Synthesis and Characterization of Ni$_2$O$_3$ as a Phase of Nickel Oxide Nanomaterial

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Received: 16/10/2021 Accepted: 10/2/2022 Published: 30/11/2022

Abstract

Ni$_2$O$_3$ nanomaterial, a phase of nickel oxide, is synthesized by a simple chemical process. The pure raw materials used in the present process were nickel chloride hexahydrate NiCl$_2$·6H$_2$O and potassium hydroxide KOH by utilizing temperature at 250 °C for 2 hour. The structural, morphological and optical properties of the synthesized specimens of Ni$_2$O$_3$ were investigated employing diverse techniques such as XRD, AFM, SEM and UV-vis., respectively. The XRD technique confirms the presence of Ni$_2$O$_3$ nanomaterial with crystal size of 57.083 nm which indexing to the (2θ) of 31.82; this results revealed the Ni$_2$O$_3$ was a phase of nickel oxide with Nano structure. The synthesized Ni$_2$O$_3$ will be useful in manufacturing electrodes materials for fuel cell and production catalytic materials for electrolysis cell.

Keywords: Nickel Oxide, Nanomaterials, Chemical Method, Crystallite Size.
1. Introduction
The nanotechnology ascribe to hybrid sciences of chemical engineering branch [1]. The promising trends of nanotechnology is its use in electrolysis and fuel cells [2].

Nanomaterials have extensively attracted interest in comparison with their bulk counterparts because of their spectacular and unique (mechanical, electronical, optical, thermal, catalytic and magnetically) characteristics [3, 4, 5, 6, 7].

Among the various nanomaterials, metal oxides have gained considerable attention from researchers and scientists world-wide owing to their various and promised applications in energy supply, environment remediation, synthesis catalysts, materials science, medicine and industrial inspection [8, 9, 10, 11, 12].

In addition to metal oxide diverse applications, they have novel structural properties such as large surface area, unusual adsorptive properties, fast diffusivity [5, 11] and interesting (electrical, optical, thermal, catalytic, mechanical and magnetically) features [11, 13]. Transition metals oxides, in particular, are most attractive because of their high stability, cost-effective and high oxygen carrying capacity [14].

Transition metals oxides are prepared using different methods such as reductions of metallic salts followed by the oxidation of metallic species, electrodeposition, pulsed laser ablation, ultrasonic spray pyrolysis, chemical vapour deposition, liquid control precipitation, sol-gel route and hydrothermal technique [4, 13]. In this context, nickel oxide is a transition metal oxide [1] that has a rock salt structure [9] which submits the characteristic of P-type semiconductor with a broad energy band gap of (3.6 - 4) eV [10, 15].

Nickel oxide nanostructure is chemically stable and has high electrical conductivity also exceptional (optical, electronical and magnetically) characteristics [9, 10, 15]. Furthermore, it has ultra-fine structure with a uniform size and well dispersion [11, 13]. It has attracted a great attention because of its wide applications such as in manufacturing magnetic materials [5, 6, 7], gas sensors [1, 10], photovoltaic devices [9, 15], fuel cell [16, 17, 18] and catalytic materials [10, 11, 13].

Various methods have been adopted to synthesize nickel oxide nanomaterial such as sol-gel, spray pyrolysis, hydrothermal, chemical precipitation and pulsed laser ablation [9, 10, 11]. Most experimental methods are still limited because of low yield, high cost and high calcination temperature of at least 250° C [11, 13, 14].

The main purpose of the present work is the synthesis of NiO as a phase of nickel oxide nanomaterial using a simple chemical process of high yield with low cost, since it uses raw materials that are available and inexpensive.

2. Experiment procedure
2.1 Materials
Nickel chloride hexahydrate NiCl₂.6H₂O from Sigma and potassium hydroxide KOH from Alpha are the raw materials used for current work. The chemical materials used were analytic. Distilled water was used throughout preparation.
2.2 Synthesis of Ni$_2$O$_3$

Ni$_2$O$_3$ was synthesized using 5.34 g of NiCl$_2$·6H$_2$O dissolved at room temperature in 250 ml of distilled water as a solvent. The obtained solution was kept on a hot plate magnetic stirrer at 50 °C for 15 minutes.

