Supplementary materials - Дополнительные материалы

Синтез порошков аморфного фосфата кальция для изготовления биокерамики и композитов методами 3d-печати

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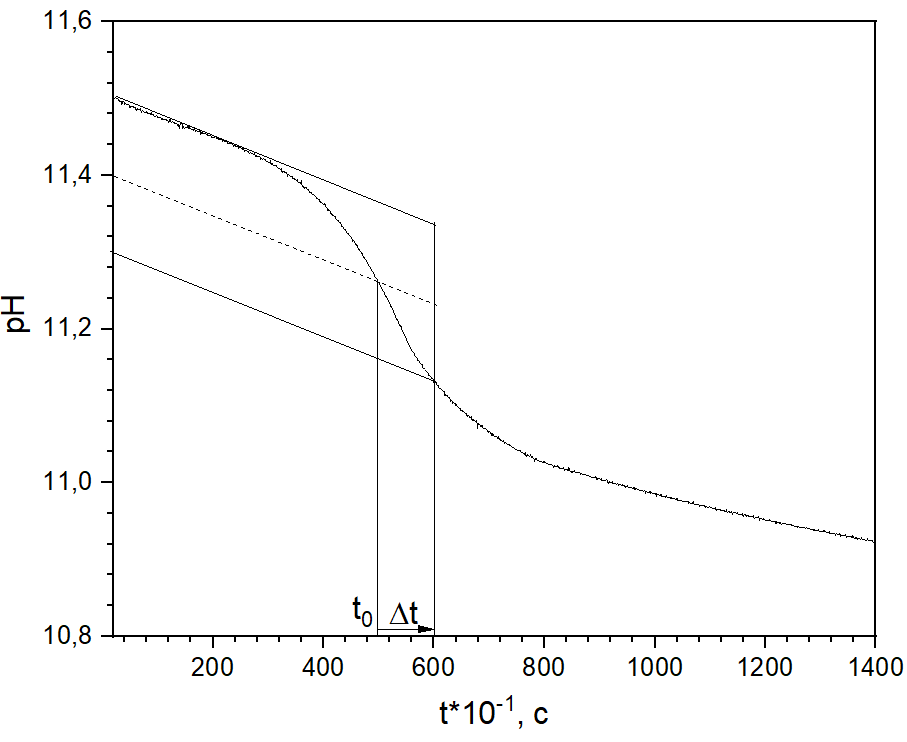
Synthesis of amorphous calcium phosphate powders for the manufacture of bioceramics and composites using 3D printing methods

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Russian Journal of Inorganic Chemistry

Журнал неорганической химии

*Effect of inhibitors on ACP crystallization*



**Fig. S1**. Determination of t0 and Δt.

**Рис. S1**. Определение t0 и Δt

|  |  |
| --- | --- |
| a | b |
| c | d |
| e | f |
| g | h |

**Fig. S2**. Variation of pH with time for the samples: a – ac, b – mg, c – cit, d – pp, e – suc, f – mg+pp, g – tm, h – tp

**Рис. S2**. Изменение pH для образцов: a – ac, b – mg, c – cit, d – pp, e – suc, f – mg+pp, g – tm, h – tp

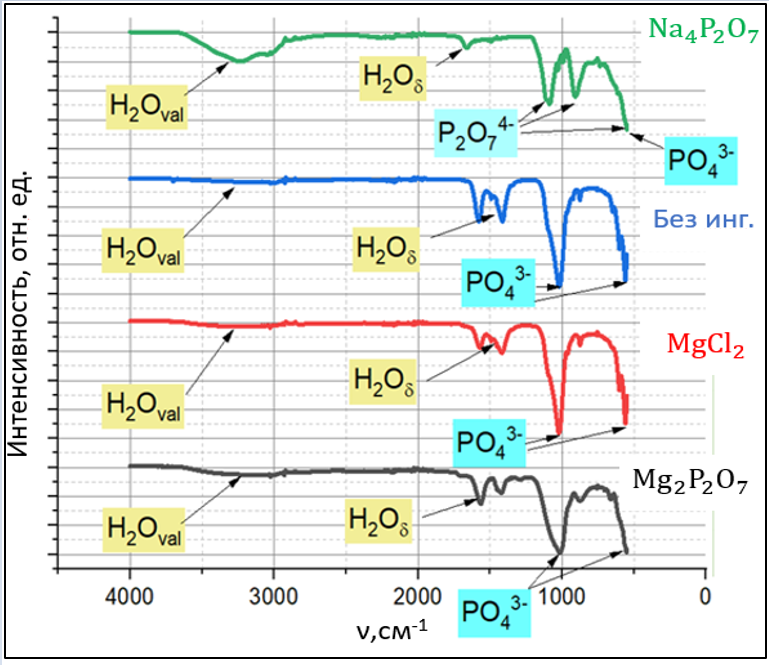
|  |  |  |
| --- | --- | --- |
| a | b | c |
| d    h | f  i | g    j |

