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Microwave hydrothermal synthesis of α-Fe₂O₃

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Abstract – Iron oxide was synthesized by the microwave hydrothermal method. The samples were characterized by IR spectroscopy, X-ray diffraction (XRD) and the morphology was evaluated by scanning electron microscopy (SEM). The resultant powder was characterized as α -hematite, the most stable phase of iron oxide. IR spectra show the formation of FeO₆ polyhedra, with a small dislocation in the position of the bands. The authors acknowledge the financial support of FINEP/MCT

Hematite has important technological applications, being used as in catalyst, magnetic material, pigments, gas sensor, biomaterial and others [1]. Synthesis of Fe_2O_3 in nanometric scale can improve some properties, especially related to surface phenomena. Among the synthesis methods available to obtain nanoparticles, the microwave hydrothermal method [2] was chosen in the present work, due to its high kinetic, which accelerates the synthesis process, besides being environmental friendly, as water can be used as solvent.

In this work, α -Fe₂O₃ synthesis was done in aqueous solution using Fe(NO₃)₃.9H₂O as precursor, NaOH as alkalinizing agent and polyethylene glycol (PEG 300) as template. The suspension was placed in a teflon reactor and coupled into the microwave oven. Synthesis was done at 150 °C for 30 or 60 min. Characterization was done by infrared spectroscopy (IR), X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

IR spectra showed well defined Me – O bands, indicating that both samples had a high short range order, with the formation of the FeO₆ polyhedra. These vibrations were observed at about 555 and 470 cm⁻¹, assigned to Fe-O bond, with some dislocation in relation to literature data [3]. The XRD patterns (Fig. 1) confirmed the crystallization of the material, with the formation α -hematite, for both synthesis times. The morphological evaluation was carried out by SEM (Fig. 2). It was observed that spherical particles were formed and with a high agglomeration degree among them.

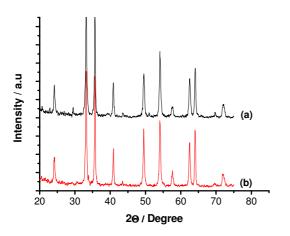


Figure 1: XRD patterns of $\alpha\text{-Fe}_2O_3$ after synthesis during: (a) 30 min; (b) 60 min.

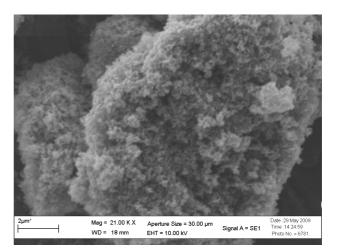


Figure 2: SEM micrograph of α -Fe₂O₃ with synthesis time of 60 min.

References

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