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Synthesis and Characterization of oxides of nickel and zinc obtained by sol-gel

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Abstract – Simple and mixed zinc and nickel oxides were synthetized by the sol-gel process. The mixed oxides were denominated $Ni_{1-x}Zn_xO$, (x = 0,1; 0,2; 0,3). The characterization of these materials was achieved by thermal analysis (TG/ATD), X-ray diffraction (XRD), N_2 adsorption (BET/BJH) and scanning electron microscopy (SEM). The characterization results showed good correlation with literature data for oxide cristallinity, homogeneity and thermal stability.

Simple and mixed oxides of transition metals are used as adsorbents to minimize sulfur compounds present in fossil fuels. The zinc and nickel are metals semiconductors and generally have good performance in tests of adsorption and catalytic [1]. Mucka et al. [2] synthesized a veriety of ZnO-NiO systems by physical mixing of oxides, which were effective in the decomposition of hydrogen peroxide. Recently, there has been significant growth is interest in sol-gel process, because the materials obtained by this method show good homogeneity of the crystalline phases and relatively low temperatures of synthesis, when compared with other methods. For these reasons the present work aims at synthesis of simple and mixed oxides of nickel and zinc by sol-gel process and thermal characterization, structural and morphology of these materials.

The reagents used to synthesis of the oxides simples and mixed ($Ni_{1-x}Zn_xO$, with x = 0; 0.1; 0.2; 0.3 and 1) were nickel acetate, zinc acetate, citric acid and glycol ethylene. Appropriated amounts of metal sources were dissolved in an aqueous solution of citric acid at 60℃ under vigorous stirring. After 2h ethylene glycol was added. This resin was submitted to thermal treatment at 400℃ for 2h to obtain the precurso r powder of the oxides. The decomposition of precursor powder was studied by thermal analysis (TG/DTA) and after thermal treatment at 500℃ and 700℃ the materials obtained were characterized by X-ray diffraction (XRD), N_2 adsorption (BET/BJH) and scanning electron microscopy (SEM).

The curves TG and DTA showed the same behavior to all the samples, and reveal the appearance of two distinct mass loss regions. The first one (between 30-150℃) was attributed to the loss of adso rbed water, which is confirmed by the endothermic peak in the DTA curves. The second mass loss (150-600℃) occurs followed by exothermic peaks with high intensity, characteristic of oxidation of organic components. The corresponding X-ray diffractograms are consistent with those expected for structure, in the temperatures of calcination (500℃ and 700℃). In the Figure 2 i s showed the XRD of the oxides mixed ($Ni_{1-x}Zn_xO$, with x = 0.1; 0.2 and 0.3), and can be observed that an increase in amount of zinc (x = 0.3) gave origin the secondary phase related to ZnO. The pore diameters obtained, by N_2 adsorption, were similar indicating that the oxides analyzed are mesoporous, with size in the range 13.74-24.11 nm. The increase in the area surface (18.86-56.89 m^2g^{-1}) with the increase amount of zinc can be due the disorganization structural. The micrograph showed that the oxides are present as particles agglomerates and non-uniform plates, with different size of particles (Figure 3).

The results showed that the oxides ($Ni_{1-x}Zn_xO$, with x = 0; 0.1; 0.2; 0.3 and 1) were obtained by sol-gel, that the oxides are thermal stable and crystallized at relatively low temperatures with particles nanometric, and that the surface area increase with amount of zinc.

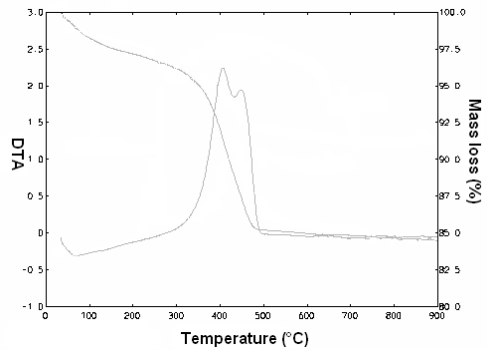

Figure 1: Curves TG and DTA of precursor powder of $Zn0.7Ni_{0.3}O$.

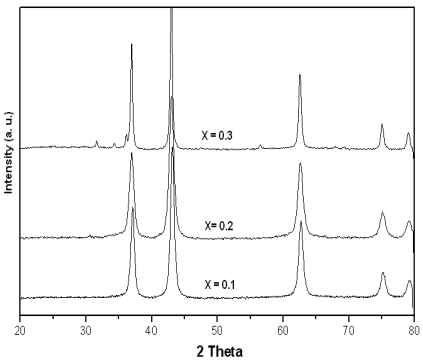

Figure 2: XRD of $Zn_{1-x}Ni_xO$ at 700℃

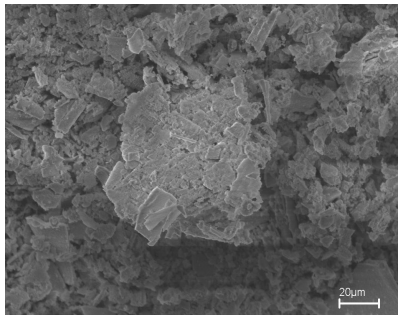

Figure 3: SEM of $Zn_{0.8}Ni_{0.2}O$ at 700℃

References

[1] Z. M. Wang, et al., Industrial & Engineering Chemistry Research, 37 (1998) 4675.
[2] V. Mucka, et al., Radiation Physics and Chemistry, 37 (2002) 177.