

Effect of Stirring Time and Sintering Temperature on the Properties of Porous Tricalcium Phosphate Using White Particles as Pore-Creating Agent

Ahmad Fadli

Department of Chemical Engineering, Faculty of Engineering, Riau University
Jln. Hr. Subrantas Km 12.5, Pekanbaru, Riau 28293, Indonesia

Bahrudin

Department of Chemical Engineering, Faculty of Engineering, Riau University
Jln. Hr. Subrantas Km 12.5, Pekanbaru, Riau 28293, Indonesia

Fitra Dani

Department of Chemical Engineering, Faculty of Engineering, Riau University
Jln. Hr. Subrantas Km 12.5, Pekanbaru, Riau 28293, Indonesia

Copyright © 2015 Ahmad Fadli et al. This article is distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Abstract

The present work reports a simple process for fabrication of porous tricalcium phosphate (TCP) ceramics from aqueous powder slurries using wheat particles as pore forming agent. The objective of this research is to learn the effect of stirring time and sintering temperature on the physical properties of porous TCP. Wheat particles incorporated in aqueous TCP slurries and stirred for 1, 2 and 3 hours. Subsequently the slurry was dried in oven at 80°C for 24 hours and 120°C for 8 hours. The dried green bodies showed shrinkage in the range 53 -56%. Removal of the pore former followed by sintering at 1000 and 1100°C produced TCP bodies with porosity 59 – 78% and compressive strength 0.3 - 2.5 MPa.

Keywords: Compressive strength, Porosity, Tricalcium Phosphate, Ceramics

Introduction

Porous TCP ceramic is essentially a bioresorbable calcium phosphate. Its tissue biocompatibility is excellent and it may act as a scaffold allowing bone regeneration and ingrowth. Because of its resorbable characteristic, the porous TCP is regarded as an ideal biomaterial for bone substitutes that should degrade by advancing bone growth [1]. In this regard, most important aspects in fabrication of scaffolds are porosity, pore size, pore interconnectivity and mechanical strength. In the terms of microstructure of the mineral phase, the bone can be classified as cortical and cancellous. Cortical bone has porosity in the range 0-30%. Porosity of cancellous bone is 30-90% [2]. Hulbert et al. [3] suggested that the optimum pore size for osteoconduction is 150 μm . Pore interconnectivity allows circulation and exchange of body fluids, ion diffusion, nutritional supply and osteoblast cell penetration [4]. Compressive strength of porous human bones vary between 2-12 MPa for cancellous bone and between 100-230 MPa for cortical bone [5].

A number of technique have been developed to fabricate porous TCP scaffolds, typically including gel casting of foams and replication of a polymer sponge. Gel casting or ceramic foaming technique can be applied to produce ceramic scaffolds with high mechanical strength. The disadvantage of this technique is that it typically results in a structure of poorly interconnected pores and non-uniform pore size distribution. Fabricating porous ceramic via the replication of a polymeric sponge produced open-celled porous ceramics. Porous ceramic prepared via this technique have shown well interconnected pores but has poor mechanical strength for load bearing applications [6].

Lyckfeldt and Ferreira [7] reported a starch consolidation process for fabrication of porous ceramics. In this process, native and chemically modified potato starch granules were incorporated into aqueous ceramic powder suspensions. Characteristics such as higher water absorption and swelling of starch at 60-80°C is used for the gellation of the slurry. The process produces porous ceramics with 30-70% porosities. Wheat is a low cost starchy material which can be used as gelling and pore forming agent in fabrication of porous TCP ceramics.

Experimental

Tricalcium phosphate (Merck, Germany) and wheat particles (food grade and procured from local market) were used. Distilled water was used for preparation of slurries. Nitric acid (Merck, Germany) was used for pH adjustments. Twelve gr of TCP powder were mixed by 35 mL of distilled water. Then the mixture was added by 6 gr of wheat particles. Adjusting the pH to 3.5 using dilute nitric acid. The slurry was stirred for 1, 2 and 3 hours. Then the slurry was cast in cylindrical open stainless steel mould and heated in oven at 100°C for 30 min. Vegetable oil was used as lubricant for easy mould removal. The gelled body removed from the mould was dried in oven at 80°C for 24 h and 120°C for 8 h. The dried samples

were burned in a furnace at a rate of 2°C/min up to 350°C, 2°C/min up to 600°C and at a rate of 5°C/min up to sintering temperature (1000 and 1100°C). A holding time of 1 h was given at 350, 600 and sintering temperature. Density of the sintered samples was determined from dimensional measurement. Mechanical strength of the sintered samples was measured in a universal testing machine (Llyold LR10K Plus, UK). Microstructure of sintered samples was observed in a scanning electron microscope (Phenom Pro-X, Netherlands).