**Fig. S3**. Diagrams of predominant ionic forms/solid phases for the systems Са2+ - РО43- - Хn- – Н+ ( [Ca2+]=75 mМ, [PO43-]=50 mМ) lg[X] – рН for Хn-: a – cit3-, b – Р2О74-, c – Р3О105-, d – mal2-, e – P3O93-, f – suc2-, h – PO43-, i – ac-, j – EDTMP8-

**Рис. S3**. Диаграммы преимущественных ионных форм/твердых фаз для систем Са2+ - РО43- - Хn- – Н+ ( [Ca2+]=75 мМ, [PO43-]=50 мМ) в координатах lg[X] – рН для Хn-: a – cit3-, b – Р2О74-, c – Р3О105-, d – mal2-, e – P3O93-, f – suc2-, h – PO43-, i – ac-, j – EDTMP8-

*IR spectroscopy*

IR spectroscopy was carried out to assess the composition of the obtained samples. It can be concluded that for each of the samples, all vibrations corresponding to the phosphates functional groups were found, as well as vibrations caused by the presence of crystal water in the structure. Also, in the first and fourth spectra, the peaks corresponding to pyrophosphate ions can be seen.

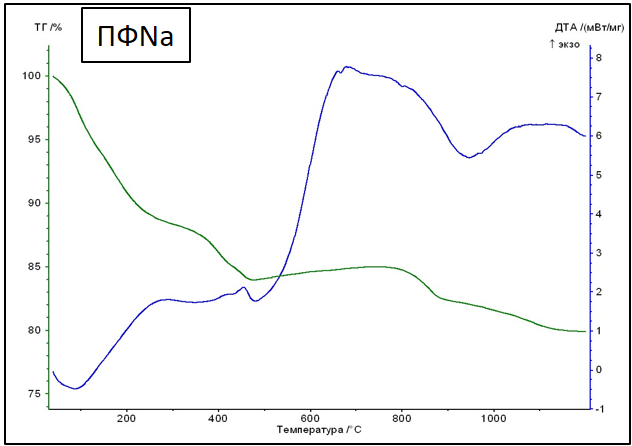
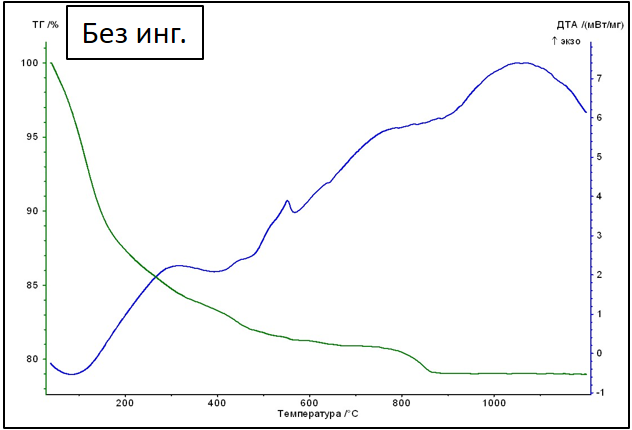


**Fig. S4**. IR spectra of some ACP samples

**Рис. S4**. ИК-спектры некоторых образцов АФК

*Thermogravimetric Analysis*

Using the obtained results, it is possible to determine firing temperature and to formulate recommendations for firing of ceramics after 3D printing. So, e.g., for the first sample (Fig.S5 a), it can be seen that after reaching the temperature of 860-870°C, no further change in mass occurs, and, therefore, no processes (such as dehydration, i.e. removal of crystallization water) go further.



**Fig. S5**. TGA/DTA of АСР samples: a - 0, b – pp

**Рис. S5**. TГ/ДТА кривые образцов АФК a - 0, b – pp

Therefore, it is possible to set the calcination temperature at ≥900°. For the second sample (sodium pyrophosphate as an inhibitor), the end of the change in mass in the case of the second sample occurs at a temperature of about 1200°C.

*SEM data*

After firing, crystallites up to 10 μm in size were formed, in some cases, they have an elongated shape.

**Table S1**. SEM images of the powders before firing

**Таблица S1**. Данные РЭМ порошков до обжига

|  |  |  |
| --- | --- | --- |
| C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\0_50k_3kV123.tif  0 | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\ac_50k_3kV142.tif  ac | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\cit_50k_3kV145.tif  cit |
| C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\mal_50k_3kV137.tif  mal | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\mg pp_50k_3kV132.tif  mg+pp | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\mg_50k_3kV139.tif  mg |
| C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\pp_50k_3kV130.tif  pp | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\suc_50k_3kV125.tif  suc | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\tmp_50k_3kV148.tif  tm |
|  | C:\Users\Envy\Desktop\научка\данные\sem_20_10_21\golubchikov\tpp_50k_3kV153.tif  tp |  |

**Table S2**. SEM images of the powders after firing at 900°C

**Таблица S2**. Данные РЭМ порошков после обжига при 900°C

|  |  |  |
| --- | --- | --- |
| *mg* | *ac* | *cit* |
| *mg+cit* | *suc* | *mg+ac* |