Structural Characterization of NiO Nanoparticles Prepared by Green Chemistry Synthesis Using Arundo donaxi Leaves Extract

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Abstract. The present work includes nickel oxide nanoparticles (NiO) synthesis via a green route using its salt and Arundo donaxi leaves extracts solution. Thus prepared particles were characterized by X-ray diffraction (XRD), spectrum of Fourier transform infrared, (FTIR) and then atomic force microscope (AFM) techniques. XRD data indicated that the particle size was in a nanoscale region and around 30 nm which seem to be agreed well with the one estimated from AFM. The surface of the nanoparticles studies using AFM demonstrated that the average particle size is 41.13 nm and also indicated that 10%, 50% and 90% of the prepared samples have a particle size being less than 34 nm, 44 nm and 48 nm respectively.

Keywords: Nickel oxide nanoparticles, Arundo donaxi leaves extracts, Characterization, Green synthesis.

1. Introduction
Nanomaterials are chemical substances or materials that are manufactured and used at a very small scale. Nanomaterials are developed to exhibit novel characteristics compared to the same material without nanoscale features, such as increased strength, chemical reactivity or conductivity. These have various applications in optical; electrical; mechanical devices; and catalysts, [1]. Many chemical / physical routes used in their synthesis including sol-gel; precipitation; electro; thermal; electrochemical methods; combustion; microwave, and pulsed explosion methods [2] and [3]. Most of these are complex and complicated and were drawbacks like use of hazardous solvents, expensive and costly reagents, deliver toxic by product, severe and drastic reaction conditions, difficulties to separate nanoparticles and also longer time require in their synthesis [4], therefore there is a need for essential procedure to develop environmentally friendly one to synthesize metal oxides nanoparticles and nanocatalyst in particular [5]. Now, the renewable nature of plant extracts and eco-friendly aqueous media and simple and mild reaction conditions make theses methods advantageous with other hazardous methods if compared [6]. In the last few years a different plant extract and / or their products
have been received attention because of its low cost; energy-efficient and also have nontoxic behaviour in approach in synthesis of nanoparticles [7]. The properties of green synthesis being usually of low cost [8] as the role of the plant extract is its reduction and conversion of the metal salts to its corresponding metal oxide nanoparticles [9].

Nickel oxide is one of the important metal oxide and has has attracted recent research due to its low cost; abundant availability; as well as reflect particular properties. Different nanostructures of NiO are synthesized and characterized and using anodic arc plasma method, co-precipitation method [10] and [11] employed extract of Neem leaves mediated preparation of NiO nanoparticles and its magnetization; coercivity; antibacterial analysis and plant based synthesis of NiO nanoparticles using salvia macrosiphon boiss /extract and examining their water treatment by [12]. Green synthesis process of NiO-nanoparticles via using the extract of Moringa oleifera with their biomedical application were covered by [13] while synthesize NiO-nanoparticles via green route using Ageratum conyzoides leaf extract and studied their catalytic behaviour and finally phytopgenic generation of NiO-nanoparticles using Stevia leaf extract with evaluation of its In-Vitro antioxidant, antimicrobial properties was studied well by [15]. *Arundo donax* is known as a perennial grass which can reach up to 20 ft (6.1 m) height and it can be much shorter when damaged or stressed, the stem resembles a corn stalk. Its Leaves are long; flat and can grow up to 1.5 ft (0.5 m) in length. They can be green or have variegated green. Threat *Arundo donax* invades wet lands such as ditches, stream banks and lake shores.

The aim of the present investigation is the synthesis of NiO nanoparticle, NiONPs via environmentally-friendly green route from its corresponding salt and extract of *Arundo donax* leaves (ADLE) and also characterize it.

2. Experimental details

**Materials used.** Analytical grade chemicals were used with no need for any purification in addition to deionized distilled water, *Arundo donax* extract, nickel (II) chloride hexahydrate (NiCl₂·6H₂O), 0.1 M sodium hydroxide (NaOH) and pure ethanol (C₂H₅OH).

3. Nickel oxide nanoparticles preparation

**Preparation of arundo leaves extract.**

*Arundo donax* leaves collected from a river side in a Diyala governorate were cleaned from suspended dirt, washed with distilled water several times and then dried in shade. They were then being ground with grinder and stored away from wet, a quantity of (5 g) of this powder was added to (400 ml) of deionized water, boiled for (30 minutes) till the color of solution change, thus obtained solution was cooled to room temperature, filtered and centrifuged the filtrate at (1200 rpm) for two minutes for removing biomaterials and finally stored the extract at room temperature until it use.

4. NiO nanoparticles synthesis

Dissolve (0.5 g) of nickel chloride hexahydrate (NiCl₂·6H₂O) in 400 ml of deionized water using continuous stirring and then 10 ml of ADLE was then added gradually with a continuous stirring also and at room temperature, then adjust the pH of the solution by sodium hydroxide (0.1 M) until precipitate with a light green color form, then it is filtered, washed with deionized water for several times and with ethanol solvent to remove present impurities and finally dry in oven at (70°C) for two hours. (Fig.1 and Fig.2) show steps and flow diagram for the process to prepare nickel (II) nanoparticles using ADLE.
5. Characterization of nickel oxide nanoparticles

The X-ray diffraction pattern of the prepared oxide were recorded using XRD-6000 with CuKα (λ=1.5406Å) that have an accelerating voltage of 220/50 HZ which is produce by SHIMADZU company. Atomic force microscopy (AFM) used to study surface morphology of the samples was AFM model AA 3000 SPM 220 V- angstrom Advanced INC USA, and the FTIR spectra were recorded with 65 FTIR Perkin Elmer Spectrophotometer, Waltham, Massachusetts, USA over the range of 4000–400 cm⁻¹.

