

BIOMATERIALS

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CERAMICS BASED ON CALCIUM HYDROXYAPATITE SYNTHESIZED FROM CALCIUM ACETATE, CALCIUM HYDROXIDE, AND POTASSIUM HYDROPHOSPHATE

T. V. Safronova,^{1,3} S. A. Korneichuk,¹ V. I. Putlyaev,¹ and V. K. Krut'ko²

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Calcium hydroxyapatite (HAP) powder was synthesized from a water solution of potassium hydrophosphate and a suspension of calcium hydroxide in a water solution containing calcium and potassium ions, hydroxide ions, and acetate ions. The $\text{Ca}(\text{OH})_2$ amount in the calcium containing component was 50, 75, and 100%. The HAP powder synthesized was used to produce ceramic. After kilning at 700°C the density of the ceramic obtained from the HAP powder containing a by-product of the reaction was 2.9–2.6 g/cm³ and bending strength 35–45 MPa with grain size 200 nm. The phase composition was represented by HAP and calcium-potassium double orthophosphate. After kilning at 1200°C the density of the HAP-based ceramic made from powder with no by-product of the reaction was 2.2–2.3 g/cm³ and the bending strength was 45–55 MPa with grain size 500–1000 nm.

Key words: hydroxyapatite, ceramic, sintering, potassium acetate, potassium carbonate.

Hydroxyapatite $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ (HAP) is a promising biomaterial, widely used in medicine. At the present time methods for synthesizing HAP powder as well as for obtaining ceramic based on it are being actively improved. The effect of different substances (additives), introduced into the powered material at different stages of the production of a ceramic, on the formation of the microstructure of the ceramic based on this calcium phosphate is under study.

The most widely used and convenient method of obtaining HAP powder is precipitation from solutions, since this is the conventional way to obtain sinter-active powders. Methods of obtaining HAP powder using water solutions of compounds containing phosphate ions or calcium ions are well known. Phosphoric acid or soluble phosphates of ammonia, potassium, and sodium are used as the initial compounds containing phosphate ions. Soluble salts are used as the calcium compounds: nitrate, chloride, and acetate (RF Patent No. 2038293, USSR Inventor's Certificate No. 710928, RF Patent No. 2050317, USSR Inventor's Certificate No. 1450852).

In the course of synthesis HAP adsorbs ions present in solution, so that in this method of synthesis the powder can contain a substantial amount of byproduct from the reaction (BPR) [1, 2, 3]. The ions present in the reaction zone can also be incorporated into the HAP crystal lattice, affecting the structure of the crystal and the morphology of the particles [4]. HAP powders obtained from different reagents will have different properties, since each combination of reagents possesses unique properties.

After synthesis HAP powders synthesized from calcium nitrate can contain ammonium, potassium, and sodium nitrates. Washing the BPR out of the HAP powder does not remove these salts completely because the HAP nanopowder traps the BPR by different mechanisms. As a result, because nitrate ions are present, the powders remain caustic to living tissues. HAP powders synthesized from calcium chloride can contain ammonium, potassium, and sodium chlorides. Even in this case the BPR cannot be completely removed by washing. Sodium and potassium chlorides present in mammalian tissues are regarded as biocompatible, and in certain definite concentrations do not harm living tissues. HAP powders synthesized from calcium acetate can contain ammonium, potassium, and sodium acetates. After washing, the presence of some potassium or sodium acetates in the HAP powder like-

¹ M. V. Lomonosov Moscow State University, Moscow, Russia.

² Institute of General and Inorganic Chemistry of the National Academy of Sciences of Belarus, Minsk, Belarus.

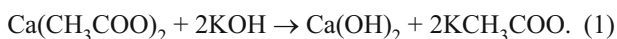
³ E-mail: t3470641@yandex.ru.

wise will not lead to undesirable consequences. We use sodium chloride in food. Potassium chloride, potassium acetate, and sodium acetate are components of some medicinal preparations. When ceramics are obtained from HAP powders synthesized from soluble calcium salts and soluble phosphates, BPR such as ammonium nitrate and ammonium acetate will decompose during kilning. BPR containing sodium or potassium compounds will have a strong effect on the sintering of the ceramic as well as on the formation of the phase composition and microstructure of the ceramic [5].

