26th Congress of SCTM

Sept. 20-23, 2023, Metropol Lake Resort, Ohrid, N. Macedonia

Morphological and Thermal Characteristics of the Mechanochemicaly Activated Calcium Phosphates

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Mechanochemical activation is an effective method for obtaining uniform, nano-sized and surface-activated powders. It increases the energy of the system, affecting the morphology of the particles, and hence the thermal properties of the materials and often leads to the formation of new phases.

The effect of the revolutions and duration of the mechanochemical activation on the properties of equimolar mixtures of $Ca(OH)_2$ and $CaHPO_4$ was investigated in order to clarify the nature of ongoing processes. Experiments were performed in FRITSCH 6 ball mill with 300, 450 and 600 rpm and duration of 5, 11, 24, 48 and 120 h.

The results show different behavior of the two starting substances. The $CaHPO_4$ particles considerably decrease in size with increasing revolutions and grinding time (from 89.9 nm to 24 nm at 300rpm (120 h); to 28.8 nm at 450 rpm (24h) and to 42.2 nm at 600rpm (11h)), which leads to a decrease in the temperature effects of its phase transformations in the DTA curves. For $Ca(OH)_2$ the effect is less pronounced. A new phase of nonstoichiometric poorly crystalline hydroxyapatite was identified at 120 h at 300 rpm, at 24 h at 450 rpm, and at 5 h at 600 rpm. Higher revolutions create conditions for obtaining more and larger defects in the crystal structure of $CaHPO_4$, which accelerates the transformation to hydroxyapatite. Identical fragments in the structures of $CaHPO_4$ and $Ca_5(PO_4)_3OH$, stimulates additionally the phase transformation. The amount and crystallinity of hydroxyapatite increases with increasing milling time and reaches 94% after 48 h activation at 600 rpm, a quantity that no longer changes.

Keywords: mechanochemical activation, calcium phosphate, morphological characteristics, thermal properties

Acknowledgements: The authors thank the Bulgarian Ministry of Education and Science for the financial support under the projects: KP-06-H49-6/2020 and D01-272/02.10.2020 "European Network on Materials for Clean Technologies".