

FORMATION OF POROUS ALUMINA USING WHEAT FLOUR AND WHEAT STARCH THROUGH SOL GEL CASTING

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Alumina is an advanced ceramic material which can be used as abrasives, sintering aid and biocompatible material for medical and dental composites. In biomedical application, alumina with porous structure is needed to increase adhesion of implants to bone. Production method for porous materials via sol-gel with addition of pore forming agents has gained considerable attention among various wet chemical methods due to its simplicity, reproducibility, lower cost of production, less time and less energy consumption. In the present study, alumina gel was first synthesized and later added with pore forming agents. Two pore forming agents were used i.e wheat flour and wheat starch because their effects on sol gel casting were not well defined besides their cost effectiveness. The amount of pore forming agents were varied (10, 15, 20 wt%). The gel was then casted and dried before being sintered at 1200 °C. Characterization methods were done using X-ray diffraction, Fourier transform spectroscopy and scanning electron microscope. X-ray diffraction shows that the samples were pure alumina while Fourier transform spectroscopy confirms that there was no pore forming agents present in the samples. Whereas scanning electron microscopy shows that pores produced were in the range of suitable for orthopedic implant application.

Keywords: Sol gel, alumina, wheat flour, wheat starch

INTRODUCTION

Alumina has been utilized since the early 1970's as an implant material, especially for artificial joint prostheses, mostly hip joints as well as teeth [1, 2]. Clearly, alumina has high biocompatibility with living tissues as well as possessing suitable mechanical properties, especially friction and wear) for orthopaedic applications [3]. However, alumina with porous structure is needed to increase adhesion of implants to bone

Porous alumina can be produced via direct consolidation method. In this method, pore-forming agents are used to generate pores on alumina body. Starch, is the most popular and common pore-forming agent used to produce pore in alumina body [4]. However, other pore forming agents like: globular protein (egg albumin) [5], plastic substance [6] and egg yolk [7] have been reported to be used as well. Starch consolidation is a newly developed forming technique suitable to produce porous materials. The advantage of using starch includes, simplicity of process, possibility of forming complex shapes using nonporous moulds with different pore volume fractions and pore sizes also low cost of equipments [8]. Although having similar composition with starch, wheat flour was originally

used to reduce the cost of processing of porous alumina. After a few test, it turns out the pore characteristics differ from that of starch consolidation method [4].

Since producing porous alumina with pore forming agents with alumina gel as a starting material had not been done by other people, the properties of such porous alumina are not known. It is therefore crucial to characterize the properties of the produced porous alumina. Since porous ceramics are widely used in biomedical applications, properties relating to such applications should be tested. Composition of porous alumina should be determined since it involves issues of biocompatibility. Pore characteristics are important for adhesion of porous ceramics as implant material.

METHODS AND MATERIALS

The main raw materials used in this study were aluminium isopropoxide (ALP) (Merck, Germany) and aluminium nitrate nonahydrate (ALN) (Merck, Germany). Whereas, wheat flour (Jalanan Metrik Sdn Bhd, Malaysia) and wheat starch (Jalanan Metrik Sdn Bhd, Malaysia) were used as pore forming agents.

Firstly, 0.5M ALN was prepared and while stirring, ALP was added and the mixture was continuously stirred. The prepared mixture was then gone through aging and evaporation to increase the viscosity. During the evaporation, pore forming agent was added at various composition (10 wt%, 15 wt%, and 20 wt%) later named as F10, F15, F20 for wheat flour and S10, S15 and S20 for wheat starch. Gel obtained was then poured into an expendable mould followed by drying in an oven at 90 °C for 10 hours. This duration was chosen after preliminary studies were done and it was believed to be sufficient time for the gel to dry up to become solid. Dried sample was then sintered at 1200 °C for 1 hour with a heating rate of 5 °C/ minute.

Effects of using different pore forming agent to the porous alumina produced were characterized using X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Fourier Transformed Infrared Spectroscopy (FTIR) (IR spectroscopy V 5.0.2, United State).

RESULTS AND DISCUSSION

XRD result in Fig. 1 shows the phase present in the sample sintered at 1200 °C which was also observed in other samples. The XRD spectra matched with reference pattern aluminum oxide, Al₂O₃ (ICDD 00-001-1243) with no other phases were found. This indicates that the sample is pure alumina with no impurities. In other words, the pore forming agent which was added before sintering process was completely burnt off.

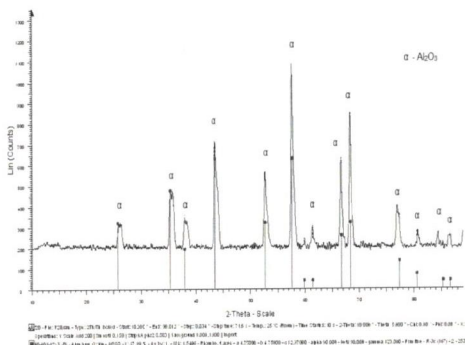


Fig. 1. XRD spectrum of porous alumina produced

These results were in agreement with FTIR results (Fig. 2) where there was no chemical bonding observed referred to the pore forming agents used in this study.

Fig. 2 shows the FTIR spectrum for sample S10 which represent all samples, since all the samples exhibit similar results.

