Poster/Oral Presentation
Date of presentation
Time of presentation

Studies of pristine and dissolved Nb-doped hydroxyapatite prepared via low-temperature mechanochemical method

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Calcium-phosphate based materials play a crucial role in the tissue engineering development. They exhibit high biocompatibility, excellent ability to undergo varying degrees of resorbability[1]as well as relatively high bioactivity and due to that they are commonly used as implants. A great effort is devoted to improve their physico-chemical properties by for example tuning their degree of crystallinity and doping them with metal ions is one of the most prominent strategies. The results found in the literature show that synthesized niobium-doped calcium-phosphate compounds exhibit a good biocompatibility, very low cytotoxicity in respect to calcium-phosphates doped with other metals and additionally can enhance human osteoblast function[2,3]. As of today, the structure of these materials is not fully described.

In this work, we present the results of systematic studies of niobium-doped hydroxyapatite of general composition $77{\cdot}CaO\text{-}(23\text{-}x){\cdot}P_2O_5\text{-}x{\cdot}Nb_2O_5$, where x = 0.00; 2.30; 4.60 (which corresponds to 0, 10 and 20 mol % of phosphorous, respectively), prepared via low-temperature mechanochemical method. Subsequently, the materials were dissolved in deionized water at 37°C for 4 weeks.Then, a detailed, systematic structural investigation by means of, i.e. FTIR spectroscopy, SEM imaging, EDX, XRD, XPS and XAS spectroscopy, was realized to shed light on the relationship between materials structure as well as dissolution and the presence of dopant.

The incorporation of niobium was confirmed by EDX and XRD. However, the amount of dopant turned out to be less than the target amount.XPS analysis showed that Nb on the surface of materials has a valence of +5 predominantly.The grain size of nanoceramics increased with the addition of niobium. On the other hand, the presence of Nb decreased the crystallinity and the size of the crystallites.XAS measurements revealed that the phosphorus chains in the tested samples are short, while the calcium on their surface is in the unstable +1 oxidation state.Moreover, the results showed that the surface of the materials had a different composition than the volume.The effect of the dopant on the dissolution process was also observed.

The presented systematic analysis of niobium-doped hydroxyapatite confirms the incorporation of the dopant into the structure and its influence on the structural properties of nanoceramics and their susceptibility to bioresorption. The obtained results will help to better understand the structure of this group of materials and link it with their biological properties.

Acknowledgements: This work was developed under the provision of the Polish Ministry of Education and Science project: "Support for research and development with the use of research infrastructure of the National Synchrotron Radiation Centre SOLARIS" under contract nr 1/SOL/2021/2. We acknowledge SOLARIS Centre for the access to the Beamline PEEM/XAS, where the measurements were performed and dr Marcin Zając for technical support.

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