CHARACTERISATION OF ZINC WHITLOCKITE SYNTHESISED UNDER HYDROTHERMAL CONDITIONS

Agne Kizalaite, Tomas Murauskas, Aleksej Zarkov

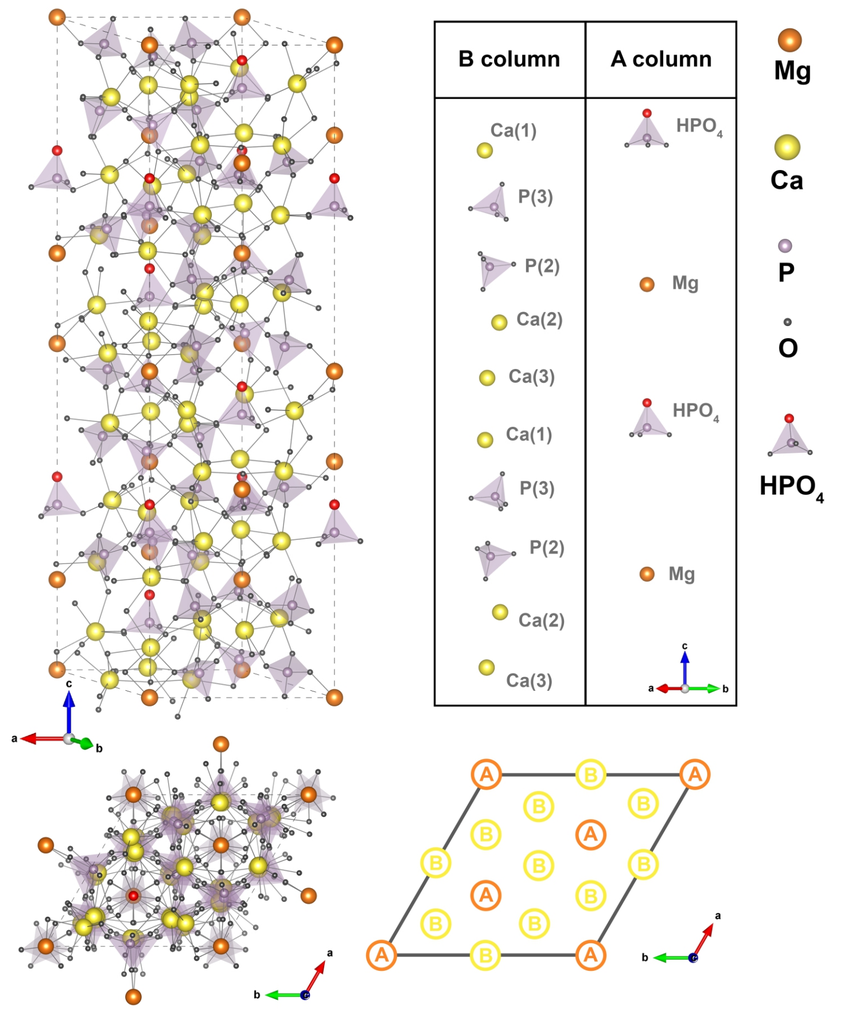
Institute of Chemistry, Vilnius University, Naugarduko g. 24, LT-03225 Vilnius, Lithuania
agne.kizalaite@chgf.vu.lt

Introduction

Magnesium whitlockite ($Ca_{18}Mg_2H_2(PO_4)_{14}$) is one of the major mineral components of human body constituting to approximately 20–35 wt% of human hard tissue [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]. Incorporation of biologically active ions into the whitlockite structure could result in superior biological performance and expanded clinical application of the material. One of the potential substituents in whitlockite is Zn which can enhance the rate of metabolic processes and give antibacterial properties to calcium phosphates [3]. These properties can accelerate bone regeneration processes and decrease the infection rate.

Experimental

In the present work, whitlockite powders with different amounts of Zn ions were synthesized using calcium hydrogen phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$) and zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) as starting materials. Synthesis conditions such as temperature, time and pH were carefully studied and optimized. Dissolution-precipitation process was used for the synthesis: starting materials were dissolved in a mixture of water and phosphoric acid, then the pH of the solution was increased which induced the formation of low crystalinity precipitates. All synthesized compounds were obtained at 200 °C temperature under hydrothermal conditions. Synthesized compounds were analysed by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy and X-ray photoelectron spectroscopy (XPS).



