



Studying The Structural Properties of Nickle Oxide Nanoparticles Prepared Using Cellulose Extract by Ultrasonic Method for Photocatalyst Application

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Abstract

Nickel oxide nanoparticles (NiO NPs) were prepared using cellulose extract with 1 M NiCH₃ salt by ultrasonic method at 600°C. Prepared NiO NPs were characterized by x-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FE-SEM). The XRD results confirmed that prepared NiO NPs are crystalline with face center cubic (FCC) and the crystallite sized was 9 nm. FE-SEM measurements show that the particle size of NiO NPs ranges from 40.19 nm to 73 nm with the nano-ball structure. Photocatalytic activity of NiO NPs has been studied through degradation ratio of methylene blue (MB) dye under different illumination time by UV-light. The degradation ratio of MB dye degradation increased from 40% to 90% when illumination time was increased from 15 to 40min.

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1. Introduction

Establishing environment-friendly nano-particulate technology will be a welcome step in the modern scientific era. Much research that concerns nanotechnology aims to create a new material with different physical methods. Nanoparticles (NPs) can be defined as small particles with a diameter ranging less than 100 nm in dimensions, synthetic of inorganic or organic materials, and having new properties that contrast to the bulk materials [1]. Among the NPs, nickel oxide nanoparticles (NiO NPs) are significant materials that have great future advantages compared with other materials [2,3], special physiochemical properties (such as low toxicity), high catalytic activity, small size, and different physical properties [4,5].

Green synthesis of nickel oxide NPs using plant extracts as biological components has numerous benefits, such as cost-effectiveness, atom economy, simplicity, benign nature, nontoxicity, elimination of toxic and dangerous materials, easy availability, and easy removal of toxic substances for environmental remediation [6-9]. Cellulose by-products are rich in potentially valuable proteins, minerals, enzymes, pigments, and flavors. Off-the-fish by-product fish mucus is considered more valuable and has been reported to contain antimicrobial proteins. In cellulose, the epidermal mucus is considered a key component of innate immunity and plays a role in the prevention of colonization by parasites, bacteria, and fungi [10-11].

Preparation of NiO NPs is an important for different application specially in the industrial. In general, laboratory samples must be liquefied before analysis. Ultrasonic homogenization and dissolving is a fast and reliable method for preparing samples of all sizes. In industrial production, preparing homogeneous and well-solved solutions is often the determining factor to guarantee consistent product characteristics and quality. Compact lab devices and complete industrial and commercial ultrasound dissolvers are also available. Ultrasound is a well-known, reliable tool for preparing samples in the laboratory. Typical applications include homogenization, emulsification, dispersion, extraction, vacuum, and engineering treatments [12-14].

The NiO NPs were prepared using the ultrasonic method at 600°C of (cellulose) extract. Structural properties, morphology as well as photocatalyst activity under UV-light of prepared NiO NPs are studied.

2. Experiments

2.1 Materials

NiCH₃ salt and (cellulose) extract were purchased from a local market (Baghdad, Iraq). All

the experiment's glass wares that were used in this study were from Borosil.



2.2 Preparation of cellulose extract

Cellulose extract was prepared from a 5 g mixture powdered in 100 mL deionized water. The solution was boiled at 70°C for 1 hour on a magnetic stirrer. The resulting sol was frozen at room temperature and purified using a Whatman filter sheet. Figure 1 explains the steps of converting the (cellulose) in extract.

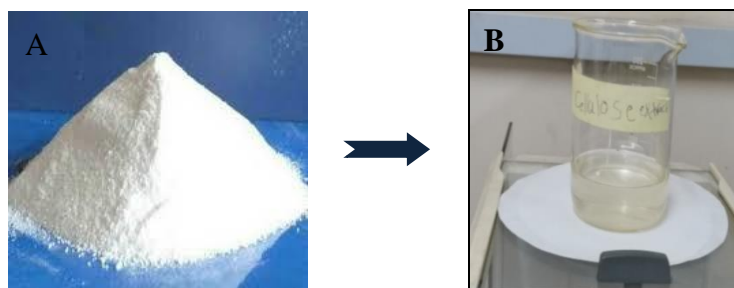


Figure 1: Cellulose in extract, (A) the cellulose, (B) cellulose extract.

