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CALCIUM PHOSPHATE CERAMICS WITH INORGANIC ADDITIVES

The aim of this work is to development of compositions and technological parameters for the production of calcium phosphate ceramics with Al_2O_3 additives obtained by solution combustion synthesis (SCS), to establish the relationship between physicochemical properties of the synthesized materials, their structure and composition of initial mixture.

The first phase of the research focused on the synthesis of hydroxyapatite. It was conducted using calcium nitrate tetrahydrate $Ca(NO_3)_2 \cdot 4H_2O$ (chemically pure grade, GOST 4142); ammonium hydrogen phosphate $(NH_4)_2HPO_4$ (grade A, GOST 8515); ammonium hydroxide NH_4OH (25 % NH_3 in H_2O, ultrapure grade, GOST 24147) and distilled water according to the following equation:

$$Ca(NO_3)_2 + 6(NH_4)_2HPO_4 + 8NH_4OH \rightarrow Ca_{10}(PO_4)_6(OH)_2 + \\ + 20NH_4NO_3 + 6H_2O.$$

The solution obtained by dissolving $Ca(NO_3)_2 \cdot 4H_2O$ in a beaker was added dropwise into a solution of $(NH_4)_2HPO_4$ with constant stirring. By adding NH_4OH, the pH of the solution was kept constant at 9.0 throughout the synthesis process. The synthesis was carried out at 60 ºC. The precipitate and the mother liquor were cooled down to room temperature and exposed to SHF electromagnetic radiation for 30 min. The resulting precipitate was separated by filtration, washed with distilled water and dried at 80 ºC. Particle size of obtained powder was between 0.05–50 μm (Fig. 1, Analysette 22, Germany), X-ray phase analysis (diffractometer DRON-2, Russia) indicated hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$.

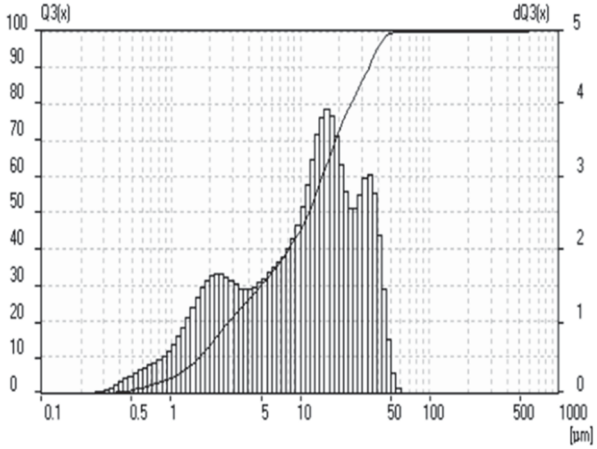

Figure 1 – Particle size distribution of the synthesized hydroxyapatite powder

Aluminium oxide were synthesized by SCS method during the second phase of the research. The following materials were used: aluminum nitrate ninehydrate – $Al(NO_3)_3 \cdot 9H_2O$ (analytical grade, GOST 3757), urea CH_4N_2O (analytical grade, GOST 6691). The quantities of used reactant are summarized in Table.

Quantities of reactants used in preparation of reactive solutions

No. solution	Fuel / oxidizer ratios (φ)	Reactant (wt.%)	
		$Al(NO_3)_3$	CH_4N_2O
1	0.75	41.78	22.09
2	1.00	41.78	29.46
3	1.25	41.78	36.83
4	1.50	41.78	44.19
5	1.75	41.78	51.56

The reactive mixture was dissolved in distilled water. The obtained solutions were burned in thermal explosion mode in preheated to 500 °C a muffle furnace in air atmosphere, leading to the formation of a fluffy powder, which was rapidly cooled in air. All powders formed during the combustion had foam-like microstructure (Fig. 2, JSM-5610 LV scanning electron microscope with an EXS JED-2201 JEOL chemical analysis system, Japan). The X-ray phase analysis showed that the sample had corundum phase.

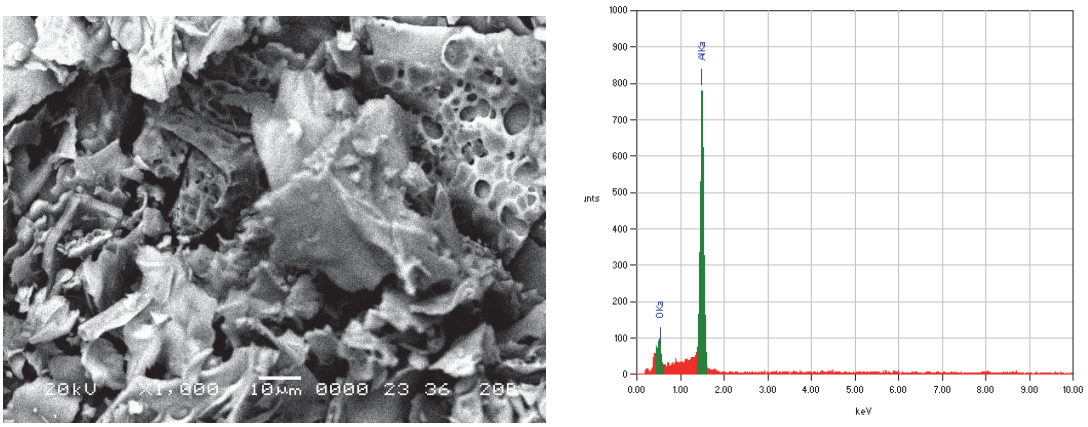

Figure 2 – SEM images and EDS spectrum of the powder No. 1

The prepared powders were mixed in ratio of hydroxyapatite : corundum – 5 : 1 – 20 : 1 and moistened. Moisture content of the obtained ceramic compound was not more than 25–30 wt. %. The samples were prepared by molding. After drying at 80 °C it was fired at 1200 °C. The physical and chemical properties of the calcium phosphate ceramics were the following: water absorption – 8.2–51.4 %, apparent porosity – 19.1–62.3 %, bulk density – 1120–2520 kg/m^3.

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