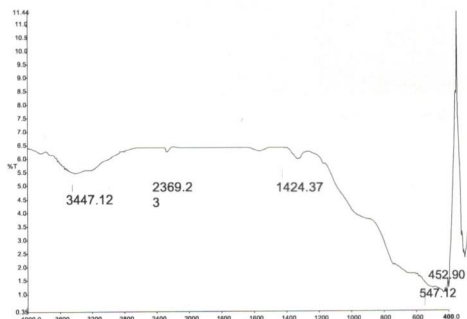


Fig. 2. FTIR spectrum for sample S10

Four regions with peaks are observed; 4000 – 3500, due to the OH bonds which suggests water absorption in the sample, 2400 – 2250 cm⁻¹ due to O=C=O stretching, 1900 – 1400 cm⁻¹, which is mainly due to C=O stretching and 800 – 400 cm⁻¹ which refers to chemical bond that cannot be resolved.

Lee and Condrate [9] obtained similar results in their study and they concluded that this region is refers to sample of metal oxide. In addition, the metal oxide bond present in this study has proven by XRD as aluminum oxide. These FTIR results also show that the organic bonds from pore forming agents, which were added during the synthesis process, does not present in the final samples.

SEM results in Fig. 3 shows that pores appeared in all samples with different characteristics which are simplified in Table 1.

The occurrence of irregular shape is due to the irregular shape of wheat flour. Although wheat flour relies on protein assisted forming mechanism to create pores, which involves gas releasing [4], the amount of pore forming agent used in these two samples were not sufficient to create pores with spherical shape. On the other hand, sample F20 shows spherical pores which take the shape of gas bubbles released by wheat flour.

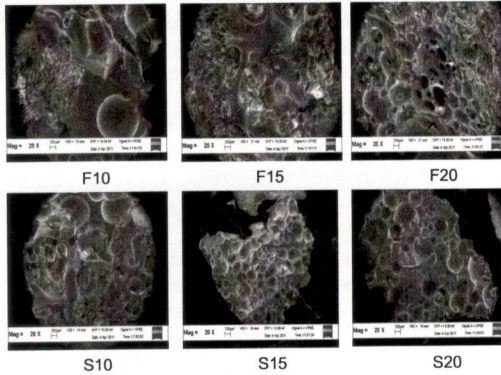


Fig. 3. Microstructure of porous alumina powder obtained using wheat flour and wheat starch pore former agents at various compositions (magnification 20X)

Table 1 Pore characteristics of the samples

No.	Sample	Pore Shape	Average pore size (µm)	Pore Inter - connectivity
F10	With 10 wt% wheat flour	Irregular	-	No
F15	With 15 wt% wheat flour	Irregular	342.7	No
F20	With 20 wt% wheat flour	Irregular	315.9	No
S10	With 10 wt% wheat starch	Irregular	-	No
S15	With 15 wt% wheat starch	Circular	277.4	Minimal
S20	With 20 wt% wheat starch	Circular	276.8	Extensive

Samples prepared using wheat starch (S10, S15, S20) on the other hand had spherical pores. This is due the fact that wheat starch relies on swelling of starch granules and become sacrificial pore formers. They are being burnt off without releasing gas [4]. Originally, wheat starch particles are in spherical shape, even with swelling and maintain their shapes. Therefore, after being burnt off, the particles will leave a spherical pore on the alumina.

In term of size of the pores, although all samples show porosity on the surface, sample F10

and sample S10 did not show homogeneity on the pore distribution. Therefore, it was not appropriate to discuss pore size data for these two samples.

Generally it was observed that sample using wheat flour have larger pore size compare to sample using wheat starch. According to Gregorova et al., [4], porous body that uses flour as pore forming agent will have larger pores due to the difference in mechanism of creating pores. However, based on the pore size analysis, most of the pores are in the range of 100 µm to 600 µm. This pore size range is suitable for orthopedic implant application.

CONCLUSION

In this research, porous alumina had been successfully produced using alumina gel from sol gel method as starting material. The gel casting method with the addition of pore forming agents proved to be a reliable and repeatable method for creating porous ceramics. The procedure was relatively inexpensive and simple. Porous alumina samples (with no other phase occurred) produced have pore size between 100 µm and 600 µm which is in the range for orthopedic implants, as this pore size range is suitable for oseoconductivity of cells.

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REFERENCES

- [1] Akiyoshi, Y., (1998). *Quantitative Characterization and Performance of Porous Implants for Hard Tissue Applications: A Symposium*. Vol. 417
- [2] Bose S., Darsell J., Hosick H.L., Yang L., (2002) *J. of Mat. Sc.*, 13, 23-28
- [3] Park, J. (2008). *Bioceramics: Properties, Characterizations, and Application*, Springer
- [4] Gregorová E., Pabst W., Zivcová Z., Sedlářová I., S. Holíková (2010). *J.I of European Cer. Soc.*, 30, 2871-2880
- [5] Bhattacharjee S., Besra L., Bimal P. Singh., (2006). *J. of European Cer. Soc.*, 27, 47-52
- [6] Isobe T., Kameshima Y., Nakajima A., Okada K., (2006) *J. of European Cer. Soc.*, 26, 957-960

- [7] Fadli A. and Sopyan I., (2009). *Mat. Res. Innov.*, Vol. 13, pp 327-329
- [8] Rodriguez-Lorenzo, L.M. Vallet-Regi, M., Ferreira, J.M.F., (2002), John Wiley & Sons, Inc, pp 232-240
- [9] Lee D.H., Condrate Sr. R.A., (1995), *Mat. Lett.*, 23, 241-246