2.3 Preparation of NiO NPs from cellulose extract by ultrasonic method

The NiO NPs synthesized were prepared adding (50 mL, 1 M) of NiCH₃ solution into 100 mL of cellulose extract. Then, this solution was placed on a magnetic stirrer at 70°C for 40 minutes. Through this method, the color of the reaction solution changes refer to the formation of NiO NPs. Next, the solution was left cooled under at room temperature. After that, 50 mL of the solution of NiO NPs was put in a ceramic eyelid in the oven at 600°C for 3 hours to obtain a nano powder. Figure 2 explains the stages of preparation of NiO NPs using cellulose extract.

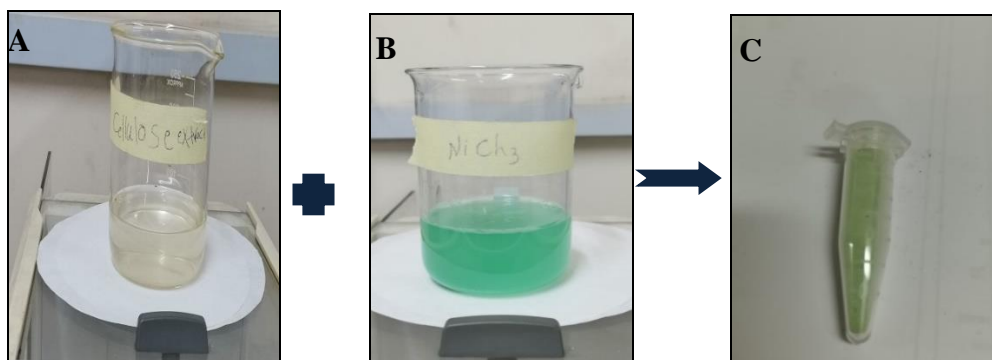


Figure 2: Preparation stages NiO NPs using (A) cellulose extract, (B) NiCH₃ extract, and (C) Obtained NiO NPs powder.

2.4 Characterization of NiO NPs

The XRD pattern of prepared NiO NPs using (XRD-6000, Shimadzu) by range from (10 to 80) degree. which operated at a speed of 40 kV and 30 MA with the values of the Joint Committee

on Powder Diffraction Standards (JPDS). FE-SEM provides details for the morphology and size distribution of synthesized NPs. The samples were analyzed in Iran-Mashhad by SEM (Tescan Mira3 FE-SEM-Czech Republic).

2.5 Photocatalytic activity for degradation of MB dye

In order to obtain a final concentration of 10 mg/L, the photocatalytic activity of iron oxide NPs was assessed by the mixing of a known amount of methylene blue (MB) (1 mg, 3 digestible 10-5 M) with deionized water of 100 mL. The mixture was then spread over a glass beaker with 3 mg of NiO NP powder, combined with a magnetic stirrer in the darkness for 10 minutes to keep the NiO NP suspension in place. Finally, the 10-minute centrifuge was made of around 4 mL of suspension, and the absorption was determined by UV-Vis (Shimadzu, UV-1800) spectrophotometer using the absorption limit $\alpha = 664$ nm. The absorption maximum was monitored. The interaction between the adsorbent substance and the dye molecules (the adsorbent substance) is primarily associated with changes in the adsorbent dye molecules and the region. The equation used to determine the efficiency of MB teal degradation with equation (1):

$$\text{The degradation efficiency (\%)} = \left[C_{\text{ini}} - \frac{C_{\text{fin}}}{C_{\text{ini}}} \right] \times 100 \% \quad (1)$$

Where, C_{ini} = initial concentration of (MB) dye, C_{fin} = final concentration of (MB) dye.

3. Results and Discussion

3.1 XRD measurements

XRD patterns of NiO NPs show that peaks are not found and not crystallite of the NiO NPs when using cellulose extract using ultrasonic method 0.5 M at 600°C for 3 hours, as shown in Figure 3 (A) [15], whereas in Figure 3 (B) it is observed that peaks can be indexed in agreement to the predictable (fcc) structure of NiO NPs. The peaks showed a crystalline structure in the (220), (311), (111), and (216) according to NiO NPs (JCPDS 47-1049), as shown in Fig. 3; this result is similar to [16-17].

The crystallite sizes are estimated by the following Scherrer's equation(2) [18-19].

$$D = K \cdot \lambda / \beta \cos \theta \quad (2)$$

where k is called shape factor (0.9), λ is the wavelength (0.15418), ($\text{CuK}\alpha$), β is full width at half maximum (FWHM), and θ is a diffraction angle.



