HYDROXYAPATITE/CHITOSAN HYBRID-BASED BIOMICROCONCRETES AS NOVEL BONE SUBSTITUTES – IN VITRO STUDIES

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Introduction

Hybrid materials gained great attention in recent years. In our study materials composed of hybrid hydroxyapatite/chitosan (HAp/CTS) granules as aggregate, αTCP as a setting phase, and pectin solutions as a liquid phase, were developed. Hybrid granules combine advantages of hydroxyapatite (i.a. biocompatibility, bioactivity) and CTS (i.a. rapid biodegradation, high biocompatibility and antibacterial properties).

Materials and Methods

The following components were used to prepare biomicroconcretes: hydroxyapatite/chitosan (HAp/CTS) hybrid in the form of precipitate (18% dry weight), fine powder and granules (300-400um), as well as α-TCP powder, and pectin solutions. HAp/CTS hybrid materials, containing different chitosan content (15, 20, and 25 wt.%), and highly reactive α-TCP powder were synthesized using a wet chemical method. Two types of low esterified amidated pectins from citrus peels (CP) and apple pomace (AP) were used. The materials were prepared by mixing the HAp/CTS phase, α-TCP powder and pectin solutions. Materials were incubated in simulated body fluid (SBF). Mass change, swelling, phase composition (XRD, FTIR) and microstructure (SEM/EDX) of the biomicroconcretes were monitored during incubation. Furthermore, the changes in pH, as well as calcium and phosphate ion concentration (ICP-OES) in SBF were measured.

Results and Discussion

The biomicroconcretes showed a gradual increase in mass during incubation in SBF (FIG. 1A). Mass changes can be related to two processes: degradation of the materials (especially organic phase – chitosan and pectins) and inorganic phase changes (α-TCP transformation to HAp and HAp precipitation as a result of SBF supersaturation). The results indicated that the second process is predominant. However, after 35 days of incubation, biomicroconcretes containing HAp/CTS hybrid phase with the lowest content of CTS (15 wt.%) exhibited the highest increase in mass. It may indicate that after a longer incubation period degradation rate of the materials composed of HAp/CTS hybrid phase with a higher content of CTS (20 and 25 wt.%) was higher. In the first day of incubation, the swelling ratio depended on CTS concentration in hybrid phase – the highest value was recorded for HAp/25CTS_AP material (FIG. 1B). Between 3rd and 14th day of incubation, the swelling ratios of all materials reached similar values (~10%) and did not change significantly. After longer incubation periods (21 and 35 days), the values increased with increasing CTS concentration in hybrid phase.
Phase composition analysis showed that just after 3 days of incubation in SBF almost complete transformation of α-TCP to HAp occurred (TABLE 1). Furthermore, the gradual decrease of calcium and phosphate ion concentration in SBF, as well as SEM observations indicated the formation of HAp layer on the surface of biomicroconcretes, indicating their bioactivity.

TABLE 1. Phase composition of the biomicroconcretes based on XRD analysis.

Sample name	7 days in air		3 days in SBF		7 days in SBF	
	αTCP [wt%]	HAp [wt%]	αTCP [wt%]	HAp [wt%]	αTCP [wt%]	HAp [wt%]
HAp/15CTS_AP	25	75	3	97	1	99
HAp/20CTS_AP	22	78	3	97	2	98
HAp/25CTS_AP	23	77	4	96	1	99

Conclusions

Obtained biomicroconcretes showed bioactivity and rapid transformation of setting phase (α-TCP) to HAp. The results indicated that the degradation and swelling rates of the materials can be modulated by the use of HAp/CTS hybrid phase with different chitosan concentration.

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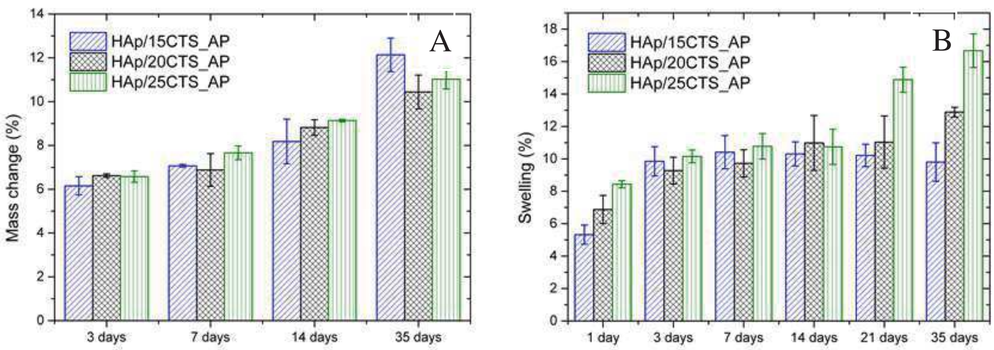

Figure 1. Mass change and swelling of the biomicroconcretes during incubation in SBF.