The effect of successive decanting of solutions of calcium acetate, potassium hydrophosphate, and potassium hydroxide on the properties of HAP powder and the microstructure of a ceramic based on these powders was studied in [6]. Of the components indicated the predominant component of the BPR in HAP synthesis was potassium acetate, which heating converted into potassium carbonate. Because potassium hydroxide was used to maintain a prescribed pH in the HAP synthesis reaction KOH was present in the BPR, since an amount of this compound higher than that calculated according to the reaction had to be used. As a result of the presence of potassium carbonate and potassium hydroxide in the HAP-based powder blank melt formed during sintering, facilitating the attainment of maximum density at 700°C. The decomposition of carbonates at temperature above 700°C had the effect of decreasing the density, which was minimal at 1100°C. Carbonates are introduced into the initial mix in order to obtain porous inorganic materials [7]. The carbonate ion CO_3^{2-} can become incorporated into the HAP structure during synthesis in air [8]. On heating above 700°C such HAP decomposes with carbon dioxide being released; this likewise promotes the formation of porosity.

When in HAP synthesis first potassium hydroxide and then a water solution of potassium hydrophosphate are added to a water solution of calcium acetate, the calcium-containing system before the addition of potassium hydrophosphate consisted of a $\text{Ca}(\text{OH})_2$ suspension in a water solution containing potassium and calcium ions as well as acetate and hydroxide ions.

When KOH is added to a solution of calcium acetate as a pH regulator and a source of OH groups necessary for HAP synthesis, calcium hydroxide is formed in the reaction



In the case where the calcium-containing component consisted of a suspension of the low-soluble compound $\text{Ca}(\text{OH})_2$ in a solution containing acetate ions, hydroxide ions, potassium ions, and calcium ions the post-synthesis HAP powder contained two times more adsorbed products than in the case where KOH was added in the same amount to a potassium hydrophosphate solution. The mass loss of HAP powder synthesized from suspension was 11%⁴ at 200°C versus 6% for HAP powders synthesized from ho-

mogenous solutions, while at 500°C it reached 17% versus 11%. A higher content of adsorbed BPR could be an indication that the specific surface area of the synthesized powdered product is larger and correspondingly the particles formed (individual crystallites) are smaller. This is why it is necessary to pay close attention to synthesis where HAP is synthesized from a calcium-containing component consisting of freshly prepared suspension of the low-soluble compound $\text{Ca}(\text{OH})_2$ in a solution containing acetate ions, hydroxyl ions, hydroxide ions, potassium ions, and calcium ions.

Different methods of HAP synthesis where the calcium-containing component is a suspension of a low-soluble compound are examined in published articles and patents. The low-soluble calcium compounds can be brushite $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, monetite CaHPO_4 (US patent No. 5427754), pyrophosphate $\text{Ca}_2\text{P}_2\text{O}_7$ [9, 10], gypsum dihydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ [11], calcium carbonate CaCO_3 (US patent No. 4938938, international patent WO 88/07498), calcium hydroxide $\text{Ca}(\text{OH})_2$ [12], calcium oxide CaO (RF patent No. 2077475, US patent No. 5217699, international patent WO 94/20416) [10]. It should be noted that when a water suspension of a low-soluble compound is used calcium is present in the form of the ion Ca^{2+} and in the form of a precipitate of an undissolved compound. At some stage of synthesis the Ca^{2+} concentration in solution will be constant. Entering into a reaction, Ca^{2+} will be depleted from the solution, which will create conditions for the as yet undissolved compound, for example, calcium hydroxide, to dissolve.

