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STRUCTURAL AND MORPHOLOGICAL CHARACTERIZATION OF THE CHROMIUM-DOPED β-SPODUMENE TO CERAMIC PIGMENT

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Introduction: Synthetic β-spodumene has been evaluated for optically stimulated luminescence (OSL) response and its great potential use for beta or gamma radiation dosimetry and synthesis techniques have been improved for this material [1-2]. However, its application as a promising ceramic pigment has not been reported. Pigments have the ability to color materials from the dispersion of insoluble particles and their color occurs due to the action of a chromophore ion incorporated in the structure [3]. In this paper the structural and morphological chacacteristics of synthetic chromium-doped β-spodumene were evaluated aiming its application in a novel ceramic pigment.

Material and method: Chromium-doped β-spodumene samples were prepared following the procedure described by Lima et al. (2014). Samples were produced containing weight percentages of Cr_2O_3 (NEON, purity of 98%) – 0.5, 1, 1.5, 2, 2.5, and 3 %.

For structural characterization measurements were taken of X-ray diffraction (XRD). The measurements were taken with a powder diffractometer Rigaku Miniflex. The experimental results obtained were compared with the the teorical patterns available in the PDF2 (Powder Diffraction File) crystallographic database using the software X'Pert HighScore Plus (PANalytical B. V.). The Rietveld refinement method was provided to ratify the occurrence of the β-spodumene phase for the samples produced, using the DBWSTools 2.4 program [4].

The morphology of the material was investigated by microscopy scanning electronics (SEM). Micrographys with magnifications of 100, 500, 1000 and 5000 times of SEM were obtained using a scanning electron microscope.

Results: XRD experimental patterns confirmed the occurrence of β-spodumene with tetragonal structure and crystallographic parameters are: a = b = 7.5410 Å, c = 9.1560 Å, α = β = γ = 90º; density = 2.37 g/cm^3; and volume = 520.67 x 10^6 pm^3.

Rietveld refinement confirmed that this single phase has a good fit even considering chromium-doped samples. The size of the crystallites is of a nanometric order. No reduction was observed for these values with the increase in the percentage of doping, which indicates that the interstitial presence of chromium up to the analyzed concentration (2,5% by weight) does not cause significant microdeformations in the crystal lattice. SEM micrographs reveal irregular particles with well-defined edges, characteristics of the tetragonal crystal system of β-spodumene. Morphological interferences with the chromium doping process were not identified. The preservation of the structure and morphology of β-spodumene allows characterizing this material as stable and inert, as well as pure β-spodumene, and its use can be evaluated in several applications.

Conclusions: This preliminary study shows the formation of the crystal structure of β-spodumene and the XRD characterization and Rietveld refinement indicated the absence of significant distortions in the lattice with doping up to 2.5 % chromium-doping. No morphological changes were observed from SEM analysis either, indicating that the material possibly preserves the physical characteristics of pure β-spodumene.

References:

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