INFLUENCE OF YOLK ADDITION ON PROTEIN FOAMING-CONSOLIDATION POROUS ALUMINA CONTAINING

HYDROXYAPATITE NANOPOWDER

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ABSTRACT
Present paper reports the influence of yolk addition on physical properties of porous alumina-hydroxyapatite composites prepared using protein foaming-consolidation method. Alumina and hydroxyapatite (HA) powders were mixed with yolk at an adjusted mass ratio to make slurries. The slurries were cast into molds and the dried for foaming- consolidation process. The dried bodies were burned at 600ºC for 1 hour, followed by sintering at temperature of 1550ºC for 2 hours. The addition of yolk into the slurry shifted the rheological properties from shear thinning behavior to a Newtonian fluid and resulted in bigger foaming capacity. The porous alumina-HA composites with shrinkages in the range of 43.3 vol.% – 58.4 vol.% were obtained. The shrinkage of bodies increased with increasing concentration of yolk. The compressive strength was 7.5 MPa at 44.6% porosity 2.6 MPa at 57% porosity.
Key word: alumina, hydroxyapatite, composites, protein foaming-consolidation

INTRODUCTION

Recently, calcium phosphate ceramics have been widely used in biomedical field especially for bone surgery (Aguado et al, 2007). Hydroxyapatite (HA) is most preferable because it is a suitable material for body implantation and chemically close to calcium phosphate that is a mineral phase of bone (Yusof et al, 2008). Porous hydroxyapatite acts as a scaffold for the rapid ingrowths of vascularized connective tissue and bone. Porous HA exhibits strong bonding to the bone, because the porosity and bioactivity allows the in-growth of bone tissue to achieve full integration with the living bones. The porous HA are usually very brittle and prone to fracture upon sudden impact, particularly during the healing stage. Conversely, porous alumina has been attracting considerable attention for cell loading and bone grafts due to biocompatibility, inertness and chemical stability. Because of bioinert of nature of alumina, it has lack of bioactivity properties even though provides a very high mechanical properties. Therefore, it is desirable to develop scaffold implant materials with both reliable mechanical properties and porous structures, similar or superior to natural bones (Hong et al, 2008). A possible strategy of finding a material both string and bioactive is to use a composite where combine the higher strength of the alumina scaffold with the biological advantages of the hydroxyapatite surface.

In this paper, porous alumina-hydroxyapatite composites were prepared using protein foaming-consolidation method. The effect of yolk addition on physical and mechanical properties of porous body is investigated.

METHODOLOGY

The slurries were prepared by mixing 24 g of alumina (Sigma-Aldrich, USA) and 3.2 g of HA (Sigma-Aldrich, USA) powders with yolk (24 g, 34 g, 44 g and 54 g) in a beaker glass. The slurries were mechanically stirred (Heidolph, RZR 2052 control model) at 150 rpm for 3 hours in room temperature. Subsequently, the slurries were cast in cylindrical open stainless steel mould with 10.75 mm diameter and 15.10 mm height and dried in an air oven (Memmert, 100-800 model) at 180°C for 1 hour. Finally, the dried samples were burned in a furnace (Protherm, PLF 160/5 model) at a rate of 10°C min^{-1} up to 600°C for 1 hour for removal of the yolk and then sintered at rate of 2°C min^{-1} up to 1550°C for 2 hours. The rheological behavior of slurries was determined using a rheometer (ThermoHaake, VT 550 model). The crystallinity of the sintered porous samples was analyzed by XRD (Shimadzu, XRD-6000). The pore size, interconnection among pores and also the grain structure were examined using SEM (JEOL, 5600 model). The mechanical strength of the porous alumina-HA bodies was measured using a universal testing machine (Lloyd, LR10K plus model).

RESULTS AND DISCUSSION

The addition of yolk resulted in a significant decrease in viscosity over the shear rate considered (Figure 1). It is believed that since the yolk has generally low viscosity, the viscosity of slurry would decrease with increasing yolk content. A viscosity as high as 28.55 Pa s was observed for the slurry containing 34 g yolk at low shear rate ($10\ s^{-1}$) but it decreased significantly to 3.93 Pa s when the 54 g yolk was added. The viscosity value of the all slurries at higher shear rate ($175\ s^{-1}$) was in the range 1.66 – 7.59 Pa s indicating that the slurries are pourable under shear. The slurries containing lower yolk amount (34 g and 44 g) were pseudoplastic in behavior and it shifted to Newtonian fluid when yolk concentration was increased (54 g). It is elucidated that the existence of an inter-particle of ceramic grains increases with increasing solid loading in the slurry, and then undergoes a gradual breakdown with increasing shear rate, causing a decrease in viscosity of slurries.

