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Synthesis of NiO Nanoparticles through Sol-gel Method

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Abstract

Nickel oxide (NiO) nanoparticles were synthesized through sol-gel method. The solution was controlled at pH 11 and the calcination temperature at 450 °C. The structure, morphology, and particles size of NiO were investigated. Structural analysis confirmed that the cubic structure of NiO was formed without impurity. Morphological and elemental analyses revealed the ratio of NiO, Ni and O. Morphological analysis showed NiO nanoparticles with an average diameter of approximately 32.9 nm.

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Introduction

Recently, NiO has been investigated as a type of important inorganic material[1]. The NiO is a crucial material that can be grown and used in a wide range of applications, such as solar cell[2], capacitor[3], and rechargeable lithium-ion batteries[4]. In addition, NiO nanoparticles have attracted and great attention because of their potential applications and their specific physical and chemical properties[1].

The structure, calcination temperature[1,5], and pH value;[1,6] of the solution must be controlled to produce pure NiO nanoparticles. These parameters affect the size[1,5], distribution[1] and morphology[1] of the particles. The specific physical and chemical properties of pure NiO can be determined if pure NiO is produced. Sol-gel is a suitable method to synthesize NiO nanoparticles because it exhibits homogeneoeus mixing[7], better crystallinity[7], uniform particle distribution[7], and smaller particle size[7].

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Several characterizations must be analyzed in the synthesis of NiO nanoparticles by sol-gel method. Thermal analyses, such as thermogravimetric analysis (TGA), are normally utilized to determine the calcination temperature. Different calcination temperatures will significantly influence particle size. Several calcination temperatures for NiO nanoparticles, including 400 °C[1, 5], 450 °C[1], 500 °C[1], 550 °C[1] and 600 °C[6], have been adopted. Meanwhile, structural analysis by X-ray diffraction (XRD) is applied to confirm the phase of NiO nanoparticles. Different of molar ratios of nickel nitrate/alcohol can significantly influence the reaction rate[8] and the final phases[8]. The distribution and shape of NiO nanoparticles can be observed through morphological analysis. A field emission scanning electron microscope (FESEM) is utilized to investigate the microstructure. Based on the SEM image, the average diameter for NiO nanoparticles are between 8.0 nm to 80.0 nm[1]. Elemental analysis is performed to confirm the composition of NiO nanoparticles. The typical ratio of NiO is 1:1. This analysis is conducted through energy dispersive X-ray (EDX) spectroscopy.

This work aims to synthesize pure NiO nanoparticles through sol-gel method. Several characterizations were conducted to ensure the quality of the synthesized NiO nanoparticles. Characterizations for thermal, structural, morphological, and elemental analyses are required to determine the synthesized NiO nanoparticles.

2. Experimental

2.1. Sample Preparation

Nickel (II) nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$, Merck] was dissolved in 20 ml of isopropanol alcohol [$(CH_3)_2CHOH$, Merck] and 20 ml of polyethylene glycol [$H(OCH_2CH_2)nOH$), Merck]. The solutions were stirred with a magnetic stirrer for 24 hours until chemically dissolved. Ammonium hydroxide (NH_4OH, Merck) was added until solutions reached pH 11. Triton X-100 [$C_{14}H_{22}O$ $(C_2H_4O)n$, Sigma Aldrich] was added to avoid particle agglomeration. The solutions were gradually heated at 80 °C until gel was formed. The gel was dried at 200 °C and then ground. The sample was ground again before thermal, structural, and morphological analyses. Thermogravimetric and differential scanning calorimetry (TGA/DSC) analyses (Mettlet Toledo) were conducted in air at 50 °C to 900 °C. Phase identification and structural analysis were performed by XRD (Bruker Advanced X-ray Solutions D9) in the 2θ range of 20° to 90° with monochromatized Cu Kα radiation (λ = 1.5406 Å). The morphologies of the NiO nanoparticles were observed directly by FESEM (Zeiss Supra™, 35VP) and EDX was implemented to analyze the elements of the sample.

3. Result and Discussion

3.1. Thermal Analysis

Thermal analysis of the dried sample after gelification were conducted by TGA to find the suitable calcination of the NiO formulation (Fig. 1). Minimal mass loss (8.0 %) was observed when the temperature exceeded 220 °C. The acceleratory stage occured in the temperature range of 220 °C to 400 °C, in which weight loss increased quickly to (68.0 %). Weight loss was constant at above 400 °C. Meanwhile, endothermic (60 °C and 400 °C) and exothermic (350 °C and 380 °C) peaks were observed in DSC curves (Fig. 1).

Below 220 °C, weight loss happened because of the evaporation of absorbed water. In the temperature range of 220 °C to 400 °C, weight loss is due to the decomposition of precursor materials. This finding suggests that the precursor decomposed completely at 400 °C to become nickel oxide[5]. Above 400 °C, constant weight loss occured because the decomposition reaction was almost completed. The endothermic reaction was attributed to the decomposition of water, whereas the exothermic reaction happened because of the oxidation of the decomposed product. Thus, the temperature at 450 °C was utilized for calcination.