Results

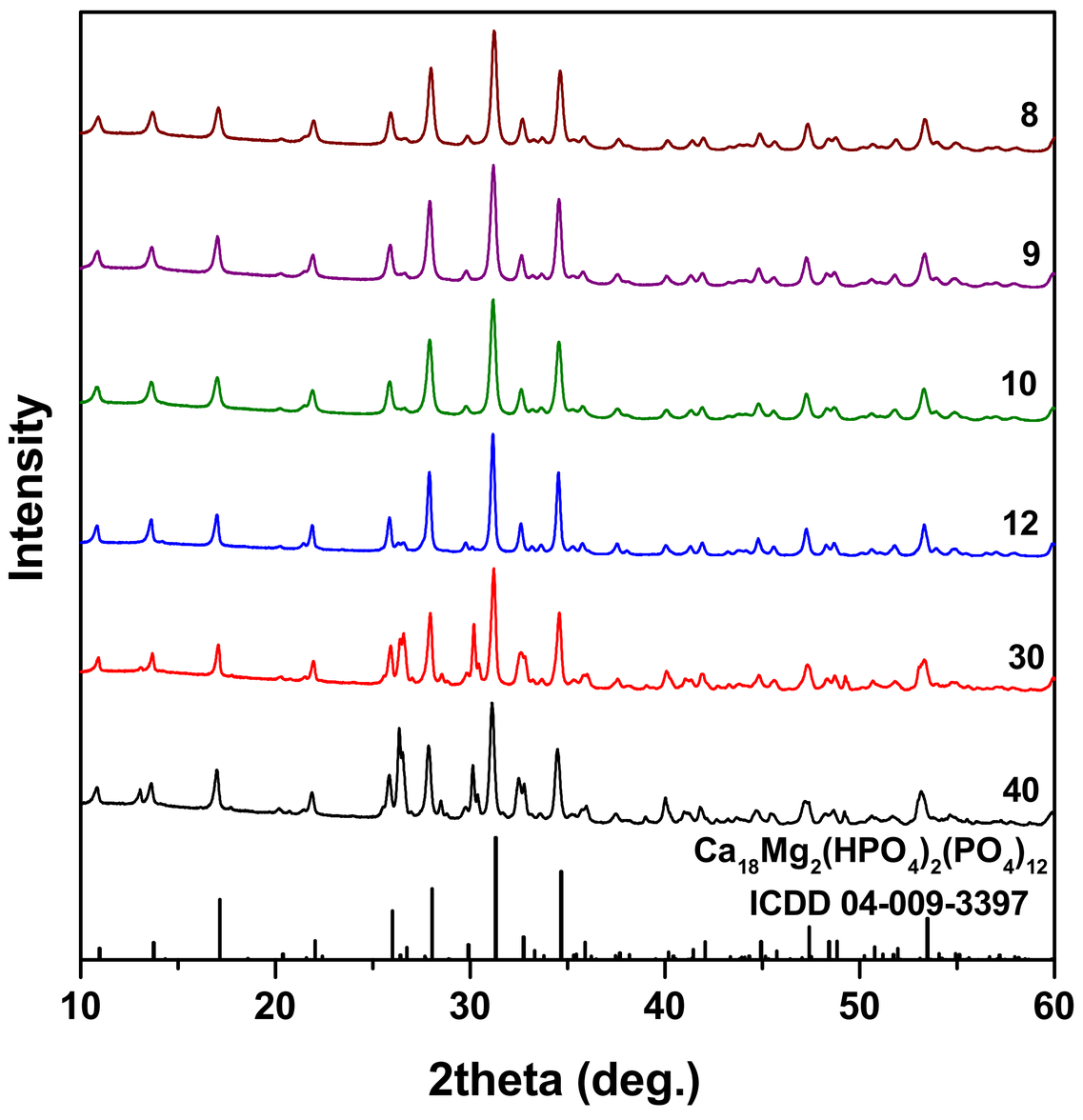


Fig. 1. XRD patterns of whitlockite powders with different Ca/Zn molar ratios.

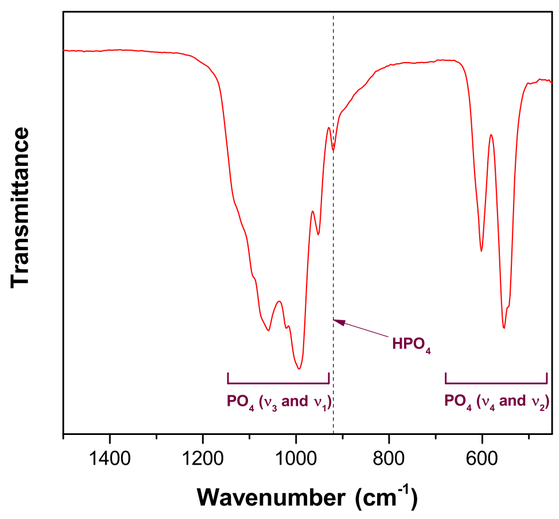


Fig. 2. FTIR spectra of synthesized whitlockite powder.