The present article is a continuation of the work presented in [6] and examines the properties of powders synthesized using a Ca-containing component, consisting of a suspension of $\text{Ca}(\text{OH})_2$ in a water solution, including calcium and potassium ions, hydroxide ions, and acetate ions. Some aspects of obtaining a ceramic based on these powders are also examined.

The objective of the present work is to investigate the properties of HAP powder synthesized from calcium acetate, potassium hydroxide, and potassium hydrophosphate as well as the production of ceramics based on this powder, where the BPR interacts with the main phase.

In the work presented here, the content of the low-soluble compound $\text{Ca}(\text{OH})_2$ in suspension was varied, using different amounts of KOH at the first stage of synthesis, which is reflected by the reaction (1). Three syntheses in which potassium acetate was added to different amounts of potassium hydroxide were performed. The $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component before HAP synthesis, i.e., before potassium hydrophosphate was added, was 50, 75, and 100%, respectively (Table 1). In all three cases the amount of the hydroxide ions was greater than required for HAP synthesis and secured pH sufficient for the formation of stoichiometric HAP. In the syntheses conducted the pH was 12, given that HAP formation occurs preferentially at $\text{pH} > 6$ and is guaranteed at $\text{pH} = 9$. For $\text{pH} < 6$ there is a possibility that other calcium phosphates will form with $\text{Ca}/\text{P} < 1.67$.

⁴ Here and below, content by weight unless stipulated otherwise.

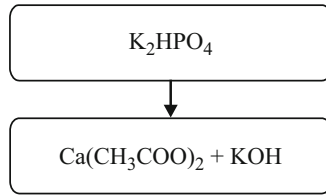
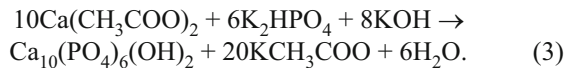


Fig. 1. Scheme used in this work for synthesizing HAP; pH = 12 in the reaction zone.

HAP powder was synthesized in accordance with the scheme shown in Fig. 1. First, 1 liter of 0.5M calcium acetate solution was formed, after which KOH was added at 60°C. Next, 1 liter 0.3 M solution of potassium hydrophosphate was added to this solution in drops over 30 min.

Since the calcium-containing component consisted of calcium hydroxide and calcium acetate, the processes occurring during synthesis can be formally represented by the reactions



The interaction via the reaction (2) could have proceeded with the participation of the calcium ion in solution as well as on the surface of the calcium hydroxide particles. Synthesis proceeded under nonequilibrium conditions and made it possible to obtain active HAP powder. At present it is impossible to determine which of the reactions was preferential in the sense of obtaining a more active (disperse) powder.

As noted in [13], because of adsorption the presence of acetate ions can impede the growth of HAP particles.

The HAP suspension obtained after the solutions were decanted was held in the mother liquor for 30 min. The residue was filtered on a paper filter on a vacuum jet pump. The filtered residue was dried in a thin layer at 20°C. Ten grams of each synthesized powder was freed from the BPR by washing five times with 200 ml of distilled water. The product obtained was disaggregated in acetone in a ball mill with the ratios acetone : powder : balls = 1 : 1 : 3. The disaggregated powder was dried at 20°C for 2 h and passed through a sieve with 200 μm cells. The isothermal soaking method was

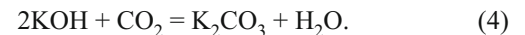
used to study the evolution of the structure of the powder blank. Rod-shaped samples with the dimensions 3 × 5 × 10 mm and approximate mass 0.2 g compacted under pressing pressure 100 MPa were used for this study. The samples were heated to different temperatures in the interval 600 – 1200°C at the rate 5 K/min and soaked at the final temperature for 6 h. One sample was used for every point in this case. The strength of ceramics based on HAP powder containing BPR was determined after kilning at 700°C and 1100°C and soaking at the indicated temperature for 6 h. The strength of ceramic based on no-BPR HAP powder was determined after kilning at 1200°C with soaking at this temperature for 6 h. The strength was calculated from the relation

$$\sigma = \frac{3LP}{2h} a,$$

where L is the distance between supports, P is the fracture force applied to the sample, h is the height of the sample, and a is the width of the sample.

