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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 material¹. The NiO is a crucial material that can be grown and used in a wide range of applications, such as solar cell², capacitor³, and rechargeable lithium-ion batteries⁴. In addition, NiO nanoparticles have attracted and great attention because of their potential applications and their specific physical and chemical properties¹.

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}, distribution¹ and morphology¹ 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 homogeneous mixing⁷, better crystallinity⁷, uniform particle distribution⁷, and smaller particle size⁷.

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 °C¹, 500 °C¹, 550 °C¹ and 600 °C⁶, 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 rate⁸ and the final phases⁸. 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 nm¹. 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(NO₃)₂·6H₂O, Merck] was dissolved in 20 ml of isopropanol alcohol [(CH₃)₂CHOH, Merck] and 20 ml of polyethylene glycol [H(OCH₂CH₂)_nOH], Merck]. The solutions were stirred with a magnetic stirrer for 24 hours until chemically dissolved. Ammonium hydroxide (NH₄OH, Merck) was added until solutions reached pH 11. Triton X-100 [C₁₄H₂₂O (C₂H₄O)_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 SupraTM, 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 occurred 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 oxide⁵. Above 400 °C, constant weight loss occurred 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.

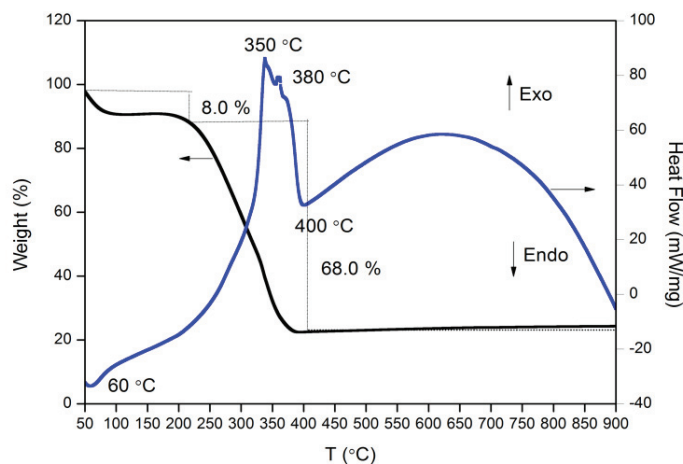


Fig. 1. TGA/DSC curves for the NiO nanoparticles

3.2. Structural Analysis

Further analysis was conducted by XRD to confirm the phase of the synthesized NiO nanoparticles (Fig. 2). Before calcination, the sample was in an amorphous phase and no NiO phase was observed. After calcination, crystalline phase presented a cubic structure, in which all diffraction lines of NiO were indexed to an ordered structure (ICSD 98-009-0203). At the crystalline phase, all diffraction peaks at (111), (200), (220), (311), and (222) were observed.

In the amorphous phase, no significant change in the nickel phase occurred. Thus, no NiO phase existed. The crystalline phase existed after calcination, demonstrating that the metallic-nickel nanoparticles were readily oxidized to NiO nanoparticles.

Quantitative XRD analysis was conducted to obtain the refined structural parameters, such as lattice parameter, crystal structure, and crystallite size. The matching between the diffracted and reference patterns (Table 1), using the R weight profile (R_{wp}) and the goodness of fit (GOF) values, were determined with the XRD Rietveld analysis of the NiO nanoparticles. The NiO nanoparticles obtained the cell parameters ($a = 4.1768 \text{ \AA}$, $b = 4.1768 \text{ \AA}$, $c = 4.1768 \text{ \AA}$). The calculated R_{wp} and GOF values are 3.826 and 1.695 (Table 1).

For the Rietveld analysis, all the refinements were rational when $R_{wp} < 4$ and $GOF < 2^9$. Thus, structural analysis confirmed the phase of the pure NiO nanoparticles and demonstrated that controlling the properties of NiO nanoparticles is an essential factor.

Table 1. Quantitative analysis of NiO nanoparticles using the Rietveld refinement method

	R expected (error factor)	R profile (reliability factor, R_p)	R weight profile (R_{wp})	Goodness of fit (GOF)
Before Calcination	-	-	-	-
After Calcination	2.939	3.424	3.826	1.695

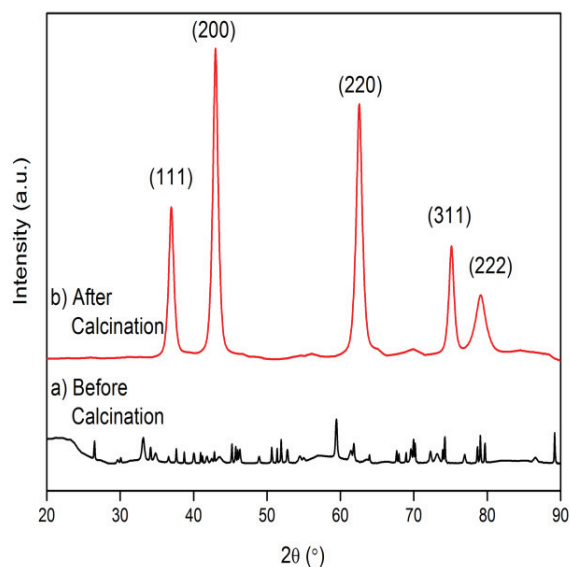


Fig. 2. XRD pattern of the NiO nanoparticle

3.3. Morphological and elemental analyses

At high magnification (50,000 x), the NiO nanoparticles in the SEM image showed a spherical nanoparticle structure (Fig. 3). The EDX results presented the elements Ni and O. The Ni content was 57.72 at % while O content 42.28 at % (Table 2).

