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Investigation of ethylene glycol on the photoluminescence behavior of $PbWO_4$ powders processed in microwave-hydrothermal

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Abstract – $PbWO_4$ powders were synthesized by the co-precipitation method using different concentrations of ethylene glycol and processed in a microwave-hydrothermal system at 140 ºC for 30 min and 60 min. These powders were analyzed by X-ray diffraction (XRD) and photoluminescence (PL) measurements. XRD patterns indicated that these materials crystallize in a tetragonal structure. When excited with 350 nm wavelength, these ceramic compounds exhibited a broad PL band with the maximum situated in the range from 450 nm to 600 nm. This behavior was explained by means of the presence of intermediary energy levels within the band gap.

Currently, microwave-assisted synthesis methods have received special attention due to the possibility of formation of materials with different morphologies, high degree of crystallinity and easy dispersion in aqueous medium [1]. The literature [2] reports that solvents with high dielectric constants are rapidly heated up to high temperatures at short time when submitted to the microwave radiation. In addition, when the relaxation time is one or two orders of magnitude different from those corresponding to the microwave frequency, the solvent becomes an effective medium due to its large loss tan$d$. Hence, solvents with tan$d$ > 0.1 (water), such as the ethylene glycol (EG) (tan$d$ = 1.35, relaxation time = 112.87 ps), can be considered good candidates in the material syntheses or processing by microwave radiation.

In this work, $PbWO_4$ powders were synthesized by the co-precipitation method with different concentrations of EG (0, 25, 50, 75 and 100%) and processed by microwave-hydrothermal (MH). In the synthesis, tungstic acid (99% purity, Aldrich), lead nitrate (99.5% purity, Aldrich) and EG (99.5% purity, J.T. Baker) were used as raw materials. These chemical compounds were dissolved in deionized water. After co-precipitation reaction, the solution was transferred into a Teflon autoclave, which was sealed and placed inside the MH system (2.45 GHz, maximum power of 800 W). Each MH processing was performed at 140ºC for 30 min and 60 min. The heating rate was fixed at 25 ºC/min and the pressure inside the autoclave was stabilized at 294 kPa. After MH processing, the autoclave was naturally cooled to room temperature. The resulting solution was washed with deionized water several times to neutralize the solution (pH ≈7). Finally, the white precipitates were collected and dried in a conventional furnace at 75 ºC for some hours.

The X-ray diffraction patterns showed that all $PbWO_4$ powders have a scheelite-type tetragonal structure without the presence of secondary phases, in agreement with the respective Joint Committee on Powder Diffraction Standards card nº. 19-0708 [3] (Fig. 1(a, b)). Moreover, the intense and well-defined diffraction peaks indicate that these ceramic compounds have a high degree of structural order at long-range. Although using the same MH processing conditions (140 °C for 30 min), it was observed that the powders synthesized only in EG presented a high photoluminescence (PL) emission when compared to the obtained in aqueous medium (Fig. 2(a)). On the other hand, when the processing time was increased up to 60 min, the maximum PL intensity was verified for the powders synthesized with 50% of EG into the aqueous solution (Fig. 2(b)). In principle, these results suggest that the concentration of EG as well as the processing time are able to induce the formation of different intermediary energy levels within the band gap, resulting in non-linear variations on the PL intensity of the powders.

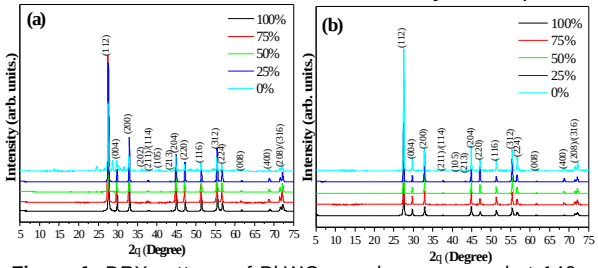

Figure 1: DRX patterns of $PbWO_4$ powders processed at 140 ºC for: **(a)** 30 min and **(b)** 60 min.

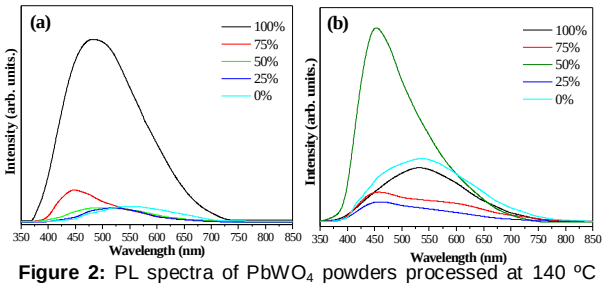

Figure 2: PL spectra of $PbWO_4$ powders processed at 140 ºC for: **(a)** 30 min and **(b)** 60 min.

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