To determine the relative density and measure the strength the samples were made in the form of 4 × 4 × 40 mm rods under specific pressing pressure 100 MPa. For each point, measurements were performed on five samples.

The XPA data (measurement interval 2 – 60° for 2θ) showed that stoichiometric HAP was obtained as a result of all syntheses performed (Fig. 2). Other compounds, specifically, the components of the BPR (potassium acetate and potassium hydroxide), are not marked in the figure. The potassium acetate peak corresponds to 2θ of the order of 10°, which falls outside the measurement interval presented. Owing to the substantial specific surface area of HAP (of the order of 30 – 50 m²/g) the conditions for adsorption of ions present in the mother liquor in adequate quantity — K⁺, CH₃COO²⁻, CO₃²⁻, and OH⁻ — are extant. The carbonate ion is formed as a result of alkali solutions absorbing carbon dioxide gas from air



Thus, on the basis of the ionic composition of the mother liquor potassium acetate, potassium carbonate, and potassium hydroxide appear in the by-product. The content of

TABLE 1. Conditions of Synthesis

Sample No.	Ca ²⁺ content in the Ca-containing component	KOH amount		KOH excess	KOH excess relative to the amount required by the HAP synthesis reaction (3)	Molar composition of the Ca-containing component, %		Ions in mother liquor of the suspension of the Ca-containing component	K ₂ HPO ₄ concentration	Ions in the mother liquor after synthesis
		used in synthesis	required according the reaction			Ca(CH ₃ COO) ₂ in Ca-containing component	Ca(OH) ₂ in Ca-containing component			
1	0.5M	0.5M	0.4M	0.1M	10/8 = 1.25	50	50	Ca ²⁺ , K ⁺ ,	0.3M	K ⁺ ,
2	0.5M	0.75M	0.4M	0.35M	15/8 = 1.88	25	75	CH ₃ COO ⁻ ,	0.3M	CH ₃ COO ⁻ ,
3	0.5M	1M	0.4M	0.6M	20/8 = 2.5	0	100	OH ⁻	0.3M	OH ⁻

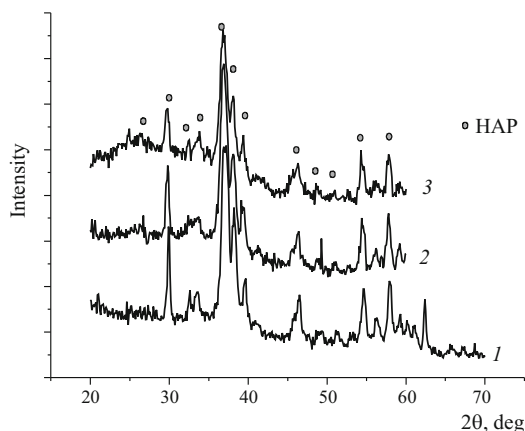


Fig. 2. XPA of powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = n\text{Ca}(\text{OH})_2 / [n\text{Ca}(\text{CH}_3\text{COO})_2 + n\text{Ca}(\text{OH})_2] = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

each BPR component does not exceed 5%, i.e., the limit of sensitivity of XPA, so that the present analysis did not show these compounds. For powders synthesized with calcium hydroxide molar content v in the calcium-containing component equal to 50 and 100 mol.%, the BPR amount is 12–14% based on an estimate of the mass of the synthesized powder after drying and about 11% for powder synthesized with molar ratio of the indicated components 75%. Here

$$v = n\text{Ca}(\text{OH})_2 / [n(\text{Ca}(\text{CH}_3\text{COO})_2 + n(\text{Ca}(\text{OH})_2)],$$

where v is the fraction of calcium hydroxide $\text{Ca}(\text{OH})_2$ in the calcium-containing component, mol.%, and n is the number of moles of matter (calcium hydroxide or acetate).

