

MICROWAVE ASSISTED SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF NICKEL OXIDE NANOPARTICLES

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ABSTRACT

Nickel oxide (NiO) nano-particles were produced via a simple microwave method from the Ni(OH)₂ precursor, which was obtained by slow drop-wise addition of 0.1M sodium hydroxide to 0.1M nickel nitrate. The mixture was vigorously stirred until the pH reached 7.2. The mixture was then irradiated with microwave to deposit Ni(OH)₂ at a better precipitation rate. Drying the precipitate at 320°C resulted in formation of NiO nanoparticles. High Resolution Transmission Electron Microscope (HRTEM), Scanning Electron Microscope (SEM) and X-ray diffraction (XRD), employed for the structural characterization of the as-prepared NiO nanoparticles, revealed their good crystallinity and high-purity. Microwave irradiation increased homogeneity and decreased the mean particle size of the produced NiO particles.

Keywords: NiO, microwave synthesis, nanoparticles, HRTEM, SEM, XRD.

1. INTRODUCTION

Nano-particle oxides of transition metals have attracted materials scientists. These materials have exceptional properties which stimulate many advanced applications (Duran *et al.*, 2003; Wang *et al.*, 2005 Mazaheri *et al.*, 2008). Nano-structured nickel oxide is a prominent example having a large exciton binding energy and a wide band gap ranging from 3.6 to 4.0eV. This p-type semiconductor can be used in optical, electronic, catalytic and super-paramagnetic devices like transparent conductor films, gas sensors, alkaline battery cathodes, dye-sensitized solar cells and solid oxide fuel cells (SOFC) (Bhadur *et al.*, 2008; Sato *et al.*, 1993). Versatile methods such as sol-gel (Ghosh *et al.*, 2006; Wu *et al.*, 2007), chemical precipitation (Bhadur *et al.*, 2008; Bahari Molla Mahaleh *et al.*, 2008) and anodic arc plasma method (AAPM) (Hongxia *et al.*, 2009) have been used to produce nanomaterials. Microwave heating has such advantages as high-efficiency, nanoparticle rapid-formation, narrow crystallite size distribution and agglomeration decrease when compared to the conventional methods (Krishnakumar *et al.*, 2009). Microwave methods apply electromagnetic waves having 0.001 to 1m wavelength to accelerate the chemical reaction of interest. These wavelengths correspond to frequencies between 0.3 to 300GHz. Synthesis via microwave routes is simple, energy efficient, time saving and produce great of samples (Krishnakumar *et al.*, 2009). Production of nickel oxide nanoparticles by microwave chemical synthesis,

their morphological characterization and their structural study are discussed in this paper.

2. MATERIALS AND METHODS

2.1. Preparation of NiO nanoparticles

Microwave synthesis of NiO nanoparticles comprised three stages: (1) formation of Ni(OH)₂ precursor, (2) microwave irradiation of Ni(OH)₂ and (3) annealing of Ni(OH)₂ to convert into NiO. Ni(OH)₂ precursor was obtained by drop-wise slow addition of 0.1M NaOH to 0.1M Ni(NO₃)₂ while vigorous stirring of the solution continued until the pH reached 7.2. The mixture was then irradiated by microwave (2.45GHz, 900W, SAMSUNG) until a dry green precipitate formed. Simultaneous thermal analysis (TG-DTA) was carried out using (Universal V4.5A TA Instrument) to determine the Ni(OH)₂ to NiO conversion temperature under air. After determining the temperature of nickel hydroxide to nickel oxide conversion by thermal analysis, the oven-dried cake was heated up to 320°C for 1hour to form dark grey particles. The resulting powder was filtered and washed several times with distilled water and finally with ethanol to remove the residual by products.

2.2. Characterization of the prepared nanoparticles

X-ray diffraction (X'per PRO model) was used for structural study and characterization of the sample. Phase purity of the initial powder was also investigated by XRD. Morphological study was carried out by Scanning Electron Microscope (HITACHI Model S-3000H). High Resolution

Transmission Electron Microscope (Jeol Gem Model) was used to analyse the particle size of the NiO nanoparticles.

3. RESULTS AND DISCUSSION

3.1. Thermal Analysis

Fig. 1 shows TG-DTA curves of the Ni(OH)₂ precursor. It can be seen that two endothermic reactions take place between ambient temperature and 600°C in the sample Ni(OH)₂. Both reactions accompanied the mass reduction due to H₂O removal from the powder, to form NiO as the end product (Kim *et al.*, 2006).