Results and Discussion

When the wheat particles mixed with water and stirred for different time at room temperature, the water was slowly absorbed by wheat particles. The particles in wheat particle-water mixture at room temperature were found non sticky. However, on heating, particles absorb water rapidly and transform the mixture into a strong gel. It has been observed that 35 gr of the wheat particles absorb 100 mL water at 100°C and form a strong gel [8]. The gelled bodies undergo shrinkage on drying in the oven. The shrinkage decreases with increasing stirring time. The drying shrinkage values for gelled bodies prepared from various stirring time is 55.87, 54.83 and 53.22% for 1, 2 and 3 hours, respectively.

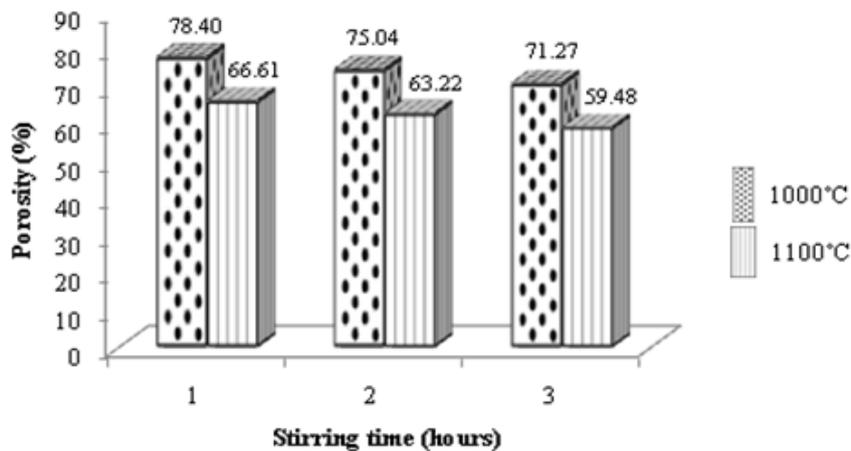


Fig. 2. The effect of stirring time on porosity of samples at different sintering temperatures

The bodies did not show any deformation or cracks during burnout of pore former and sintering. Porosity of the samples is in the range 59.48-78.40%. Fig. 2 shows porosity values of sintered samples from various variable. The porosity decreases with increasing stirring time. Conversely, a decreasing in porosity resulted in increasing density. The findings obtained in this research are in agreement with a study by Sopyan & Kaur [9] who reported a similar correlation between porosity and density. As the sintering temperature was raised, the porosity

decreases but not significantly. These result are in agreement with a study by Udoh et al. [10] who reported that TCP porosity decreases from 95 to 90% with increasing sintering temperature from 1450 to 1550°C. A tendency of decrease in porosity with increase in sintering temperature might arise from the fact that when sintering temperature increases, densification rate of particles escalates to form a denser structure with fewer pores.

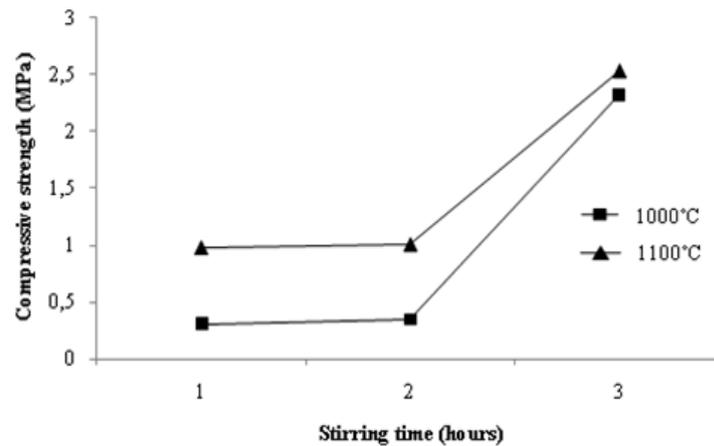


Fig. 3. The effect of stirring time on the mechanical strength of sintered porous TCP

Relationship between the compressive strength with stirring time and sintering temperature is shown in Fig. 3. It could be seen that the strength of samples at stirring time 3 hours raised significantly than two samples before. The strength of sintered samples is in the range 0.3-2.53 MPa. The strength increases with increasing stirring time and sintering temperature. Increasing the stirring time results in the breakdown of agglomerates and the slurry obtained tends to be homogenous. This in turn reduces the porosity and increases compressive strength.

