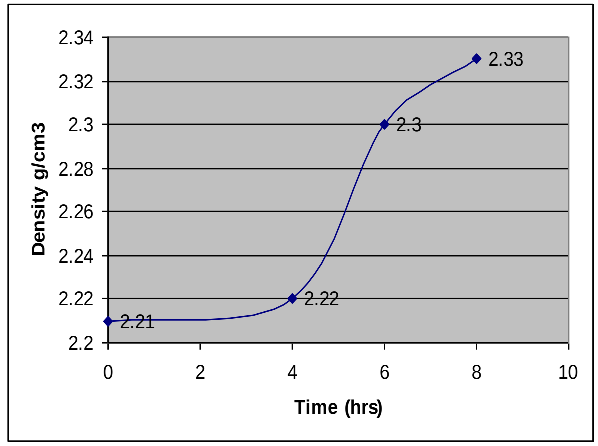


Fig.5: Effect of sintering time on density

Compressive strength and micro hardness of HA compacts increases with the increase in sintering time. Fig.6 and Fig. 7 explains that as compressive strength increases micro hardness values of HA compacts also increases with sintering time. This increase in compressive strength and hardness may also correspond to the decrease in porosity and increase in density.. Compressive strength depends on the total volume of porosity. The porosity dependence of compressive strength of hydroxyapatite was studied by Dean-Mo Liu **[10]** who described that compressive strength varies inversely with porosity. It was also studied that the increasing hardness may be a grain size/mass density effect. [**8**]

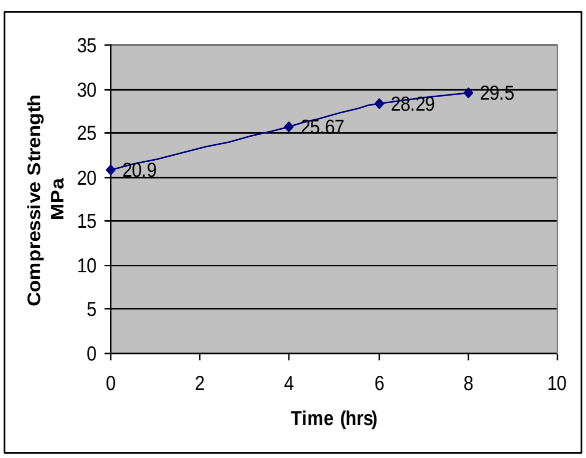


Fig.6: Effect of sintering time on compressive Strength

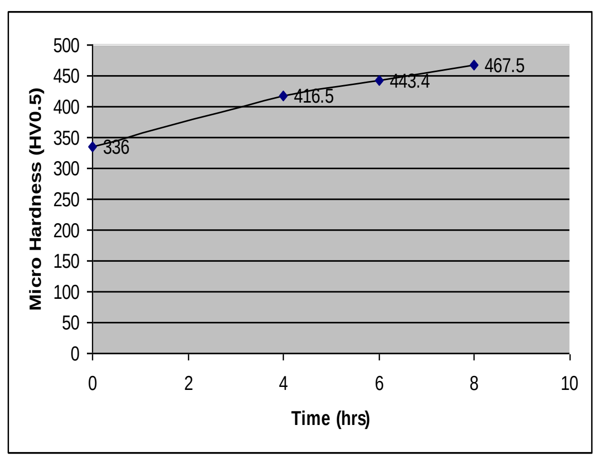


Fig 7: Effect of sintering time on Micro hardness

CONCLUSION

It is concluded that the sintering time exhibited a significant effect on micro structural as well as mechanical properties of HA compacts. These mechanical properties are interlinked as one property varies in comparison of other properties. Sintering time has a major impact on porosity and the variation in porosity is mainly responsible for the variations in other mechanical properties like density, compressive strength, and micro hardness. Densification increases from 66.8% to 77.82% of the theoretical density (3.156 g/cm^3) from isothermal holds from 0 to 8 hrs respectively. Given the increase in densification and associated decrease in porosity, micro hardness and compressive strength values increased to a maximum of 467.6HV0.5 and 36.4 MPa after 8 hrs sintering. These studies gave an idea that to get improved mechanical properties like compressive strength and hardness, sintering time should be increased. However rapid densification take place which once complete may have an adverse effect on compressive strength as well as other mechanical properties. Cracking can also occur with longer sintering holds. From these findings we can conclude that to get an optimum design of porous HA compacts with favorable mechanical properties an optimized sintering time should be suggested.

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