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.

Keywords: Nickel Oxide; Sol-gel; Nanoparticle

Introduction

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

The structure, calcination temperature, and pH value of the solution must be controlled to produce pure NiO nanoparticles. These parameters affect the size, distribution and morphology 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 mixing, better crystallinity, uniform particle distribution, and smaller particle size.
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\(^1\), 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 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. 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\(_3\))\(_2\)-6H\(_2\)O, Merck] was dissolved in 20 ml of isopropanol alcohol [(CH\(_3\))\(_2\)CHOH, Merck] and 20 ml of polyethylene glycol [H(OCH\(_2\)CH\(_2\))\(_n\)OH), Merck]. The solutions were stirred with a magnetic stirrer for 24 hours until chemically dissolved. Ammonium hydroxide (NH\(_4\)OH, Merck) was added until solutions reached pH 11. Triton X-100 [C\(_{14}\)H\(_{22}\)O (C\(_2\)H\(_4\)O)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 2\(\theta\) range of 20° to 90° with monochromatized Cu K\(\alpha\) radiation (\(\lambda = 1.5406 \text{ Å}\)). The morphologies of the NiO nanoparticles were observed directly by FESEM (Zeiss Supra\textsuperscript{TM} 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 oxide\(^5\). 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.
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 ($a = 4.1768$ Å, $b = 4.1768$ Å, $c = 4.1768$ Å). 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 $< 2$. 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

<table>
<thead>
<tr>
<th></th>
<th>R expected (error factor)</th>
<th>R profile (reliability factor, $R_p$)</th>
<th>R weight profile ($R_{wp}$)</th>
<th>Goodness of fit (GOF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before Calcination</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>After Calcination</td>
<td>2.939</td>
<td>3.424</td>
<td>3.826</td>
<td>1.695</td>
</tr>
</tbody>
</table>
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 size. 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.
Table 2. Quantitative analysis of NiO using EDX

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt %</th>
<th>At %</th>
</tr>
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<tbody>
<tr>
<td>O</td>
<td>16.64</td>
<td>42.28</td>
</tr>
<tr>
<td>Ni</td>
<td>83.36</td>
<td>57.72</td>
</tr>
</tbody>
</table>

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


