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Synthesis and Characterization of Ni_2O_3 as a Phase of Nickel Oxide Nanomaterial

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Abstract
Ni_2O_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 $NiCl_2.6H_2O$ and potassium hydroxide KOH by utilizing temperature at 250 ^oC for 2 hour. The structural, morphological and optical properties of the synthesized specimens of Ni_2O_3 were investigated employing diverse techniques such as XRD, AFM, SEM and UV-vis., respectively. The XRD technique confirms the presence of Ni_2O_3 nanomaterial with crystal size of 57.083 nm which indexing to the (2θ) of 31.82; this results revealed the Ni_2O_3 was a phase of nickel oxide with Nano structure. The synthesized Ni_2O_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.

تصنيع وتوصيف مادة Ni_2O_3 كطور من مادة النيكل اوكسايد النانوية

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الخلاصة
تم في هذا البحث تصنيع مادة Ni_2O_3 النانوية والتي هي طور من اطوار مادة اوكسيد النيكل بطريقة كيميائية بسيطة. تم استخدام مادة كلوريد النيكل المائي $NiCl_2.6H_2O$ ومادة هيدروكسيد البوتاسيوم KOH كمواد اساسية لهذا البحث وباستخدام درجة حرارة 250 مْ لمدة ساعتين . ولتشخيص الخصائص التركيبية ، المورفولوجية ، والبصرية للنماذج المصنعة تم توظيف العديد من من التقنيات مثل حيود الاشعة السينية XRD ، مجهر القوة الذرية AFM ، المجهر الالكتروني الماسح SEM ، ومطياف الاشعة فوق البنفسجية والمرئية -UV vis. على التوالي . اكدت نتائج تقنية XRD وجود مادة Ni_2O_3 بتركيبها النانوي وبحجم بلورة 57.083 نانومتر عند زاوية الحيود 31,82 وهذه النتيجة كشفت انه Ni_2O_3 هو طور لمادة اوكسيد النيكل وانها ذات تركيب نانوي . من الجدير بالذكر ان مادة Ni_2O_3 المصنعة مفيدة جدا لتصنيع المواد المستخدمة لتصنيع اقطاب خلايا الوقود وايضا صناعة المواد المحفزة في خلايا التحليل .

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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^oC [11, 13, 14].

The main purpose of the present work is the synthesis of Ni_2O_3 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 $NiCl_2.6H_2O$ 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_2O_3

Ni_2O_3 was synthesized using 5.34 g of $NiCl_2.6H_2O$ 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 ^oC for 15 minute.

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^oC and 500 rpm, a green solution was formed as shown in Figure 1.

Figure 1: Image of the synthesis process of Ni_2O_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_2O_3 material. The final product of Ni_2O_3 was stored in a tight container for further analysis.

2.3 Characterization technique

The structural properties of the synthesized samples of Ni_2O_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_2O_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_2O_3 with hexagonal crystalline structure. This result confirm that the synthesized Ni_2O_3 is a phase of nickel oxide according the standard spectrum (JCPDS, Card No. 14-0481).

The average crystal size of synthesized Ni_2O_3 was estimated by Scherrer relation, indexing to the (2θ) of 31.82, was (57.083 nm).

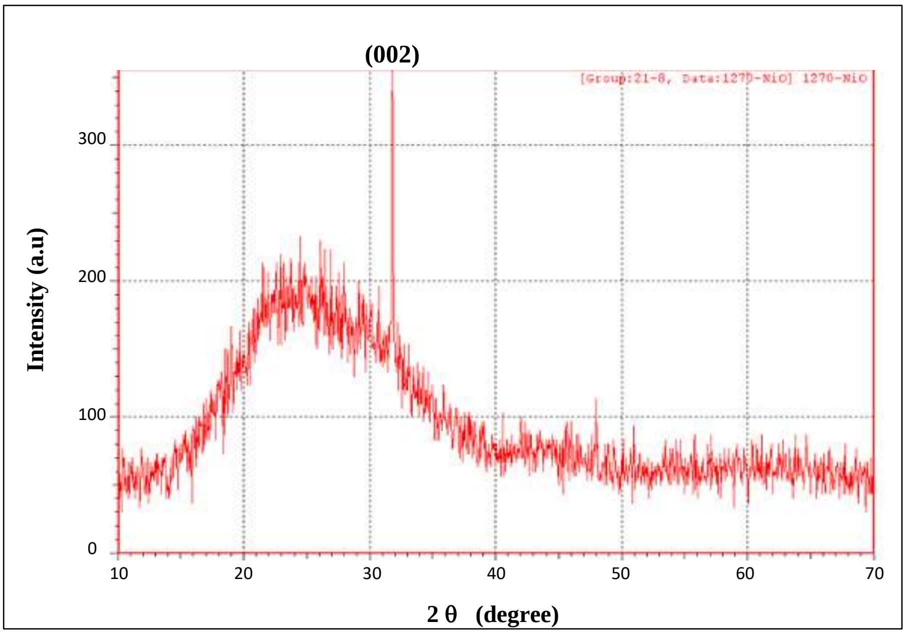
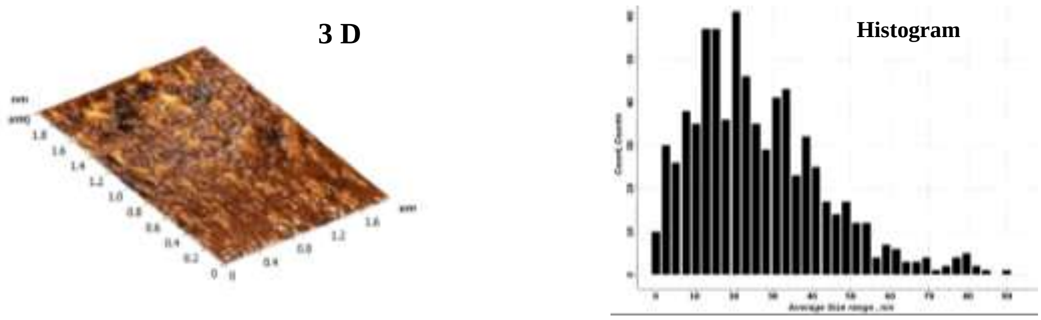


Figure 2: XRD pattern of synthesized Ni_2O_3

3.2 Morphological properties

The surface morphological features of Ni_2O_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_2O_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.



