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# Ceramic materials based on magnesium orthophosphate for biomedical applications

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Equipment Description:

Powder X-ray diffraction (PXRD) data were collected on a Rigaku D/Max2500 X-ray diffractometer with a rotating anode (Japan) using $CuK_\alpha$ radiation ($\lambda$ = 1.5418 Å). PXRD data were collected at room temperature over 2$\theta$ range between 2° and 70° with a step interval of 0.02°. The shooting was carried out in quartz cuvettes without averaging rotation. A solvent (acetone) was used to fix the powder samples.

Scanning electron microscope LEO SUPRA 50VP (Carl Zeiss, Germany) was used to study the microstructure of the obtained ceramics. Samples were attached to a copper plate using a conductive carbon adhesive tape and coated with a 25 nm layer of chromium to prevent charging of samples. The accelerating voltage of the electron gun was 2–21 kV. Images were obtained in secondary electrons at magnifications up to 10000x using a SE2 type detector. The cross-section of the material was investigated.

Differential thermal analysis (DTA) was performed using a synchronous thermal analyzer with vertical sample loading (STA 409 PC Luxx, Netzsch, Germany) to assess the behavior of the materials upon heating. The measurements were performed in air at 25–1200°C temperature range with 5°C/min heating rate in alumina crucibles.

Dilatometric analysis was used to measure the linear shrinkage of compressed samples in the form of a tablet. To study the samples by dilatometry, tablets with a diameter of d=8 mm and a height of about 0.2 mm were prepared using a manual press. The studies were carried out in isothermal mode with exposure for 10 hours in isothermal mode to a temperature of 1250°C with a heating rate of 5°/min in a horizontal dilatometer DIL 402 C (Netzsch, Germany).

The resorption kinetics of magnesium orthophosphate ceramic granules was studied on the Excellence T-50 titrator (Mettler-Toledo). The temperature value was maintained at 25°C on the Polystat thermostat. Prepared 0.025 M citric acid solution was added in solution ceramic granules with distilled water until the set pH value was reached. Initially, pallets with 6 mm diameter were

prepared. The weight of each pallet was 0.3 g. Then, the obtained samples were calcinated at 800°C for 10 h. After calcination the samples were milled in a mortar, and ceramic granules were sieved through a polyester sieve Saatilene HiTechTM to obtain uniform particle size.

The Debye–Scherrer's equation is

$D_{hkl} = K\lambda/(\beta_{hkl} \cos \theta)$,

where crystallite size is denoted as $D_{hkl}$, hkl means Miller indices, crystallite-shape factor – K, which is generally peer to 0.9, the wavelength is indicated as λ, $\beta_{hkl}$ is a full-width at half-maximum (FWHM) of an X-ray diffraction peak and a Bragg angle defined as θ. Debye–Scherrer's crystallite size calculations with correction for instrumental broadening were performed using reflections in the 2θ range of 24–41° on PXRD patterns.

a) b)

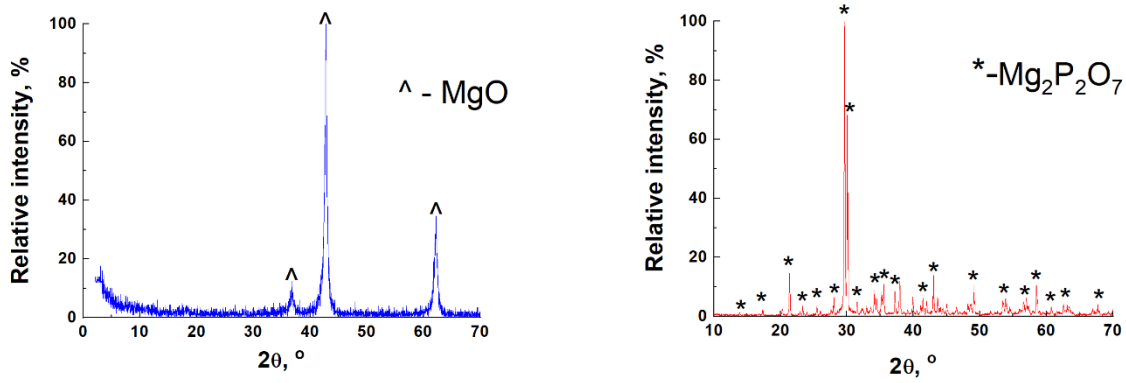


**Figure S1** Diffractograms of powders of magnesium oxide, MgO (a), (PDF-2, 45-946) and magnesium pyrophosphate, $Mg_2P_2O_7$ (b) (PDF-2, 72-19).

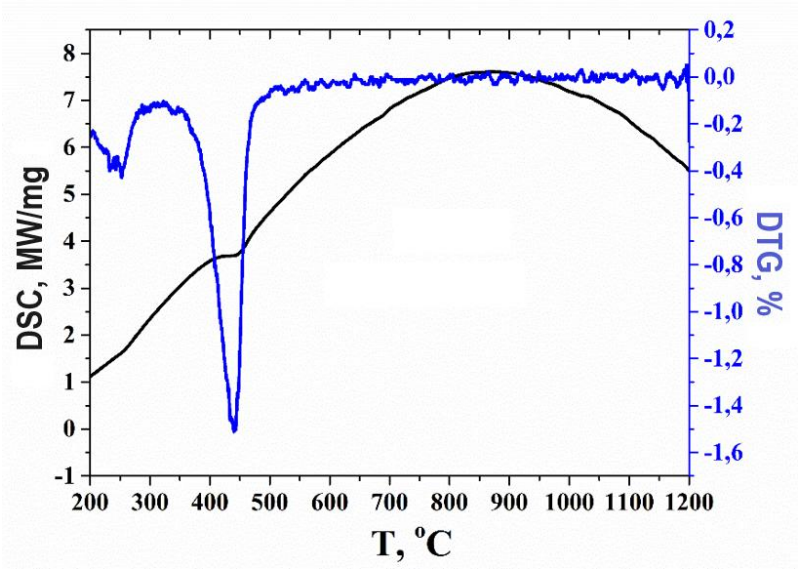


**Figure S2** The results of thermal analysis for a mixture of $4MgCO_3 \cdot Mg(OH)_2 \cdot nH_2O$ and $Mg_2P_2O_7$.

| **Table S1** Shrinkage of ceramics based on $Mg_3(PO_4)_2$ | |
|---|---|
| Heat-treatment temperature, °C | Shrinkage, % |
| 900 | 0.2 ± 0.05 |
| 950 | 2.0 ± 0.4 |
| 1000 | 2.2 ± 0.5 |
| 1150 | 7.6 ± 0.9 |
| 1250 | 10.3 ± 1.2 |