

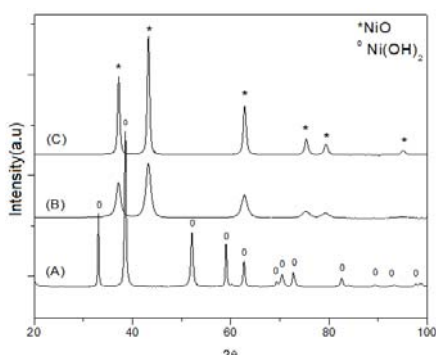
## Synthesis and characterization of NiO plates through thermal decomposition of Ni(OH)<sub>2</sub> precursors by microwave heating

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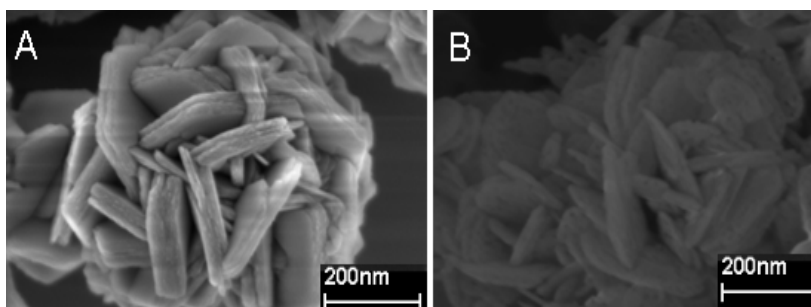
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**Abstract** – NiO nanostructures were obtained by thermal decomposition of Ni(OH)<sub>2</sub> precursors synthesized by the microwave-hydrothermal method. The structural and morphological properties were analyzed by X-ray diffraction (XRD), field emission gun scanning electron microscopy (FEG-SEM) and temperature-programmed reduction (TPR). XRD patterns indicated the formation of pure NiO phase when the Ni(OH)<sub>2</sub> precursors were calcined at 300 °C and 500 °C for 5 min in a calcining microwave oven. FEG-SEM micrographs showed that the NiO powders are composed by several aggregated particles with plate-like morphology. The TPR measurements suggested different reduction profiles for the NiO phase in both calcining temperatures.

Nickel oxide (NiO) is an interesting material due to its technological applications, such as: battery cathodes, catalysts, gas sensors, magnetic devices. Generally, the literature<sup>[1]</sup> has reported the formation of NiO nanostructures by means of the decomposition process of Ni(OH)<sub>2</sub> precursors previously obtained by the conventional hydrothermal method. In principle, the energy transfer mechanism using microwave radiation is more different from those well-established for the heating transfer: conduction, radiation and convection. Currently, microwave-assisted synthesis routes have been successfully employed for the preparation of nanosized inorganic materials<sup>[2]</sup>. In this work, we report on the formation of NiO plates through the microwave heating employed on the Ni(OH)<sub>2</sub> precursors. These precursors were synthesized by the microwave-hydrothermal method at 100 °C for 1 min, using an aqueous solution containing nickel nitrate (Ni(NO<sub>3</sub>)<sub>2</sub>) and ammonium hydroxide (NH<sub>4</sub>(OH)). The NiO plates were obtained when the Ni(OH)<sub>2</sub> precursors were heat treated at 300 °C and 500 °C for 5 min in a calcining microwave oven, respectively. In Figures 1A-C, the diffraction peaks confirmed that the Ni(OH)<sub>2</sub> precursors have a hexagonal structure, while the NiO phase crystallize in a cubic structure. FEG-SEM micrographs indicated that the Ni(OH)<sub>2</sub> precursors are composed by plate-like microparticles with agglomerated nature (Figure 2A). Also, FEG-SEM micrographs of NiO powders obtained after microwave heat treatment performed at 500 °C for 5 min showed the formation of plate-like morphologies with the presence of pores on its surfaces (Figure 2B). These pores can be arising from the water dehydroxylation process on the Ni(OH)<sub>2</sub> precursors during the microwave heat treatment. The TPR analyses suggested that the NiO reduction profiles have different behaviors for the two calcining temperatures (300 °C and 500 °C). These materials presented a reduction from NO to N<sub>2</sub> in the presence of CO and they reached 90% conversion for both reactants.



**Figure 1:** XRD patterns of (A) Ni(OH)<sub>2</sub> precursor, (B) NiO obtained after microwave heat treatment of Ni(OH)<sub>2</sub> at 300 °C for 5 min and (C) at 500 °C for 5 min.



**Figure 2:** FEG-SEM micrographs of (A) Ni(OH)<sub>2</sub> precursors obtained by the microwave-hydrothermal method at 100 °C for 1 min and (B) NiO plates formed after microwave heat treatment of Ni(OH)<sub>2</sub> at 500 °C for 5 min.

### References

- [1] Y. Qui, H. Qui, C. Lu, Y. Yang, Y. Zhao, J. Mater. Sci. 96 (2005) 479.  
 [2] T-L. Lai, Y-Y. Shu, G-L. Huang, C-C. Lee, C-B. Wang, J. Alloys Comp. 450 (2008) 318.