A

B

Figure 3: AFM images of synthesized Ni_2O_3 (A. Three dimension and B. Histogram).

The FESEM images (with various magnification) of the synthesized Ni_2O_3 are shown in Figure 4. The result showed that the synthesized Ni_2O_3 material have a spherical grain shape with homogenous distribution and the grain size of Ni_2O_3 is in the Nano size range. Moreover, the chemical stability of nickel oxide with the phase of Ni_2O_3 in addition to the low temperature used in the preparation and deposition of the Ni_2O_3 sample prevented the

Ni_2O_3 molecules from bonding with each other and not accumulating this prevented aggregations and cracks, as the FESEM images clarified.

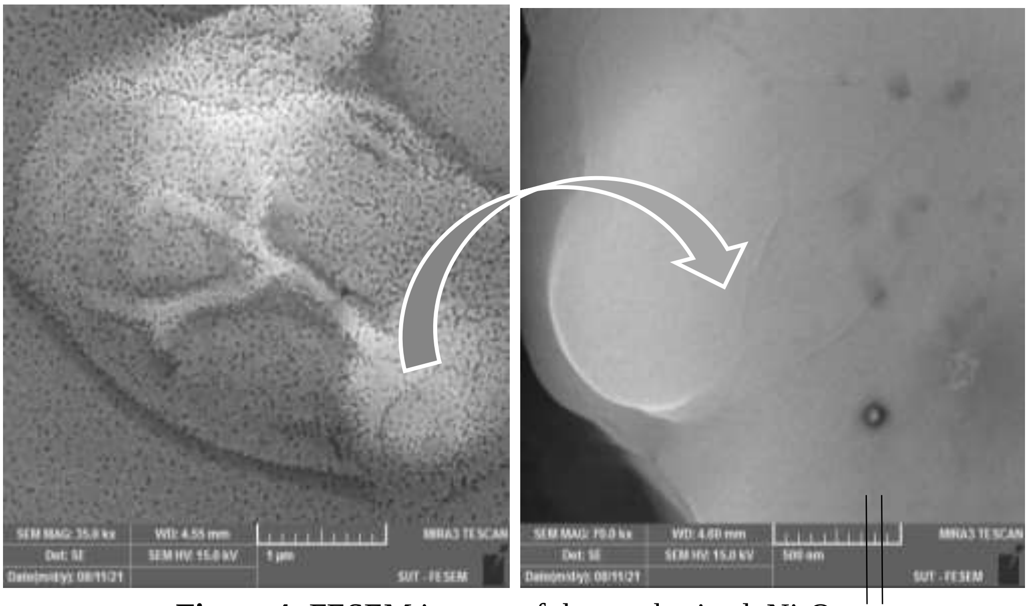


Figure 4: FESEM images of the synthesized Ni_2O_3

3.3 Optical properties

The UV-vis. spectrum (Figure 5) was performed to study the optical characteristics of the synthesized Ni_2O_3. The absorption spectrum clarified the spectral behaviour of the synthesized Ni_2O_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_2O_3. This result is in agreement with the results obtained from the characterization techniques performed in the present work.

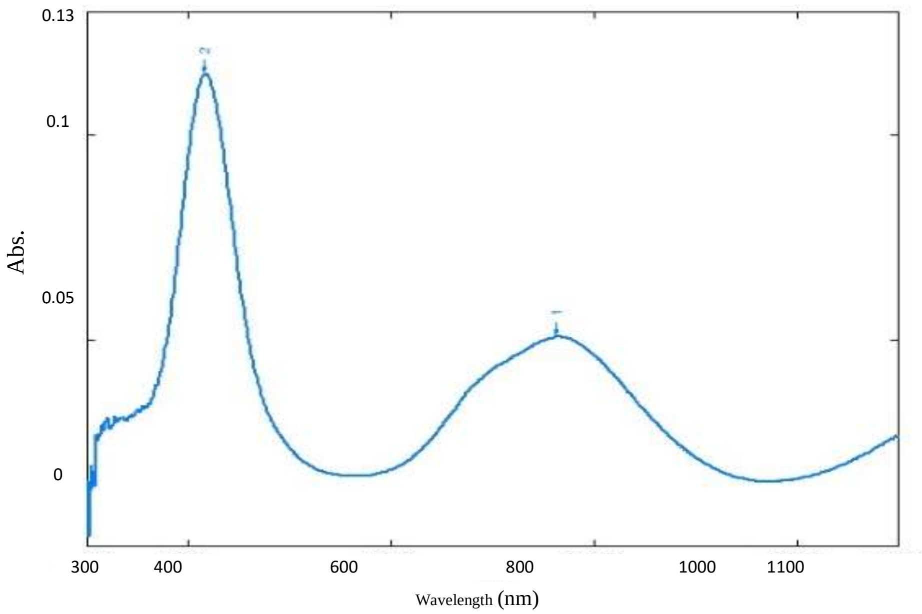


Figure 5: The UV-vis. spectrum of synthesized Ni_2O_3

4. Conclusion

Ni_2O_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_2O_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_2O_3 nanomaterial.

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