SURFACTANT-ASSISTED FABRICATION AND EVALUATION OF MACROPOROUS CALCIUM PHOSPHATE BONE CEMENTS

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Introduction

An ideal bone substitute should possess many properties attributed to autologously transplanted tissue. The implant should allow rebuilding not only the bone tissue itself but also its vascularization. To make it happen, the new bone should be constantly supplied with nutrients. For this purpose, materials used in bone substitution should exhibit adequate porosity. The biomimetic approach to bone tissue engineering requires the use of specialized macroporous scaffolds. Calcium phosphates cements (CPCs) are an alternative to traditionally sintered porous ceramics for applications in bone tissue engineering.[1-3] Cement preparation does not require high-temperature processing.[4] Recent research has approached the problem of inducing macroporosity inside the bone cement without influencing its normal setting.[5-6] The aim of this study was a surfactant-assisted fabrication and preliminary evaluation of the novel macroporous chemically bonded α-TCP scaffolds. The effect of surfactants addition on cement paste foamability and phase composition of final materials was investigated. Furthermore, the chemical structure of the obtained materials was determined by vibrational spectroscopy (ATR-FT-IR).

Materials and Methods

The solid phase of obtained cements consisted of the highly reactive α-tricalcium phosphate. The initial α-TCP powder was synthesized by the wet chemical method. For the synthesis, CaOH (POCH, Poland) and 85% phosphoric(V) acid (POCH, Poland) were used, both with chemical purity. As the liquid phases 2 wt% Na_2HPO_4 solutions with 10 wt% various surfactant addition were used. A number of surfactants were proposed: Tween 20 (Polyoxyethylenesorbitan monolaurate) – TW20, Tween 80 (Polyoxyethylenesorbitan monooleate) – TW80 and Tetronic 90R4 (Ethylenediamine tetrakis(ethoxylate-block-propoxylate) tetrol) – 90R4.

Two different methods of obtaining the macroporous cements were used. One of them was mixing the powder with an already foamed surfactant solution. The second method consisted in foaming already mixed cement paste. Foaming step was performed with a domestic food mixer (BOMANN, Germany) for 30s. The control samples - without any surfactant, were obtained using the same methods. Identical molds were used for all of the materials.

Foamability [%] of the cement pastes was measured according to the equation:

$$\text{Foamability} = \frac{Vs - Vc}{Vc} \; x \; 100\%$$

where:
Vs – volume of the studied cement
Vc – volume of the control cement

Phase composition and chemical structure (XRD, D-2 Phaser, Bruker; ATR-FT-IR, Tensor 27, Bruker) of the obtained cement type materials have been studied.

Results and Discussion

Foamability of the cement pastes is shown in TABLE 1.

TABLE 1. Calculated foamability of the obtained cements in comparison with the control cement.

	TW20	TW80	90R4
Method I	150%	175%	50%
Method II	80%	40%	0%

The foamability of the cements depended on the foaming method which was used during their preparation. The first method led to higher foamability of the obtained cements. The powder X-ray diffractograms of the examined samples are presented in FIG. 1. The diffractograms of all prepared cements after setting and hardening revealed the presence of two crystalline phases: αTCP and HAp.

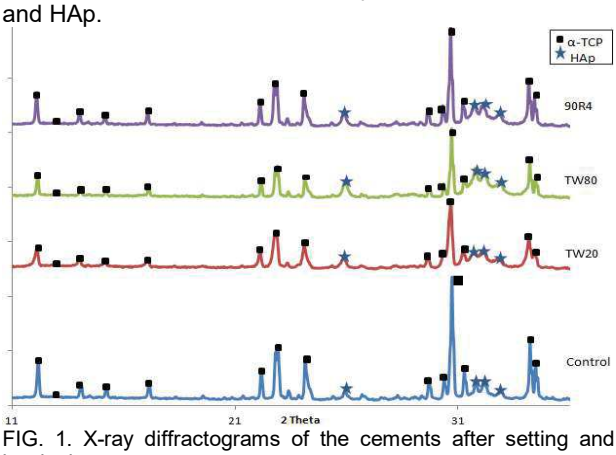

FIG. 1. X-ray diffractograms of the cements after setting and hardening.

ATR-FT-IR analysis of the obtained cements revealed that all spectra revealed the strongest bands in the regions 1200–900 and 650–500 cm^{-1} which are attributable to the vibrations of phosphate groups. The broad band from 3650 to 2600 cm^{-1} is assigned to O–H stretching (residual water). In the cements with an addition of Tweens (TW20 or TW80) C=O stretching band at 1738 cm^{-1} caused by ester structure were present. The carbon–carbon bonds of poloxamine Tetronic 90R4 appeared at 2860–2880 cm^{-1}. N-H bending in 90R4 could not be found at the same time because it coincided with the O-H stretching originated from water.

Conclusions

Macroporous calcium phosphate bone cements have been successfully obtained. The foaming method had a significant influence on their foamability. This parameter differed from 0 to 175%. Cement pastes with Tween (20 or 80) addition revealed higher foamability values than pastes with Tetronic 90R4.

The diffractograms of all studied samples revealed the presence of only two crystalline phases: αTCP and HAp.

FT-IR spectra confirmed the presence of surfactants in the obtained materials.

Macroporous, foamed cements need further research.

Acknowledgments

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