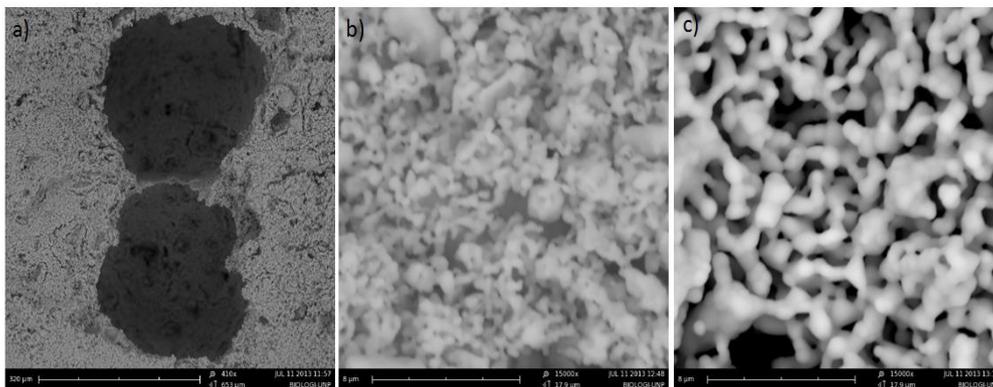


Fig. 4. (a) Macrostructure of sample prepared using slurry stirred for 1 hour and sintered at 1100°C. Microstructure of samples sintered at temperature of (b) 1000°C (c) 1100°C and stirring time of 3 hours

The pore sizes of sintered body of tricalcium phosphate are approximately 300 μm as shows at Fig. 4a. The change in morphology of pore structure as the sintering temperature was increased. It could be seen that pore interconnectivity in the porous TCP fired at 1100°C (Fig. 4c) was better than fired at 1000°C (Fig. 4b). Clinically, this kind of interconnective microporosity is a desirable feature since it facilitates the diffusion of calcium and phosphorus ions for mineralization [11].

Conclusion

Porous tricalcium phosphate ceramics were fabricated using wheat particles as a pore forming agent. The porosity and compressive strength of the samples were controlled by adjusting the stirring time and sintering temperature. Sintered tricalcium phosphate bodies with compressive strength of 0.3 – 2.5 MPa at porosities of 59-79% were obtained. Microstructures analysis revealed the existence of 300-310 μm macropores suitable for medical application

References

- [1] A. Uchida, S.M.L. Nade, E.R. McCartney, W. Ching, The use of ceramics for bone replacement, A comparative for study of three different porous ceramics, *Journal of Bone and Joint Surgery*, **66B** (1984), no. 2, 269-275.
- [2] D.R. Carter, W.C. Hayes, The compressive behavior of bone as two phase porous structure, *Journal of Bone and Joint Surgery*, **59A** (1977), no. 7, 954-62.
- [3] S.F. Hulbert, J.C. Bokros, L.L. Hench, J. Wilson, G. Heimke, Ceramics in clinical applications: past, present and future, *Journal of the American Ceramic Society*, **15** (1987), 189-213.
- [4] A. Ravaglioli, A. Krajewski, Implantable porous ceramics, *Journal of Materials Science*, **250** (1997), 221-230.
- [5] L.L. Hench, J. Wilson, *An Introduction to Bioceramics*, World Scientific, Singapore, 1993. <http://dx.doi.org/10.1142/2028>
- [6] T. Abdurrahim, I. Sopyan and Toibah A. Rahim, Recent progress on the development of porous bioactive calcium phosphate for biomedical applications, *Recent Patents on Biomedical Engineering*, **1** (2008), no. 3, 213-229. <http://dx.doi.org/10.2174/1874764710801030213>
- [7] O. Lyckfeldt, J.M. Ferreira, Processing of porous ceramics by starch consolidation, *Journal of the European Ceramic Society*, **18** (1998), no. 2, 131-140. [http://dx.doi.org/10.1016/s0955-2219\(97\)00101-5](http://dx.doi.org/10.1016/s0955-2219(97)00101-5)

- [8] K. Prabhakaran, A. Melkeri, N.M. Gokhale, S.C. Sharma, Preparation of macroporous alumina ceramics using wheat particles as gelling and pore forming agent, *Ceramics International*, **33** (2007), no. 1, 77-81. <http://dx.doi.org/10.1016/j.ceramint.2005.07.020>
- [9] I. Sopyan, J. Kaur, Preparation and characterization of porous hydroxyapatite through polymeric sponge method, *Ceramic International*, **35** (2009), no. 8, 3161-3168. <http://dx.doi.org/10.1016/j.ceramint.2009.05.012>
- [10] K. Udoh, M.L. Munar, M. Maruta, S. Matsuya, K. Ishikawa, Effects of sintering temperature on physical and compositional properties of α -tricalcium phosphate foam, *Dental Materials Journal*, **29** (2010), no. 2, 154-159. <http://dx.doi.org/10.4012/dmj.2009-079>
- [11] H. Yuan, Z. Yang, Y. Li, X. Zhang, J.D. De Bruijn, K. De Groot, Osteinduction by calcium phosphate biomaterials, *Journal of Materials Science: Materials in Medicine*, **9** (1998), no. 12, 723-726. <http://dx.doi.org/10.1023/a:1008950902047>

Received: October 1, 2015; Published: December 5, 2015