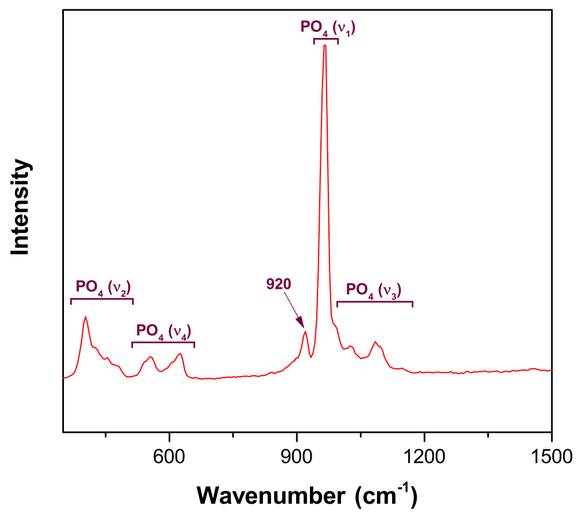


Fig. 3. Raman spectra of synthesized whitlockite powder.

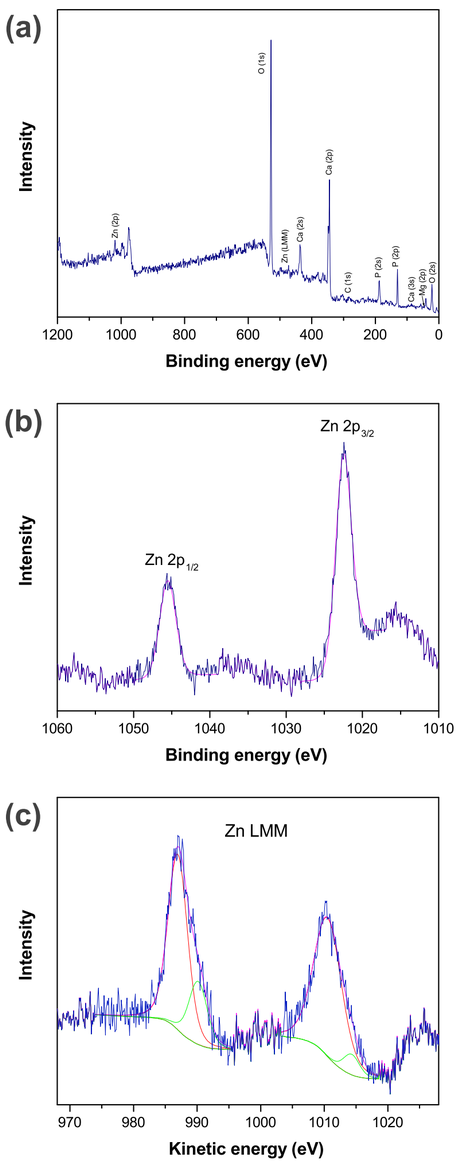


Fig. 4. XPS Survey spectra (a) of Zn whitlockite, high-resolution spectra and fitting results of Zn 2p peaks (b) and Auger Zn_{LMM} spectra (c).

Conclusion

It was demonstrated that single-phase Zn whitlockite with different amount of Zn ions in the crystaline lattice can be obtained. However, the amount of Zn in the final compound could only be varied in a very narrow range when Ca to Zn ratio in a reaction mixture is between 8 and 10. FTIR and Raman spectrum confirmed the formation of whitlockite structure. XPS analysis showed that positions of peaks, as well as the value of the modified Auger parameter of 2010 eV correspond to the Zn^{2+} chemical state. The (Ca+Zn)/P ratio for the material calculated from XPS data is 1.35, which is slightly lower that the expected theoretical value of 1.43 in stoichiometric WH.

References

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2. H. L. Jang et al. Advanced Healthcare Materials 5(1) p. 128-136 (2016).
3. I. V. Fadeeva et al. BioNanoScience 7(2) p. 434-438 (2017).

Acknowledgement

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