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Solvent-Free Green Synthesis of $Cu_{0.5}Ti_2(PO_4)_3$ and $Cu_{0.5}TiO(PO_4)$ NASICON-Type Compounds from Ilmenite Mineral Sand

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Abstract

Ilmenite is a naturally available black or steel-gray color mineral, called titanium iron oxide, $FeTiO_3$. This valuable mineral comprises a significant amount of titanium that can be utilized as a titanium source to synthesize various materials. This study focused on synthesizing copper titanium phosphate compounds via an eco-friendly solid-state reaction route. α-titanium bismonohydrogen orthophosphate monohydrate (α-$Ti(HPO_4)_2.H_2O$/α-TiP), derived from beach sand ilmenite used as the starting material for the synthesis. α-TiP was obtained by digesting ilmenite with 85 wt% phosphoric acid under reflux conditions. The XRD pattern of the obtained white powder confirmed the formation of α-TiP. The synthesized α-TiP and copper (II) acetate were ground with different molar ratios (1:2, 1:1, and 2:1) until it forms a fine powder following a solvent-free solid grinding method. These powder mixtures were calcined at 800 °C for 4 hours utilizing a muffle furnace. The obtained solid solutions developed green and two different shades of yellow color. The samples were characterized by XRD, Thermogravimetry (DSC/TGA) techniques, and Raman spectroscopy. The XRD results revealed the formation of solid solutions containing copper titanium phosphate ($Cu_{0.5}Ti_2(PO_4)_3$) at 1:1 and 2:1 molar ratios of α-TiP to copper (II) acetate while the products obtained at 1:2 and 1:1 molar ratios confirmed the formation of copper titanium oxyphosphate ($Cu_{0.5}TiOPO_4$). Moreover, TGA results confirm the development of these mixed metal phosphates which can be further analyzed for applications in the ceramic industry. Therefore, calcination of the reaction mixtures by changing the molar ratios produced different colored stable compounds that can potentially be used as ceramic pigments, coatings, and electrode materials.

Keywords: Copper titanium phosphate, α-titanium bismonohydrogen orthophosphate monohydrate, Solid-state reaction route