Granulometric analysis (Table 2) showed that the size of the particles (aggregates) for all powders obtained was 1.14–3.38 μm (FRITCH Analyzette (Germany) particle size analyzer). Scanning electron microscopy showed that the size of an individual crystallite did not exceed 50 nm (LEO SUPRA-50VP Zeiss scanning electron microscope, Germany). In the case of synthesis from solutions or suspensions of low-soluble compounds conditions are created for the formation of particles of such size as a result of primary crystallization.

Table 2 shows some rheological properties of the powders obtained. The decrease of bulk density observed in a series of syntheses could be due an increase of the anisotropy of the particles and an increase of their surface roughness, which prevents closer packing of the particles.

The difference in the values of the bulk density and the pressing density could be due to the difference in the content of the by-product, which can play the role of a plasticizer or technical binder (temporary or irremovable).

Thermogravimetric and differential thermogravimetric analyses (Perkin Elmer Pyris, USA, thermal analyzer) de-

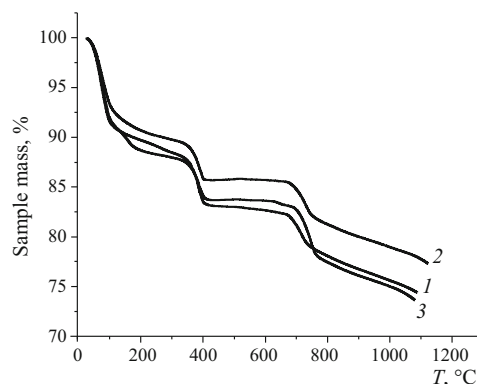


Fig. 3. Mass of HAP powder samples synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR versus the temperature with heating rate 5 K/min. The samples were prepared using powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

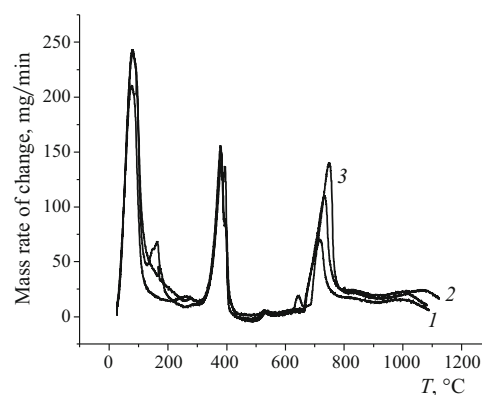


Fig. 4. Mass loss rate for samples of HAP powder synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR versus temperature with heating rate 5 K/min. The samples were prepared from powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

tected mass losses to 25% with heating to 1150°C, proceeding in several stages (Figs. 3 and 4). The first stage (6–10%, to 150°C) can be associated to removal of adsorbed water and acetone, since disaggregation of the powders was con-

TABLE 2. Some Properties of the Synthesized Powders

Sample No.	Size of individual particles according to SEM data, nm	Most likely size of aggregates, μm	Bulk density, g/cm^3	Compact density, g/cm^3	Mass loss at 600°C, %
1	20–30	3.83	0.51	1.49	17.4
2	20–30	2.42	0.43	1.52	14.3
3	20–30	1.14	0.37	1.41	16.4

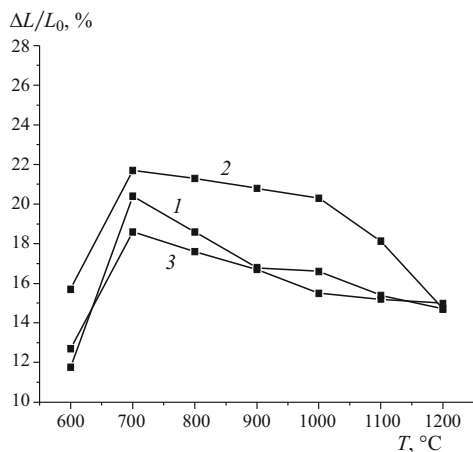
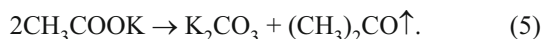