6. Results and Discussion

Preparation of nickel oxide nanoparticles. The ADLE acts as a reducing agent [1] by containing a high amount of polyphenols and other organic groups which take part in reaction mechanism involving reduction of precursor to metal nano-particle in two steps [18]. First precursor forms a complex by breaking the OH bond and forming a partial bond with a metal ion. Second, there is breakage of the partial bond and the transfer of electrons to form the
metal hydroxide which is reacted with OH$^-$ of sodium hydroxide to nickel oxide nanoparticles and liberated H$_2$O and thus itself get oxidised to orthoquinone. Elemental analysis of prepared NiO nanoparticles

Atomic absorption spectroscopy was used determine the nickel content and then oxygen content of thus prepared nanoparticle which show that Ni : O constitutes 78.55 : 21.45 and 67.75 : 32.25 as a weight and atomic ratios respectively.

**Fig.3.** Show the mechanism of preraration of NiO

**7. X-ray Diffraction analysis**

The XRD technique was used to determine and confirm the crystal structure of the prepared nanoparticles. XRD pattern of as prepared nickel (II) oxide nanoparticles is shown in (Fig.4) with the data of strongest three peaks shown in (Table 1) The peaks position of the samples exhibited the monoclinic structure and single phase of NiO nanoparticles and are in a good agreements with those reported in JCPDS file (NO.48-1548), no other impurity peak was observed in the XRD patterns. The broadening of the diffraction peaks indicates that the crystal size is small.

The particle sizes were calculated from formula given below [16]

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \quad \text{............... (1)}$$

Where: D is the crystallite size, $\lambda$ is the wave length of radiation, $\theta$ is the Bragg’s angle, $\beta$ is the full width at half maximum (FWHM).

The calculated particle size is 30 nm); the presence of sharp peaks in the XRD and particle size being less than 100 nm refers to the Nano-crystalline nature.
Table 1. The strongest three peaks in XRD spectrum of prepared NiO nanoparticles

| No. | 2θ (deg) | d (Å)    | FWHM (deg) | Intensity (counts) |
|-----|----------|----------|------------|-------------------|
| 1   | 33.18    | 2.6990   | 0.796      | 131               |
| 2   | 38.36    | 2.3591   | 3.66398    | 96                |
| 3   | 37.22    | 2.3819   | 14.96341   | 84                |

Fig.4. XRD pattern of prepared nickel oxides nanoparticle

8. Fourier transform infrared spectrum.
Figure 5 shows the FTIR spectra of NiO nanoparticles, which show several significant absorption peaks. The broad absorption band in the region of 600–700 cm$^{-1}$ is assigned to Ni–O stretching vibration mode [17] and [10], the broadness of the absorption band indicates that the NiO powders are nanocrystals. The size of samples used in this study was much less than the bulk form NiO, so that NiO nanoparticles had its IR peak of Ni–O stretching vibration and shifted to blue direction. Due to their quantum size effect and spherical nanostructures, the FTIR absorption of NiO nanoparticles is blue-shifted compared to that of the bulk form.

It can be seen also in Figure 4 that the FTIR spectra of NiO nanoparticles showed broad absorption band centered at 3440 cm$^{-1}$ which is attributable to the band O–H stretching vibrations and weak band near 1635 cm$^{-1}$ being assigned to H–O–H bending vibrations mode were due to the adsorption of water in air when FTIR sample disks were prepared in an open air. These observations provided the evidence to the effect of hydration in the structure. Mean while, it implied the presence of hydroxyl in the precursor, and the broad absorption at around 767 cm$^{-1}$ is assigned to the bond C=O stretching vibrations. The serrated absorption bands in the region of 1000–1500 cm$^{-1}$ are assigned to the O–C=O symmetric and asymmetric stretching vibrations and the C–O stretching vibration [17] and [10], but the intensity of the band has weakened, which indicated that the ultrafine powers tend to strong physically absorbed H$_2$O and CO$_2$.
Fig. 5. FTIR of prepared nickel (II) oxide nanoparticles

9. Atomic force microscope

The surface morphology and average grain size distribution of prepared nickel oxide nanoparticles was studied utilizing atomic force microscope [9]. Fig. 6 is typical AFM images of the NiO nanoparticles, it shows images measured with size = 2032 X 2027 nm, and ability analytical pixel = 392, 39.(Fig. 6-A) is the AFM images in three dimensions (3D), it explain structure shape for grain.

In (fig.6-B) is the images in two dimensions (2D), it is found that average roughness is (0.311 nm). The Root mean square (RMS) is (0.3581 nm). Table 2 and Fig. 7) show the granularity cumulating distribution and average diameter data which demonstrated that the average particle size is 41.13 nm and also indicated that 10%, 50% and 90% of the prepared nano NiO have a particle size being less than 34nm, 44nm and 48nm respectively.

Fig. 6. AFM images of prepared nickel (II) oxide nanoparticles
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![Diagram](image)

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