CuO Synthesized by Microwave Hydrothermal method at different conditions

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Abstract – Copper 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 monoclinic CuO. XRD pattern showed a good crystallization for CuO alkalized with NaOH for 1 min, while the absence of alkalinizing agent led to an incomplete reaction, with dislocation in the position of IR bands and low crystallinity degree. Acknowledgements: The authors acknowledge the financial support of FINEP/MCT

Copper oxide (CuO) is a p-type semiconductor material which has been studied for applications in photothermal, photoconductive, magnetic and superconductor devices. Various efforts have been directed toward the synthesis of nanostructured CuO to enhance its performance in currently existing applications and different morphologies, as nanometric rods, wires, ribbons and belts, were already obtained [1,2].

In this work, CuO synthesis was done in aqueous solution using copper (II) acetate \( (\text{CuOOCCH}_3\text{CH}_2\text{O})_2\text{H}_2\text{O} \) as precursor, NaOH and \( \text{NH}_4\text{OH} \) as alkalinizing agents. The solution or suspension (when alkalinized) was placed in the Teflon reactor and coupled into the microwave oven. Synthesis was done at 150°C and 120°C for 1, 60 and 180 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 the samples had a high short range order. In the spectra of CuO synthesized without alkalinizing agent, vibrations were observed at about 586, 509 and 493 cm\(^{-1}\), being assigned to Cu-O [3]. By the literature data, these peaks confirmed the CuO crystallization by hydrothermal synthesis [4], but some dislocation was observed. When alkalinized with \( \text{NH}_4\text{OH} \) and \( \text{NaOH} \) these peaks appear around 601, 509 and 432 cm\(^{-1}\). The XRD patterns (Fig. 1) confirmed the crystallization of the material, with the formation of single phase monoclinic CuO. A small crystallization degree was observed for the sample synthesized without alkalinizing agent, at 120°C for 60 min. For these samples, the reaction was not complete and \( \text{Cu}^{2+} \) was present in solution after synthesis. On the other hand, the alkaline media favored the crystallization process. The best result was for syntheses in NaOH, which occurred in only 1 min. The morphological evaluation was carried out by SEM (Fig. 2). It was observed that spherical particles were formed and the lack of alkalinizing agent led to a low agglomeration degree.

![Figure 1: XRD patterns of CuO: (a) without alkalinizer, 120°C, 60 min, (b) without alkalinizer, 150°C, 180 min, (c) \( \text{NH}_4\text{OH}, 150°C, 60 \text{ min} \), (d) \( \text{NH}_4\text{OH}, 150°C, 180 \text{ min} \) (e) \( \text{NaOH, 120°C 1 min} \).](image1)

![Figure 2: SEM micrograph of CuO synthesized without alkalinizer at 150°C for 180 min](image2)

References