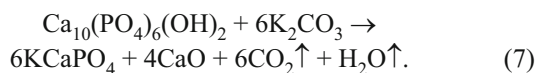
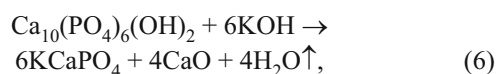
Fig. 5. Length shrinkage of samples versus the kilning temperature and soaking at the final temperature for 6 h. The samples were prepared from powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$; ΔL) change of length; L) sample length before kilning.

ducted in acetone. At the second stage potassium acetate decomposed into potassium carbonate and acetone (3.5 – 5%):

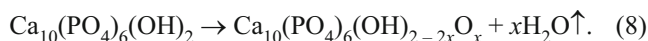


This stage is observed in the temperature interval 300 – 400°C, which is in agreement with the published data on the decomposition of potassium acetate.

After 600°C HAP interacts with potassium hydroxide and potassium carbonate K_2CO_3 with formation of renanite-type double potassium-calcium orthophosphate:



In addition, the mass loss in this temperature interval could be due to partial dehydration of HAP with formation of oxy-hydroxyapatite (8):



The simultaneous presence of calcium phosphate and potassium compounds creates the conditions for the formation of melt in the system $\text{CaO}-\text{P}_2\text{O}_5-\text{K}_2\text{O}$. It should be noted that the lowest temperature at which a eutectic melt forms in the indicated system is 686°C [14].

Dilatometry (DIL-402C, Netzch, Germany) shows that the shrinkage onset temperature for powders synthesized via the interaction of calcium acetate and potassium hydrophosphate lies in the interval 200 – 300°C. The shrinkage rate for powders is highest in the temperature interval 500 – 700°C, which could be due to the melting of potassium

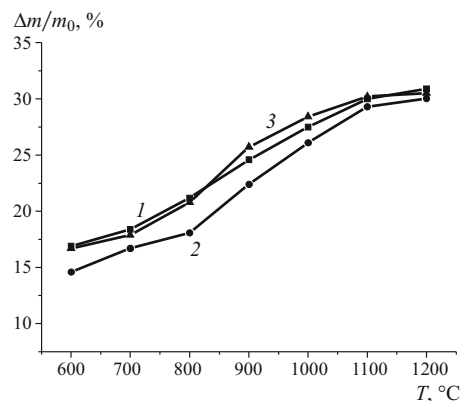


Fig. 6. Mass change of samples versus the kilning temperature and soaking at the final temperature for 6 h. The samples were prepared using powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$; Δm) mass change; m) mass before kilning.

carbonate or low-temperature eutectic melt in the system formed by the BPR or products of thermal decomposition of the BRP components: potassium acetate ($T_{\text{melt}} = 292^\circ\text{C}$), KOH ($T_{\text{melt}} = 405^\circ\text{C}$), and potassium carbonate ($T_{\text{melt}} = 894^\circ\text{C}$).

The greatest shrinkage 22% occurring during kilning in the interval 600 – 1200°C with isothermal soaking for 6 h was observed at 700°C for a sample synthesized with molar content of calcium hydroxide in the calcium-containing component 75% (Fig. 5). The shrinkage was 20% or powders with $v = 50\%$ and 18% for $v = 100\%$. The powders obtained with $v = 100\%$ show the lowest shrinkage. Thus, the dependence of the shrinkage on the value of v is nonmonotonic with a maximum at $v = 75\%$. This could be due to the particulars of the BPR composition formation with a change of the composition of the mother liquor due to an increase of the amount of calcium hydroxide $\text{Ca}(\text{OH})_2$ and an increase of the number of potassium ions in the mother liquor.