Thereafter, 1.97 g of KOH was added to the solution with continuous stirring until a pH of 8 was reached. After 2 hours of magnetic stirring at 250°C and 500 rpm, a green solution was formed as shown in Figure 1.

![Synthesis process of Ni$_2$O$_3$](image)

**Figure 1:** Image of the synthesis process of Ni$_2$O$_3$

The temperature of the obtained solution was gradually reduced to the same environment temperature. The formation of the green solution indicates the synthesis of Ni$_2$O$_3$ material. The final product of Ni$_2$O$_3$ was stored in a tight container for further analysis.

2.3 Characterization techniques

The structural properties of the synthesized samples of Ni$_2$O$_3$ were accomplished using X-ray diffractometer (type Lab X (XRD 6000) Shimadzu). The X-ray generator was operated with 40 kV and 30 mA with a wavelength radiation of 1.5440 Å of Cu-Kα as source. For the optical properties, the UV-vis. spectrophotometer (type of (UV-1800) Shimadzu) was used at the wavelength range (190-1100) nm.

3. Result and discussion

3.1 Structural properties

The XRD pattern obtained for the Ni$_2$O$_3$ specimen is shown in Figure 2. The result revealed the distinctive peak at (2θ) of 31.82 corresponding to (002) plane. The peak was identified as that of Ni$_2$O$_3$ with hexagonal crystalline structure. This result confirms that the synthesized Ni$_2$O$_3$ is a phase of nickel oxide according to the standard spectrum (JCPDS, Card No. 14-0481).

The average crystal size of synthesized Ni$_2$O$_3$ was estimated by Scherrer relation, indexing to the (2θ) of 31.82, was (57.083 nm).
3.2 Morphological properties

The surface morphological features of Ni$_2$O$_3$ thin film deposit at room temperature, using drop casting technique, was performed via an Atomic Force Microscope (AFM) and a Field Emission Scanning Electron Microscope (FESEM).

Figure 3 (A and B) shows a three dimension AFM image and the histogram for Ni$_2$O$_3$. One can observe that the surface is not highly uniform, very highly spaced and randomly oriented with an average grain size around (20-45) nm; this result is comparable with the XRD result.

The FESEM images (with various magnification) of the synthesized Ni$_2$O$_3$ are shown in Figure 4. The result showed that the synthesized Ni$_2$O$_3$ material have a spherical grain shape with homogenous distribution and the grain size of Ni$_2$O$_3$ is in the Nano size range. Moreover, the chemical stability of nickel oxide with the phase of Ni$_2$O$_3$ in addition to the low temperature used in the preparation and deposition of the Ni$_2$O$_3$ sample prevented the
Ni$_2$O$_3$ molecules from bonding with each other and not accumulating this prevented aggregations and cracks, as the FESEM images clarified.

**Figure 4:** FESEM images of the synthesized Ni$_2$O$_3$

### 3.3 Optical properties

The UV-vis. spectrum (Figure 5) was performed to study the optical characteristics of the synthesized Ni$_2$O$_3$. The absorption spectrum clarified the spectral behaviour of the synthesized Ni$_2$O$_3$ which covered different spectral regions (UV and Visible).

The figure also shows a prominent peak around 775 nm. This absorption peak is related to the decrease of energy band gap due to the quantum confinement effect of nanostructure. This result confirms the Nano size of the synthesized Ni$_2$O$_3$. This result is in agreement with the results obtained from the characterization techniques performed in the present work.

**Figure 5:** The UV-vis. spectrum of synthesized Ni$_2$O$_3$
4. Conclusion

Ni$_2$O$_3$ nanomaterial was easily produced in the current research by using a fast and efficient process such as the simple chemical method. This process eliminates the need for high calcination temperature, which is extensively used in most previous studies. This process synthesized nanostructure Ni$_2$O$_3$, as was confirmed by the results obtained from characterization techniques.

Therefore, the method which have been reviewed and performed is a novel, rapid and low-cost process with a large harvesting amount of Ni$_2$O$_3$ nanomaterial.

5. Acknowledgements

Authors of this research work are grateful and sincere gratitude for the scientific support of Dr. Hind Hadi and Dr. Haleemah Jaber Mohammed.

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