FOAMED CALCIUM PHOSPHATE CEMENTS AND THEIR BEHAVIOUR DURING INCUBATION IN DISTILLED WATER

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Introduction

Bioactive, foamed calcium phosphate bone cements (fCPCs), obtained by surfactant-assisted foaming, are a relatively new group of bone substitutes[1]. Thanks to their porosity, these materials can act as scaffolds for bone tissue regeneration.

The selection of surfactants for the preparation of foamed bone cements is a very difficult issue. It is necessary to pay attention to many properties of the surfactant, such as its: chemical nature (ionic, non-ionic, etc.), hydrophilic-lipophilic balance value or solubility in water. The surfactant characteristics have a direct impact on the final materials properties, as can be seen from the previously described non-foamed cements [2].

In this study, the degradation behaviour of fCPCs was studied. The changes in water pH and ionic conductivity during materials incubation were investigated. The effect of 28-day immersion was evaluated by fCPCs microstructure observations using scanning electron microscopy (SEM). The release profile of the surfactants (Tween 20, Tween 80 and Tetronic 90R4) used for the cement foaming, was determined via high-performance liquid chromatography coupled with mass spectrometry (HPLC-MS).

Materials and Methods

Alpha-tricalcium phosphate (α-TCP) synthesized by the wet chemical method was used as the solid phase of cements. As the liquid phase 2 wt.% Na_2HPO_4 aqueous solutions with selected surfactant (10 $g \cdot L^{-1}$) was used. The foaming agents - surfactants used in the study were: Tween 20, Tween 80 and Tetronic90R4 (Sigma Aldrich). The liquid to powder ratio (L/P) was equal 0.7 $g \cdot g^{-1}$. Cements fTW20, fTW80 and f90R4 were obtained by mixing the powder phase with a previously foamed surfactant solution (liquid phase). The control samples did not contain any surfactant (fCTRL). The cement samples were incubated in distilled water (1g of sample for 100 mL of water) at 37°C for 28 days. The measurements of ionic conductivity and pH were performed on days 0, 1, 3, 7, 14, 21, and 28 by pH/conducto-meter (Hanna H198129 Combo). SEM observations (NovaNano SEM) of materials were performed before and after 28-day incubation. In order to study the surfactants release from cement samples, after 3, 7, 14, 21 and 28 days, 300 µl of water was collected and stored at -20°C for subsequent analysis by HPLC-MS (Agilent 1290 Infinity, MS Agilent 6460 Triple Quad Detector).

Results and Discussion

Samples immersion led to increase of water ionic conductivity. Cements with polysorbates, i.e. Tween 20 and Tween 80 increased ionic conductivity more than cements without surfactant (fCTRL) or with Tetronic 90R4. The pH of water decreased along with the materials' soaking time. After 28-day incubation, on the cement surfaces, the presence of numerous apatite forms was observed (FIG. 1). The differences between the control sample (fCTRL) and those with surfactants was in the shape of apatite crystals, which was the needle-like and oval-like, respectively. In surfactant release study, for polysorbates (Tween 20, Tween 80) no significant changes in surfactant concentration after the third day of incubation was observed. The concentration of Tetronic 90R4 decreased up to day 7 and then reached a plateau.

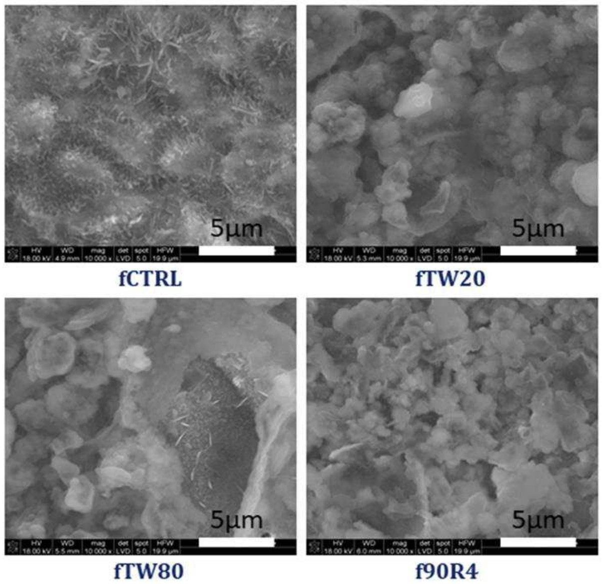

FIG. 1. Microstructure of cement surfaces after 28 days of incubation in distilled water.

Conclusions

Ionic conductivity can be related to the degree of cement foaming, which was much higher in the case of cements with Tween 20 or Tween 80. In addition, these surfactants degrading to their building fatty acids (lauric for Tween 20 and oleic for Tween 80) [3] contribute to the increase in ionic conductivity. On the surfaces of cements with surfactants, oval-shaped apatite crystals over plate- or needle-like morphologies were dominant, what stays in agreement with the results of our previous studies [4]. The release studies revealed that the surfactant concentration did not correspond to the theoretical value, i.e. the maximum amount that could be released from the material. Most likely, the released compounds had already degraded significantly before the third day of incubation. Obtained materials need further research.

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References

[1] W. Wang et al., Bioact. Mater., 2 (2017) 224–247.
[2] E. Cichoń et al., Langmuir, 35 (2019) 13656–13662.
[3] R.S.K. Kishore et al., Pharm. Res., 28 (2011) 1194–1210.
[4] E. Cichoń et al., RSC Adv., 11 (2021) 23908–23921.