Effect of sintering on the physical properties of porous β-TCP scaffolds

N.F. Ishak[1,2], Z. Mustafa[1,2,*], R. Othman[1, 2], S.H. Sheikh Md. Fadzullah[3], A.R. Mahamad Sahab[4]

[1)] Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[2)] Advanced Manufacturing Centre, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[3)] Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
[4)] Advanced Material Research Centre, SIRIM Berhad, Shah Alam, 40450, Selangor, Malaysia

[*]Corresponding e-mail: zaleha@utem.edu.my

Keywords: Tricalcium phosphate; scaffold; bioactive

ABSTRACT – This research work investigates the effect of sintering temperatures on the physical properties of porous beta-tricalcium phosphate (β-TCP) scaffolds produced via a template method. A polyurethane (PU) foam was immersed in β-TCP slurry by using a roller infiltration method. The impregnated foam was subsequently sintered to 1400ºC, 1450ºC and 1500ºC. The sintered scaffolds were then characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) for evaluation of crystalline phases as well as pore and surface morphology. An increase in sintering temperature reveal an increase in crystallinity as well as the lessening of pores and voids in the struts of the scaffold as a result of progressive sintering.

1. INTRODUCTION

Recent advances in tissue engineering aim to regenerate damaged tissues. It can be accomplished by combining cells from the body with highly porous scaffold biomaterials, which act as templates to guide new tissue regeneration [1]. Hence, the scaffold should mimic the biological bone in order to optimize integration into surrounding tissues. Due to the simplicity of processing technique and inexpensive technology used, the production of scaffolds are favourable. The drawback of this process is the difficulty in specimen handling after sintering due to their brittle and porous structure [2]. Principally, tricalcium phosphate (TCP) exists in many polymorphs but only two phases (α and β) that are commonly used in biomedical materials [3]. β-TCP is greatly biocompatible due to their bioactive nature and ability to set up a resorbable interlocking network within the injury site to encourage healing. One of the most important aspect for successful scaffold application is its porosity, in order to allow nutrient transportation during cell growth. The aim of this work is to investigate the effect of sintering temperature on the phase and microstructure characteristics of β-TCP scaffolds.

2. METHODOLOGY

Tricalcium phosphate (Rekagraph USM, Malaysia) with a Ca:P ratio (1.5) has a mean particle size of $d_{0.5}$ = 6.03 μm and a specific surface are of 2.30 m^2g^{-1} . Polyvinyl alcohol (PVA) (Sigma Aldrich, UK) is used as the binder and polyurethane (PU) foam as the template. The β-TCP powder was mixed in 2% polyvinyl alcohol aqueous solution at a powder to liquid ratio of 40:60. The mixture was then stirred using a magnetic stirrer plate for approximately 2 hours at room temperature to ensure homogenous mixing. PU foam was immersed in the solution and rolled out to ensure full infiltration of the slurry. The foam was then dried in an oven at 60 ºC overnight. The samples were subsequently subjected to three different firing protocols with maximum sintering temperatures of 1400 ºC, 1450 ºC and 1500 ºC. The compositional analysis of sintered β-TCP scaffolds were characterized using PANalytical X-ray diffraction unit (model X'Pert Pro MPD PW3060/60) operating at room temperature using Cu Kα radiation (α =1.54178 Å).. For morphological analysis, scanning electron microscopy SEM EVO 50 (Carl Zeiss SMT, UK)) was used at an accelerating voltage of 5 kV.

3. RESULTS AND DISCUSSION

The XRD patterns of sintered TCP scaffolds subjected to three different sintering regimens: 1400 ℃, 1450 ℃ and 1500 ℃ are shown in Figure 1.-These XRD patterns match that of pure β-TCP pattern with JCPDS number 55-0898 without any secondary phase formation. It can be seen that all samples sintered at different temperatures show prominent peaks with a preferred orientation at (0 2 10). However, the peak becomes more intense at higher temperatures owing to an increase in the crystallinity of the calcium phosphate. This is consistent with other previous study [4] which reported that the composition of β-TCP was not changed during the sintering process regardless of β-TCP content. Figure 2 shows the microstructures of porous β-TCP at different sintering temperatures in comparison to the microstructure of the PU template (Figure 3). These results reveal that a good pore interconnectivity characteristic. At the two lower sintering temperatures, the scaffolds contain more voids and cracks–suggestive of their brittle nature. As the temperature is increased to 1500°C, the surface appears to be smoother and denser suggesting that progressive sintering through diffusion

had taken place.

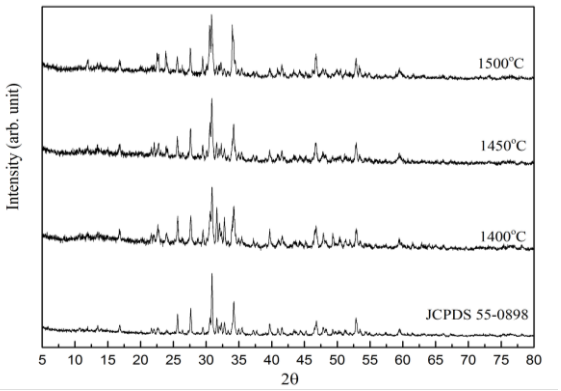

Figure 1 XRD patterns of β-TCP sintered at different temperatures of 1400°C, 1450°C and 1500°C.