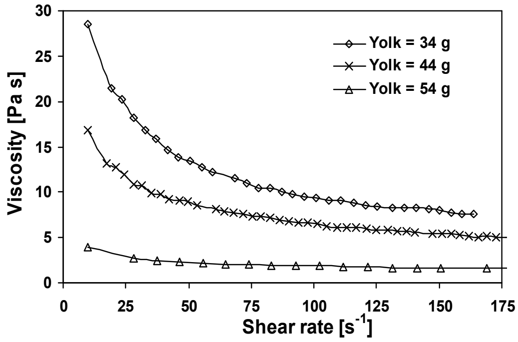

Figure 1. Rheological flow curves for shear rate vs viscosity.

Increasing yolk amount in slurry resulted in bigger pore size due to higher foaming capacity of slurry during the drying process. The amphiphilic character of protein brings a decrease in surface tension of slurry possibly leading to better foaming properties (Clarkson et al, 1999; Pugh, 1996). The foaming capacity of slurry added with 44 g and 54 g of yolk were 1.8 and 2.1 v/v. The bigger foaming capacity produces pores bigger in size and thinner walls, whereas smaller foaming capacity results in small pore size and thicker struts (Figure 2). It can be explained that increased yolk content in slurry would increase its foaming capacity, therefore pore size become bigger. The pore sizes are found in the range of 50 - 500 μm.

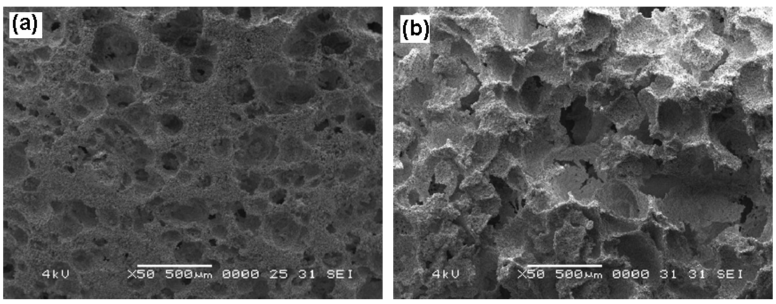

Figure 2. Macrostructures of sample bodies of (a) S1 and (b) S2.

Table 1 shows the effect of yolk addition on the physical properties of sintered porous bodies. The shrinkage of the samples increases from 46.9 to 70.3 vol.% when the amount of yolk in the green body increases from 34 to 64 gram. The yolk particles removed from green body during burn-out process, thus the shrinkage increased with the yolk amount. The increasing yolk concentration resulted in lower compressive strength. It can be explained that the increasing yolk mass in slurry would increase foaming capacity. Higher foaming capacity produced bigger pore size thus porosity increased. The compressive strength decreased from 7.5 MPa (at 44.6 % porosity) to 2.6 MPa (at 57.0% porosity) when yolk content increased from 24 g to 54 g. The mechanical properties of a porous material depend on the porosity as suggested by Gibson and Ashby, 1997.

Table 1. Effect of yolk addition on shrinkage, density, porosity and compressive strength

Yolk amount (g)	Shrinkage (Vol.%)	Compressive strength (MPa)
24	43.3	7.5
34	51.4	4.4
44	53.1	3.5
54	58.4	2.6

Figure 3 shows the XRD peaks for sintered sample containing 24 g yolk loading (sample S0). The alumina, HA and TCP phases were detected in the sample. Sintering of HA is complicated by the fact that HA is hydrated phase that decomposes to anhydrous calcium phosphates such as TCP at ~1200 – 1450˚C (Oktar and Göller, 2002).

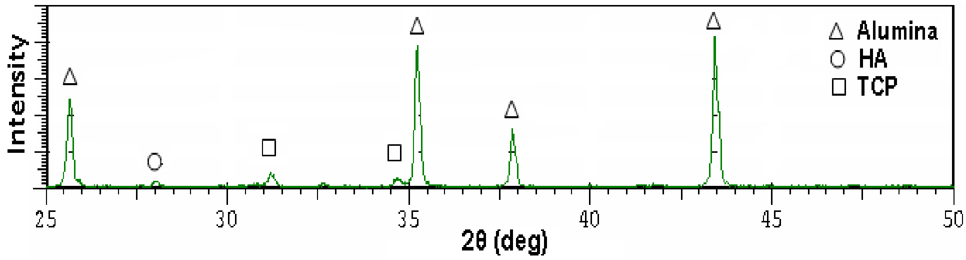

Figure 3. XRD analysis of sintered bodies at yolk mass of 24 g (S0).

CONCLUSION

The effect of yolk addition on physical properties of porous alumina-hydroxyapatite composites prepared via protein foaming consolidation method has been investigated. The yolk addition in slurry shifted rheology behaviour from shear thinning to Newtonian fluid. The shrinkage of bodies increased from 43.3 to 58.4 vol.%, respectively when the yolk addition increased from 24 to 54 g. The compressive strength obtained was in the range of 2.6 MPa-7.5 MPa with porosity between 44.6 – 57.0% with pore size of 50-500 μm.

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