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MATERIALS



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## Synthesis of Zinc Whitlockite by Dissolution-Precipitation Process

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### ABSTRACT

Magnesium whitlockite ( $\text{Ca}_{18}\text{Mg}_2\text{H}_2(\text{PO}_4)_{14}$ ) is the second most abundant mineral in human body and one of the main components of the human hard tissue, constituting to approximately 20–35 wt% [1]. This compound is known for its excellent biocompatibility and osteogenic capability which makes this material a promising candidate for application in bone regeneration [2]. Using other ions instead of Mg could further improve biological properties of the material and make it more appealing for medical usage. Zn substituted calcium phosphates are characterized by enhanced rate of metabolic processes and antibacterial properties which makes these materials very attractive for usage in medicine [3].

In the present work, whitlockite powders with different amounts of Zn ions were synthesized by dissolution-precipitation method using calcium hydrogen phosphate dihydrate and zinc acetate dihydrate as starting materials. Synthesis conditions such as temperature, time and pH were carefully studied and optimized. All synthesized compounds were obtained at low temperatures (75 °C) under atmospheric pressure. Synthesized compounds were analysed by X-ray diffraction, Fourier-transform infrared spectroscopy, scanning electron microscopy, inductively coupled plasma optical emission spectroscopy and Raman spectroscopy. Rietveld analysis was employed for calculations of lattice parameters.

Amount of Zn ions in the crystal structure was varied by varying their ratio with Ca ions in reaction mixture. It was demonstrated that single-phase Zn whitlockite can be synthesized when Ca to Zn ratio in a reaction mixture is in the range from 9 to 20. With an increase of Zn amount in the crystal lattice, decrease of lattice parameters was observed. Also, a significant change in morphology of the particles can be observed when amount of Zn in the crystalline lattice changes. When Zn concentration is low, particles are shaped like hexagonal platelets but with increase of Zn concentration it changes to a rhombic shape. Thermal stability studies revealed that Zn whitlockite is thermally unstable above 500 °C and after heat treatment decomposes to  $\beta$ -tricalcium phosphate and calcium pyrophosphate. As for our knowledge, whitlockite compounds with Zn instead of Mg ions were synthesized for the first time.

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### References

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- 2.H. L. Jang, G. Bin Zheng et al. *Advanced Healthcare Materials*, In Vitro and In Vivo Evaluation of Whitlockite Biocompatibility: Comparative Study with Hydroxyapatite and  $\beta$ -Tricalcium Phosphate, 2015, 5, 128–136.
- 3.V. Fadeeva, M. R. Gafurov et al. *BioNanoScience*, Barinov, Tricalcium Phosphate Ceramics Doped with Silver, Copper, Zinc, and Iron (III) Ions in Concentrations of Less Than 0.5 wt.% for Bone Tissue Regeneration, 2017, 7, 434–438.