The decrease of the shrinkage of the samples at higher temperatures (800 – 1200°C) can be explained by the decomposition of potassium carbonate K_2CO_3 , which is present in the powdered blank. The samples kilned at these temperatures were transparent. Swelling of the samples was observed in the same temperature interval. The mass losses for all samples after kilning at 1200°C for 6 h were about 30% (Fig. 6).

The density of the samples made from powders synthesized with different $\text{Ca}(\text{OH})_2$ content in the calcium-containing component grew to 2 – 2.5 g/cm^3 with kilning temperature increasing to 700°C and then decreased to 0.8 – 1.0 g/cm^3 with a further increase of the kilning temperature to 1100°C (Fig. 7).

The relative density of the samples after kilning at 700°C for 6 h was 60 – 80%, increasing with $\text{Ca}(\text{OH})_2$ content in the calcium-containing component (Fig. 8). The relative den-

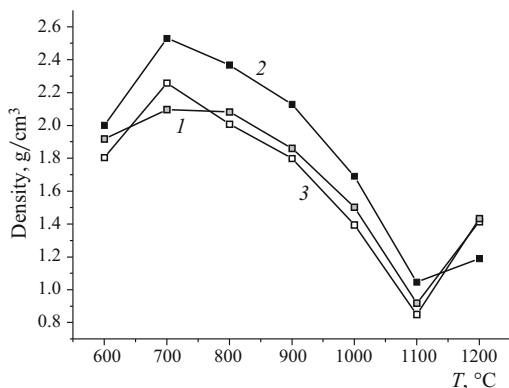


Fig. 7. Density of samples versus the kilning temperature and soaking at the final temperature for 6 h. The samples were prepared using powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

density ρ_{rel} (%) of the HAP ceramic was calculated from the relation

$$\rho_{\text{rel}} = \frac{\rho}{3.16} \times 100,$$

where ρ is the density of the samples and 3.16 is the theoretical density of HAP.

The increase of the $\text{Ca}(\text{OH})_2$ content in the calcium-containing component was determined by the conditions of synthesis and was due to an increase of the amount of KOH added to the calcium acetate. An increase of the number of potassium ions in the mother liquor unavoidably results in an increase of the potassium compounds adsorbed on the surface of the particles of the synthesized HAP powder.

Electron microscopy confirms that when the powders used are synthesized with different $\text{Ca}(\text{OH})_2$ content in the calcium-containing component the ceramics obtained are dense at 700°C and porous at 1100°C . The grain size was 200 nm for a dense ceramic and 500 nm for the porous ceramic.

The adsorbed BPR, uniformly distributed in the synthesized powder, can be viewed as a sinter-regulating additive. At 700°C , i.e., below the onset temperature of the decomposition of carbonates, a denser ceramic with uniform microstructure and small grains can be obtained because of the relatively low kilning temperature and the action of low-temperature melt, whose composition presumably lies in the system $\text{K}_2\text{O}-\text{CaO}-\text{P}_2\text{O}_5$ or $\text{CaO}-\text{P}_2\text{O}_5-\text{K}_2\text{CO}_3$.

The XPA data for samples obtained from no-BPR powders kilned at different temperatures confirm that the conditions adopted for synthesis yield stoichiometric HAP. After kilning at 700°C a BPR-containing ceramic contains, together with the HAP phase, KCaPO_4 and CaO (the latter is clearly recorded at 1100°C). The presence of CaO and potassium renanite KCaPO_4 can create an alkaline medium with resorption of the ceramic in the organism; for this reason, the

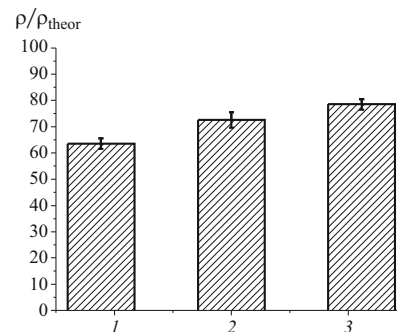


Fig. 8. Relative density of samples after kilning at 700°C for 6 h. The samples were prepared using powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$; ρ) real density of HAP; ρ_{theor}) theoretical density of HAP, equal to 3.16 g/cm^3 .