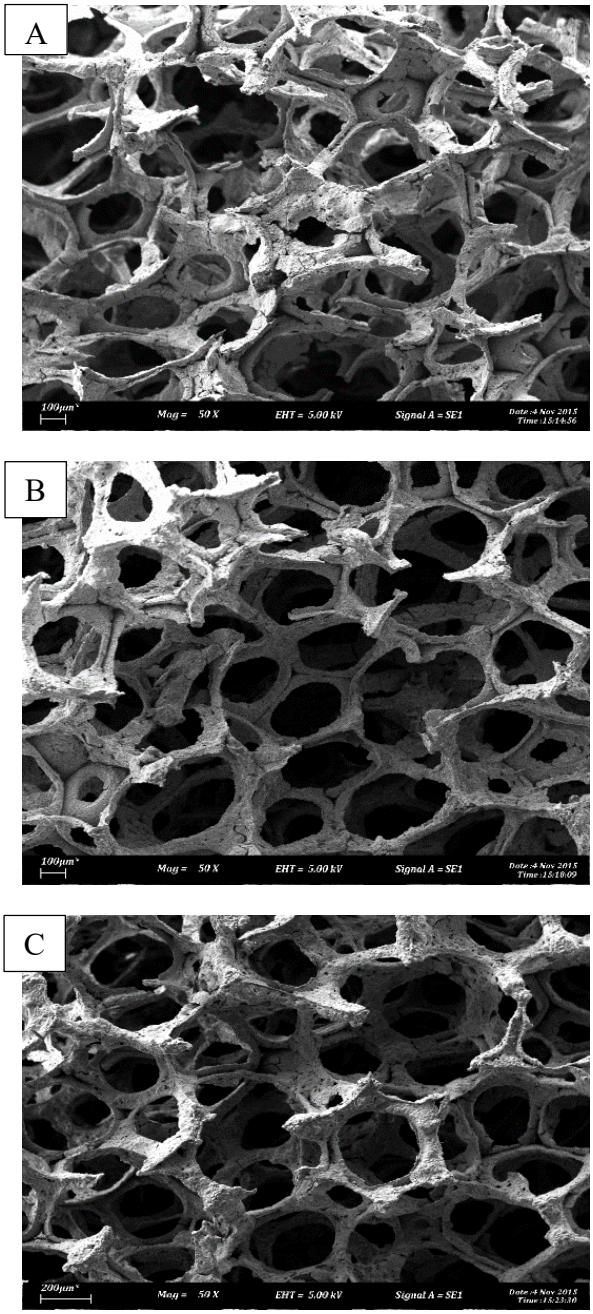

Figure 2 SEM images of porous scaffolds at different sintering temperatures (A) 1400°C; (B) 1450ºC; (C) 1500ºC.

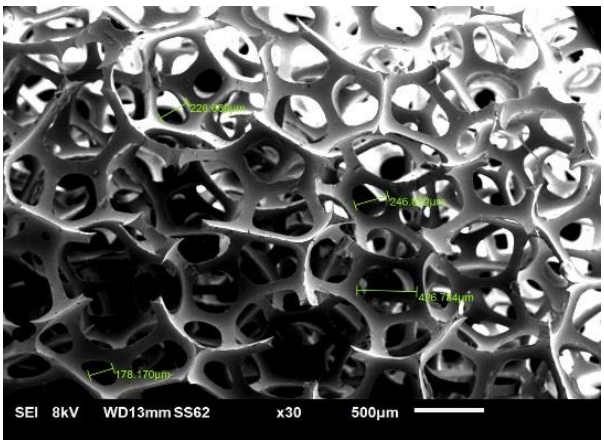

Figure 3 SEM image of PU foam.

4. CONCLUSION

In conclusion, the preliminary results showed an increase in the crystallinity of the β-TCP phase as the temperature was increased and no additional phase was identified. Morphological analyses revealed that an interconnected pore structure was successfully produced at all sintering temperatures. The scaffold struts surface appeared to be more dense with less cracks and voids with an increase of sintering temperature suggesting that better diffusion was achieved between the β-TCP particles at 1500°C.

ACKNOWLEDGEMENT

The authors are grateful to the Ministry of Education Malaysia for funding this research project under Research Acculturation Grant Scheme RAGS/1/2014/TK04/FKP/B00073 and Universiti Teknikal Malaysia Melaka (UTeM) for the use of laboratory facilities.

REFERENCES

[1] F.J. O'brien, "Biomaterials & scaffolds for tissue engineering," *Materials Today*, vol. *14*, no.3, pp.88-95, 2011.

[2] T.Y. Yang, W.Y. Kim, S.Y. Yoon, and H.C. Park, "Macroporous silicate ceramics prepared by freeze casting combined with polymer sponge method," *Journal of Physics and Chemistry of Solids*, vol. *71*, no. 4, pp.436-439, 2010.

[3] S.B. Sulaiman, T.K. Keong, C.H. Cheng, A.B. Saim and R.B.H. Idrus, "Tricalcium phosphate/hydroxyapatite (TCP-HA) bone scaffold as potential candidate for the formation of tissue engineered bone," *The Indian Journal of Medical Research*, vol. *137*, no. 6, pp.1093, 2013.

[4] Z.K. Kim, J.J. Oak, H. Kimura, T. Goto, A. Inoue and S.Y. Yoon, "Architecture of porous hydroxyapatite scaffolds using polymer foam process," *Journal of Biomechanical Science and Engineering*, vol. 4, no.3, pp.377-383, 2009.