INFLUENCE OF REACTION TIME ON THE PROPERTIES OF Zn_2SnO_4 NANOPARTICLES OBTAINED BY HYDROTHERMAL METHOD

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***Abstract*– This work investigates the influence of reaction time (ranging from 12 to 96 h) on the characteristics of zinc stannate (Zn_2SnO_4) nanoparticles obtained by the hydrothermal method at 200°C. The nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), surface area measurements (BET) and infrared spectroscopy (IR). The results showed that the reaction time influenced the properties of Zn_2SnO_4 nanoparticles. The product presented a pure crystalline phase with mean particle size about 20 nm and BET surface area ranging from 34 to 43 $m^2.g^{-1}$.**

***Keywords*– Zn_2SnO_4, synthesis, hydrothermal process.**

I. INTRODUCTION

Semiconductors composed by binary oxides of ZnO, SnO_2, In_2O_3 and CdO are known by their excellent optical and electrics properties, being widely used in a variety of applications including smart windows, photovoltaic slim films and, optical and electronic nanodevices (Nomura *et al.*, 2003; Liang *et al.*, 2001). In the last years, the application as semiconductors of ternary nanostructured oxides was verified in several studies concerning the synthesis of these oxides. Among these materials the ternary zinc stannate oxide (Zn_2SnO_4) is known by its diamagnetic and semiconductor properties (Coffen, 1953).

The Zn_2SnO_4 is a spinelic material used in gas sensors (Yu and Choi, 2002), as an anode for Li-ion battery (Belliard *et al.*, 2000) and as catalyst in the decomposition reaction of benzene in aqueous solutions (Wang *et al.*, 2002). It is usually prepared by the solid-state reaction between ZnO and SnO_2 at high temperatures (higher than 1000 °C) (Hashemi *et al.*, 1990). However, the high temperature involved in the synthesis of Zn_2SnO_4 contributes to the loss of ZnO by evaporation and the formation of SnO_2 that difficult the obtainment of a pure crystalline phase (Stambolova *et al.*, 2005).

Some strategies to overcome these drawbacks were proposed in the literature. To avoid the partial evaporation of ZnO, different thermal treatments consisting of several steps and intermediary homogenization of product (Gupta and Mathur, 1968) were used. The formation of a pure phase of the spinel has been obtained after the mixture of ZnO and SnO_2 at 1280°C (Hashemi *et al.*, 1990). Mechanical activation by milling of ZnO and SnO_2 ,previous to the thermal treatment at 1200 °C from 10 to 160 min, was also reported (Nikolic *et al.*, 2001). Co-precipitation of the hydroxides Zn and Sn with NaOH from an aqueous solution containing $ZnSO_4$ and $SnCl_4$ at molar ratio of 2:1 was used. The precipitated was converted to Zn_2SnO_4 at 500-900 °C (Cun *et al.*, 2002). Zn_2SnO_4 also has been formed after calcination of hydroxide precursor at temperature of 650-750°C. The hydroxide was obtained by the co-precipitation of $Zn(NO_3)_2$ and $SnCl_4$ and using Na_2CO_3 as mineralizing agent. The product presented average particle size in the order of 20 nm after calcination at 650 °C for 24 h (Stambolova *et al.*, 2005). Nanotubes of Zn_2SnO_4 were produced by thermal evaporation, at 1000 °C and 2 h, obtaining particles with diameter in the range of 50-100 nm (Wang *et al.*, 2004a). Nanobelts and nanorings of Zn_2SnO_4 were also obtained by the same method of synthesis and under the same experimental conditions (Wang *et al.*, 2004b). Nanoplates of Zn_2SnO_4 with a thickness of 50 nm were obtained by hydrothermal method (200 °C at 20 h of reaction time) employing hexadecil-trimethyl ammonium as surfactant (Ji *et al.*, 2010). Nanowires of 50-100 nm of diameter were obtained by vapor chemical deposition method heating a mixture of metal Zn and Sn powders at 800-900 °C (Hu *et al.*, 2009). Microtubes formed by ternary oxide were synthesized by the hydrothermal method (220 °C for 48 h), resulting in microtubes with diameter of 0.8 to 1.2 μm, which are composed of numerous nanoparticles with diameter of 10-20 nm and surface area of 41.2 $m^2.g^{-1}$ (Ai *et al.*, 2010). The spinelic oxide also was synthesized using supercritical water in a bath reactor (400°C and 30 MPa) forming particles of 0.5-1.0 μm (Lee *et al.*, 2010). The use of Na_2CO_3 as mineralizing agent under hydrothermal conditions in the range of 120-230 °C for up to 30 h resulted in the formation of Zn_2SnO_4 particles with diameters near 20-50 nm (Annamalai *et al.*, 2010). Other studies reported the influence of different amines (ethylamine, n-butylamine, n-hexylamine and n-octylamine) as mineralizing agents on properties of resulting Zn_2SnO_4 particles at 180 °C for 20 h (Fu *et al.*, 2009). Zn_2SnO_4 was synthesized via hydrothermal method at 220 °C for 72 h from zinc acetate and tin tetrachloride in various mediums containing different NaOH concentrations (0 to 6 $mol.L^{-1}$). The optimum concentration for Zn_2SnO_4 crystallization was around 2.67 $mol.L^{-1}$ (Fang *et al.*, 2001).

Although there are several studies reporting different alternatives to synthesize the Zn_2SnO_4 there are no stud-