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APPLICATION OF SOL-GEL METHOD FOR SYNTHESIS OF $Ni(OH)_2$ AND NiO NANOPARTICLES

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Abstract

The sol-gel method was used for obtaining $Ni(OH)_2$ and NiO. For structural characterization of the material we used X-Ray Diffraction (XRD). Morphological structures and chemical composition were examined by scanning electron microscopy (SEM.). The results obtained, it was found that the average particle size of the $Ni(OH)_2$ is about 15.8 nm and the thermal treatment for calcination at 350° C obtain NiO particle size is 48.5 nm. The result of DSC analysis of the precursor product showed that the proper calcination temperature was 350°C.

Key words*: characterization, sol-gel method, nickel oxide, synthesis.*

INTRODUCTION

Synthesis and characterization of new nanomaterials have a significant importance because of their essential role in fundamental research and its technological applications (Ciobanu et al., 2013, 2014a; Harja et al., 2016).

In the last decades, nanomaterials have been used in various applications due to their properties: chemical, optical, mechanical, magnetic, thermal etc. (Muneeret et al., 2015). Those properties determine for nano materials a different application ssuch as adsorption, photocatalysis, supercapacitors, batteries, sensors, solarcells etc.(Ciobanu et al., 2014b; Harja and Ciobanu, 2017 and 2018; Warnan et al., 2014).

An important p-type semiconductor is NiO, it is study recently due to extensive and attractive application in various fields: sensors, photo catalysis (Zouet al., 2006), electro catalytic (Zayimet al., 2008), magnetic area (Karthiet al., 2011) and catalysis materials (Kobayashi et al., 2011). Moreover, due to the high surface area of NiO, it is promising in the field of super capacitors and gas sensors. On the other hand, $Ni(OH)_2$ is most widely used in battery, storage for it high-power density, high proton diffusion coefficient and low toxicity (Khan et al., 2011).It is also reported that metal coating or doping on NiO can enhance the super capacitor performance. Cu-doping NiO, Fe-doped NiO hollows pheresand Au nanoparticles coating on NiO have reported (Zhao et al., 2011). Owing toth eexcellent conductive capacity and huge specific are as of Ag nanospheres, the capacitance performance of Ag doped NiO nanocomposites can be enhance defectively (Wu et al., 2007).

In the last decade were developed different methods for the synthesis of nano-NiO (Wang and Su, 2016).

The main methods for nano-NiO synthesis are: sol–gel (Nutescu Duduman et al., 2016; Ba-Abbad; Wu et al., 2007), solvothermal methods (Pan et al., 2012) micro-emulsion precipitation (Han et al., 2004; Lopez-Quintela, 2003) chemical vapor deposition (Sasi and Gopchandran, 2007; Ohta et al., 2003), sputtering (Chen et al., 2005) and liquid-control precipitation.

The more used methods for obtaining NiO are at high temperatures (Zanella et al., 2012), other techniques are complicated because is difficult to removing of templates (Zheng et al., 2005). So it is full of challenge to develop novel procedures to synthesize NiO

nanomaterials without any template. In this paper was synthesized, by facile method, $Ni(OH)_2$ and NiO that were characterized.

MATERIALS AND METHODS

For the experiment were used: Nickel Sulfate Hexahydrate ($NiSO_4 \cdot 6H_2O$) solution 0.5 M, sodium hydroxide (NaOH) solution 1M by Panreac. Morphological structure and chemical composition was examined by SEM - JEOL 6400 with an Oxford Link EDX microanalyser and Pentafet light sensing. For structural material characterization XRD method was used - Philips model X`Pert PDP3040 with a source Ka1 Cua, 40 kV and 40 mA. The program which was used for the analysis of the diffraction patterns is X`PertHihgScorePlusPANalytical (version 2.0).
The crystallite size of the prepared materials is calculated according to Scherrer equation:

$$d=0.94\ \lambda / ß \cos \theta \qquad (1)$$

where λ is the X-ray wavelength, ß is full width at half maxim um value, and θ is the diffraction angle
DSC analysis was performed with Controler DSC, Mettler Toledo.
The $NiSO_4$0.5 Msolution and NaOH1M solution were contacted slowly and pH value was monitoring. Figure 1 shows samples at different pH values.

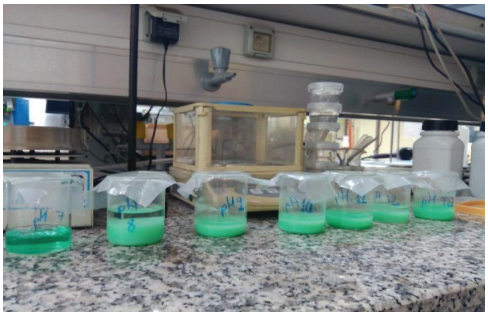

Figure 1. Samples at different pH

By adding sodium hydroxide size of synthetized materials may be controlled, in order to obtain the nanoparticles, and also leads to neutralization of reaction mass.

The solution was stirred for 2-3 hours at a temperature of 25°C, decanted for 24 hours to achieve complete phase separation. After this solid was filtered, wash with deionized water at neutral pH.
The synthetized materials were dry it in the oven at 100°C, these samples have been calcined for 2 h at 350°C.

RESULTS AND DISCUSSIONS

Figure 2 presents SEM images we can see the small powder agglomerates and EDX analysis confirms nickel, oxygen and hydrogen.
The XRD diffractogram (Figure 3) corresponds (JCPDS No. 14-17) to the major phase is the ophrastite with hexagonal structure and, to a lesser extent - $Ni(OH)_2$ and β-$Ni(OH)_2$.