Spherical nanosizes were successfully produced because of the suitable selection of calcination temperature, which significantly influences the particle size¹. Elemental analysis demonstrated that the synthesized NiO nanoparticles consisted of Ni and O with a molecular ratio of 1:1 and without any trace of other materials. Thus, nanoscale particles with an average diameter of about 32.9 nm were produced by selecting 450 °C as the optimum calcination temperature.

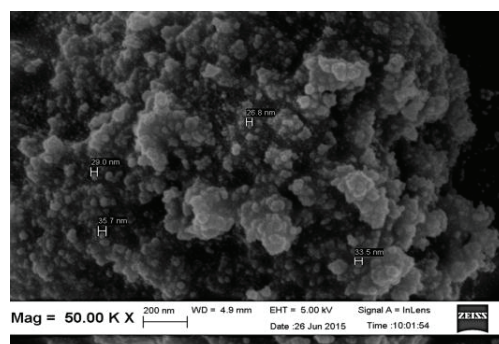


Fig. 3. SEM of the NiO nanoparticles

Table 2. Quantitative analysis of NiO using EDX

Element	Wt %	At %
O	16.64	42.28
Ni	83.36	57.72

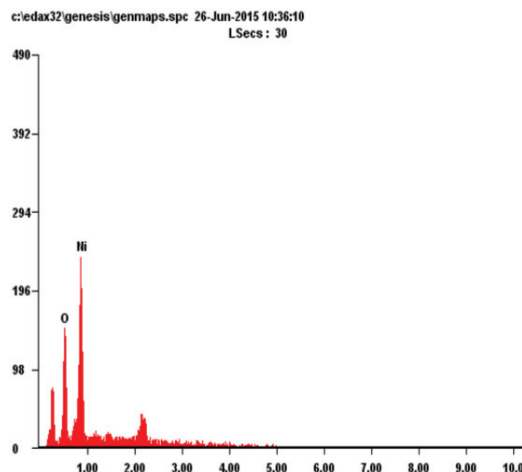


Fig. 4. EDX of the NiO nanoparticle

4. Conclusion

The NiO nanoparticles were successfully synthesized through the sol-gel method. The optimum calcination temperature of the NiO nanoparticles was determined by thermal analysis. A cubic structure was formed in the NiO nanoparticles. The sample showed that pure NiO nanoparticles were formed without any impurity. The morphological analysis was demonstrated in nanoscale range, and Ni and O elements were successfully traced.

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References

1. Wu Y, He Y, Wu T, Chen T, Weng W, Wan H. Influence of some parameters on the synthesis of nanosized NiO material by modified sol-gel method. *Mater Lett* 2007;**61**:3174-3178.
2. Bandara J, Weerasinghe H. Solid-state dye-sensitized solar cell with p-type NiO as a hole collector. *Sol Energy Mater Sol Cells* 2005;**85**:385-390.
3. Zhang F-b, Zhou Y-k, Li H-l. Nanocrystalline NiO as an electrode material for electrochemical capacitor. *Mater Chem Phys* 2004;**83**:260-264.
4. Kumar Rai A, Tuan Anh L, Park C-J, Kim J. Electrochemical study of NiO nanoparticles electrode for application in rechargeable lithium-ion batteries. *Ceram Int* 2013;**39**:6611-6618.
5. Sheena P. A, Priyanka K.P, Aloysius Sabu N, Sabu B, Varghese T. Effect of calcination temperature on the structural and optical properties of nickel oxide nanoparticles. *Phys Chem Math* 2014;**5**:441-449.

6. Palanisamy P, Raichur AM. Synthesis of spherical NiO nanoparticles through a novel biosurfactant mediated emulsion technique. *Mater Sci Eng, C* 2009;**29**:199-204.
7. Fu L, Liu H, Li C, Wu Y, Rahm E, Holze R, et al. Electrode materials for lithium secondary batteries prepared by sol–gel methods. *Prog Mater Sci* 2005;**50**:881-928.
8. Harraz FA, Mohamed RM, Shawky A, Ibrahim IA. Composition and phase control of Ni/NiO nanoparticles for photocatalytic degradation of EDTA. *J Alloys Comp* 2010;**508**:133-140.
9. Zhao Y, Cai F, Wang C, Chai Z, Zhu K, Xu Z, et al. Investigation on the evolution of microstructure and texture of electroplated Ni–Ti composite coating by rietveld method. *Appl Surf Sci* 2015;**353**:1023-1030.