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Calcium phosphate ceramics from hydroxyapatite suspensions containing sucrose and diammonium phosphate aqueous solutions

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One of the most important development directions of modern medical materials science is the creation of inorganic materials for artificial bone implants. The regenerative medicine development requires the creation of resorbable materials based on calcium phosphates in which the Ca/P ratio is smaller than in the main inorganic component of the bone – hydroxyapatite (HA, Ca/P = 1.67). Creation of such materials requires the development in methods for synthesis of calcium phosphates with Ca/P ratio = 1.5; 1.0; 0.5. The composite materials containing phase of calcium polyphosphate (Ca/P = 0.5) have the highest solubility. The purpose of the present work is to obtain ceramic materials containing calcium polyphosphate, calcium pyrophosphate and calcium orthophosphate phases.

We assume that depending on the components ratio, various bioresorbable phases are formed via following reactions during the heat treatment.

$$3Ca_{10}(PO_4)_6(OH)_2 + 2(NH_4)_2HPO_4 = 10Ca_3(PO_4)_2 + 4NH_3 + 6H_2O$$
$$Ca_{10}(PO_4)_6(OH)_2 + 4(NH_4)_2HPO_4 = 5Ca_2P_2O_7 + 8NH_3 + 7H_2O$$
$$Ca_{10}(PO_4)_6(OH)_2 + 14(NH_4)_2HPO_4 = 10Ca(PO_3)_2 + 28NH_3 + 22H_2O$$

The ceramic materials were obtained from highly concentrated hydroxyapatite suspensions in an environment of sucrose and diammonium phosphate aqueous solutions. Cord-shaped samples were formed via 2 mm diameter plastic injectors. The ability to form homogeneous paste layers and stable multi-layer constructions was studied. The most reassuring results were given by suspensions with HA/sucrose solution ratio of 0.8 g/ml and sucrose aqueous solution concentration of 0.5M.

After molding the samples were sintered in the range of temperatures from 900 °C to 1100 °C depending on the target phase. Phase composition of the sintered samples is presented below. The properties of ceramic materials obtained were also studied with thermal analysis (TA), mass spectrometry (MS) and scanning electron microscopy (SEM).

Target phase	Ca/P ratio	Sintering temperature	Phase composition
β-$Ca_3(PO_4)_2$	1.5	1100 °C	β-$Ca_3(PO_4)_2$
β-$Ca_2P_2O_7$	1.0	1100 °C	β-$Ca_2P_2O_7$, β-$Ca_3(PO_4)_2$
β-$Ca(PO_3)_2$	0.5	900 °C	β-$Ca(PO_3)_2$

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One-pot sol-gel synthesis of scaffolds for biomedical applications

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The development of three-dimensional porous structures (scaffolds) with ability to interact with the body leading to the formation of new bone at a rate equal to its own degradation rate has become a major challenge in tissue engineering.

In the present study the scaffolds for bone regeneration applications were developed, within the glass systems SiO_2-CaO-TiO_2 and SiO_2-CaO-ZrO_2 produced by one-pot sol-gel method using two types of surfactants (sodium lauryl sulphate, SLS, and polyethylene oxide, PEO) as porogenic agent. The study was focused on the influence of the type and amount of surfactant used, the introduction of alkoxides of zirconium and titanium, and the potential bioactivity of the scaffolds.

The use of a nonionic surfactant (PEO) as a porogenic agent, allowed the production of monolithic scaffolds with morphological characteristics more interesting that the ones produced by the introduction of an anionic surfactant (SLS). The former showed a homogeneous network of macropores interconnected and with larger macropores (> 50μm) as well as higher values of surface area (262m^2 / g). In addition, it was also found that the scaffolds of the system SiO_2-CaO-ZrO_2 showed significantly greater surface areas than the scaffolds produced from the system SiO_2-CaO-TiO_2, showing therefore a greater ability to interact with the surrounding medium and form an apatitic surface layer.

The bioactivity tests showed that all scaffolds exhibited the formation of a layer of calcium phosphate on their surface, and a constant degradation rate, thus showing the high potential of applying these materials in the field of bone regeneration.