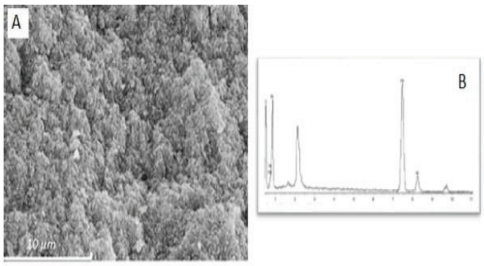

Figure 2. SEM micrographs and EDX of sample $Ni(OH)_2$.

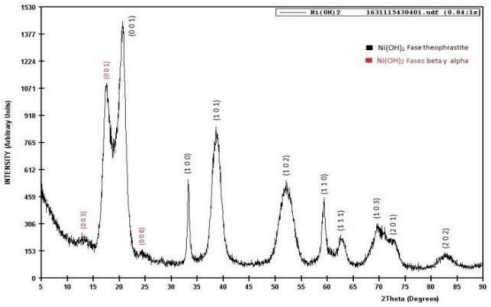

Figure 3. XRD patterns of sample $Ni(OH)_2$

The peaks have been detected and belong to the crystallographic plans (001), (100), (101), (102), (110), (111), (103), (201) and (202). After calcination in the figure 4 agglomerates with larger nanoparticles are observed and EDX analysis confirms oxygen, nickel and gold those are used to prepare the samples.

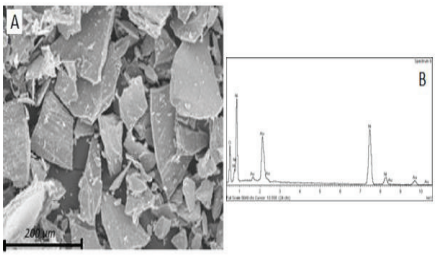

Figure 4. SEM micrographs and EDX of sample NiO

XRD diffractograms confirms the cubic structure of NiO (JCPDS No. 78-0643) and observes the crystallographic planes (111), (200), (220), (311), (222) (Figure 5).

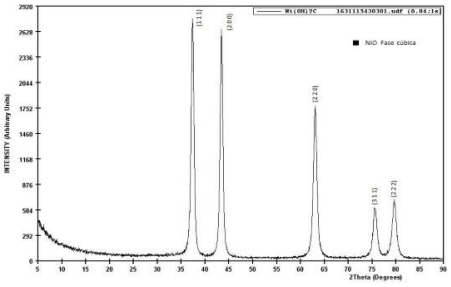

Figure 5. XRD patterns of sample NiO

Considering the results obtained, table 1 it was found that the average particle size of the $Ni(OH)_2$ is 15.8 nm and by the calcination at 650° C was obtained NiO particles, with 48.5 nm size.

Table 1. Average size of the nanoparticles calculated by the Scherrer equation

	$Ni(OH)_2$			NiO		
peak	001	101	102	111	200	220
β	0.008	0.007	0.012	0.003	0.002	0.003
θ	0.168	0.336	0.454	0.325	0.378	0.549
d (nm)	16.1	18.7	12.6	42.0	57.0	46
media(nm)	15.8			48.5		

DSC analysis, figure 6, shows the existence of two endothermic peaks. The first one (A), about 100ºC, corresponds to the water loss the sample might contain. The second peak, centered at 300°C (B), which starts at 230°C and terminating at 325°C is the transformation of nickel hydroxide to nickel oxide.

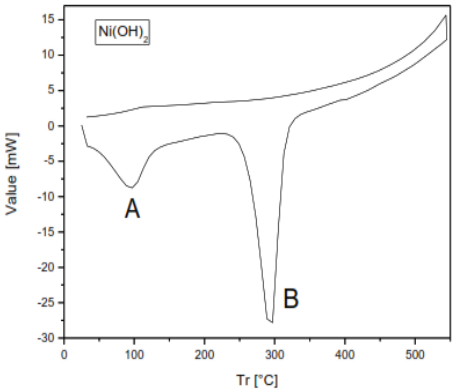

Figure 6. DSC initial sample - $Ni(OH)_2$

DSC analysis for calcined materials, figure 7, demonstrated stability of NiO.

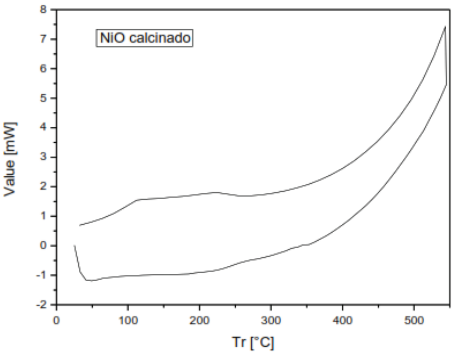

Figure 7. DSC calcined sample

After calcination at 350°C and after NiO formation, other transformations aren't observed. It is noticed that $Ni(OH)_2$ was irreversibly transformed into NiO.

CONCLUSIONS

$Ni(OH)_2$ and NiO nanoparticles were synthesized by sol-gel method. Synthesized nanoparticles were characterized by XRD, SEM, EDX and DSC. The results obtained, it was found that the average particle size of the $Ni(OH)_2$was about 15.8 nm and by the thermal treatment NiO particle obtained had 48.5 nm size. The result of DSC analysis of the precursor product showed that the proper calcination temperature was 350°C.

The synthetized nano $Ni(OH)_2$ was used to prepare nanosized NiO in a very simple and rapid procedure.

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