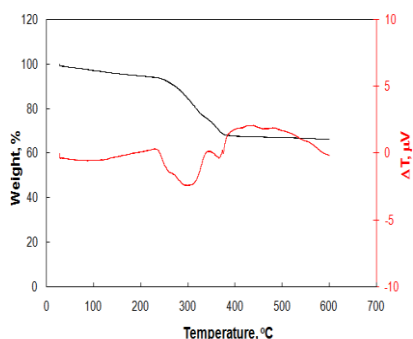


Fig. 1. TG-DTA Curves of Ni(OH)₂

3.2. XRD analysis

XRD pattern (Fig. 2) confirms the formation of nickel oxide (JCPDS card No. 22- 1189). No other components were detectable in the final product. The mean crystallite size was calculated by application of the Debye-Scherer equation, $D = K\lambda / \beta \cos\theta$, where, θ is Bragg diffraction angle, K is Blank's constant, λ is the source wavelength (1.54), and β is the width of the XRD peak at half maximum height, is 19 nm (Needham *et al.*, 2006).

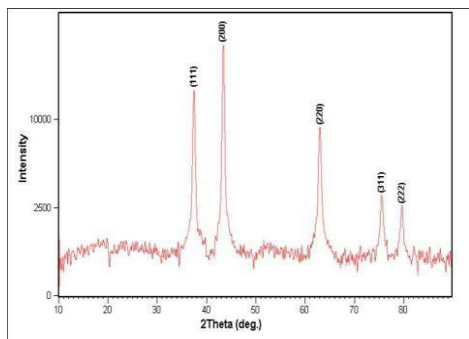


Fig. 2. XRD pattern of NiO nanoparticles

3.3. SEM Analysis

Aggregated particles around 50 to 300nm in diameter are observable in the SEM images (Fig. 3).

Their creation may be due to the influence of the interfacial energies and intraparticle magnetic interactions.

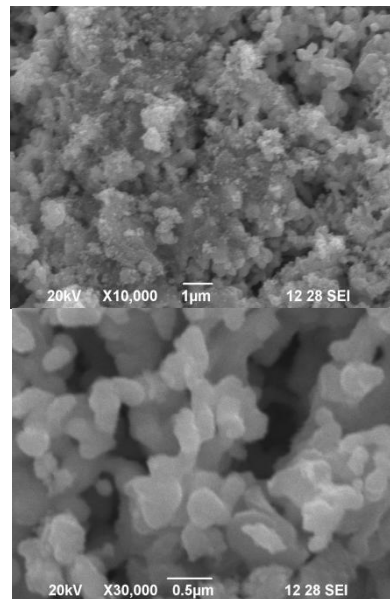


Fig. 3. SEM images of NiO nanoparticles

3.4. HRTEM Analysis

HRTEM images are shown in Fig. 4. They exhibit NiO nanoparticles having mean crystallite size of ~20nm.

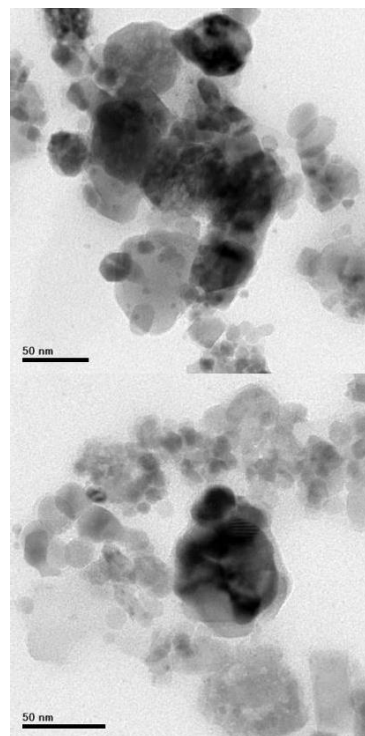


Fig. 4. HRTEM images of NiO nanoparticles

4. CONCLUSION

Highly-crystallized pure nickel oxide nanoparticles with a mean crystallite size of around 20 nm were synthesized by a microwave chemical approach. Morphology of the produced sample showed well-shaped homogeneously crystallized particles. Microwave irradiation has been found to be efficient in speeding up of the production rate and reduction of the size of particles.

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