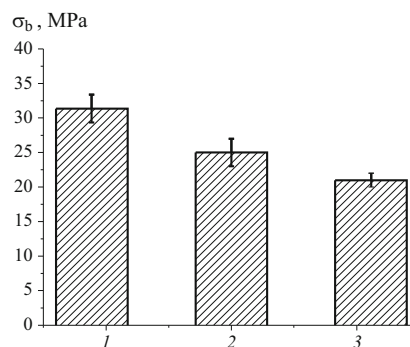


Fig. 9. Ultimate bending strength of samples after kilning at 700°C for 6 h. The samples were prepared using powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and containing BPR with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

amount of BPR remaining in the powder must be limited. On the other hand, the presence of BPR leads to the formation of a melt and transforms sintering into the liquid phase regime, making it possible to attain a definite density and porosity as a result of CO_2 release from the melt at temperatures no higher than 1000°C .

The strength of ceramic prepared from BOR-containing HAP powders kilned at 700°C and possessing the maximum density was 35 – 45 MPa. The strength is observed to decrease with increasing $\text{Ca}(\text{OH})_2$ content in the calcium-containing component of the HAP-yielding reaction (Fig. 9).

The bending strength of ceramics prepared from BPR-containing HAP powders kilned at 1200°C and possessing minimum density did not exceed 5 – 8 MPa.

The bending strength of no-BPR ceramics kilned at 1200°C with relative density 70% (Fig. 10) was 45 – 50 MPa (Fig. 11).

In summary, it has been shown in the present work that synthesis from a suspension of $\text{Ca}(\text{OH})_2$ in a water solution

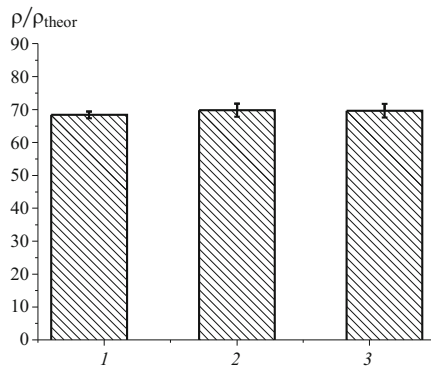


Fig. 10. Relative density of samples after kilning at 1200°C for 6 h. The samples were prepared using no-BPR powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$; ρ) density of HAP sample obtained; ρ_{theor}) theoretical density of HAP, equal to 3.16 g/cm^3 .

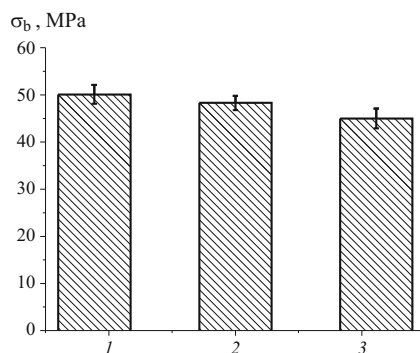


Fig. 11. Ultimate bending strength of samples after kilning at 1200°C for 6 h. The samples were prepared using no-BPR powders synthesized from a $\text{Ca}(\text{OH})_2$ suspension and BPR-containing powders with $\text{Ca}(\text{OH})_2$ molar content in the calcium-containing component: 1) $v = 50\%$; 2) $v = 75\%$; 3) $v = 100\%$.

yields sinter-active HAP powder. The BPR remaining in the synthesized powder makes compaction possible without the addition of a temporary technological binder and also plays the role of an additive that affects the flow of the sintering process and microstructure formation. When ceramic is made from HAP powder containing no BPR the synthesis conditions have virtually no effect on the values of the relative density, but the strength decreases somewhat when the $\text{Ca}(\text{OH})_2$ content in the Ca-containing component increases during synthesis.

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