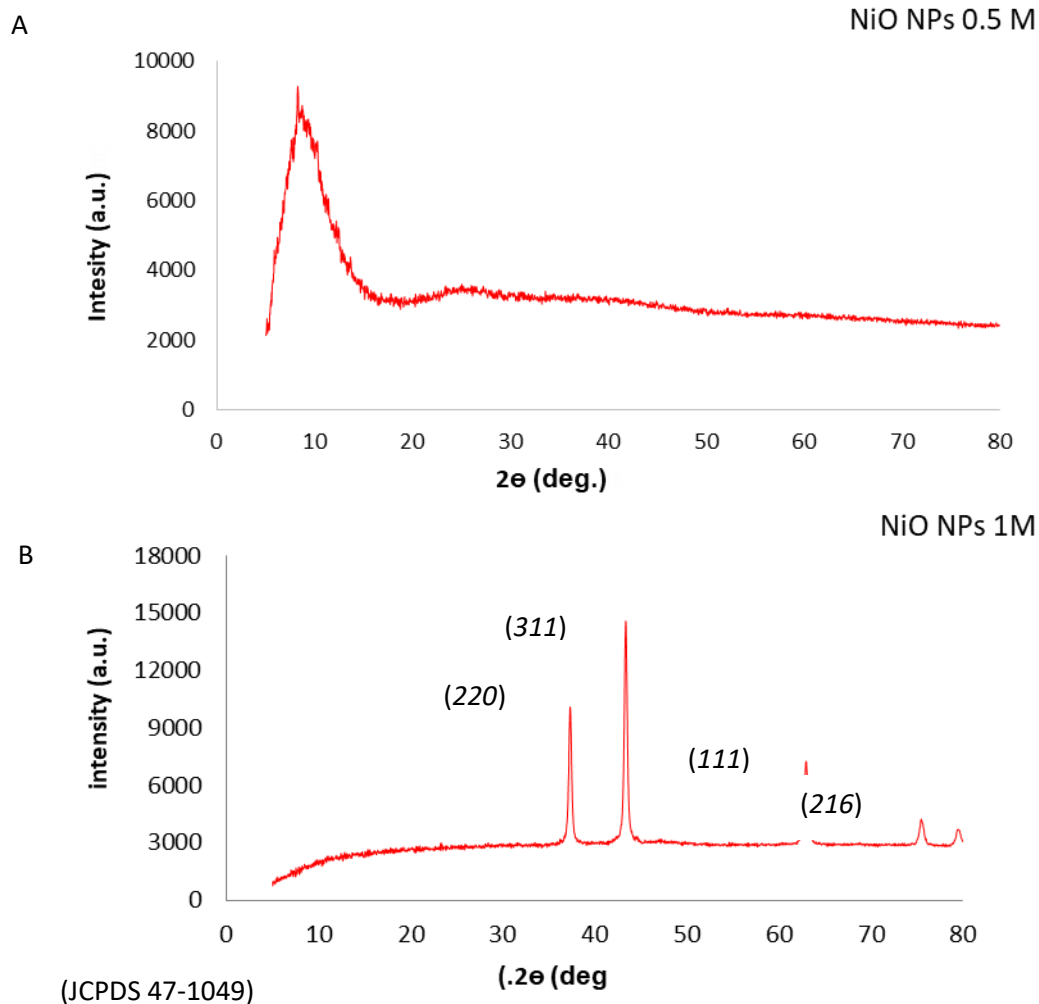


Figure 3: XRD pattern of NiO NPs from cellouise extract at 600 °C for 3 hours, A) NiO NPs 0.5 M, and B) NiO NPs 1 M.

Table 1: XRD results of NiO NPs from cellouise extract at 600 °C for 3 hours.

Plant Extract	Material	(hkl)	Crystallite size D(nm)
Cellouise	NiO	(220)	9
		(311)	10

3.2 FE-SEM measurements

An FE-SEM image for synthesizing non-violin NPs using cellulose extract manufactured on a glass substrate was used for analysis of morphology and average grain size of non-violins. Figure 4 (A-D) shows a micrograph with an average grain size of 40.19-70 nm and nano-ball morphology of the interesting nanoparticle structures [20-21].

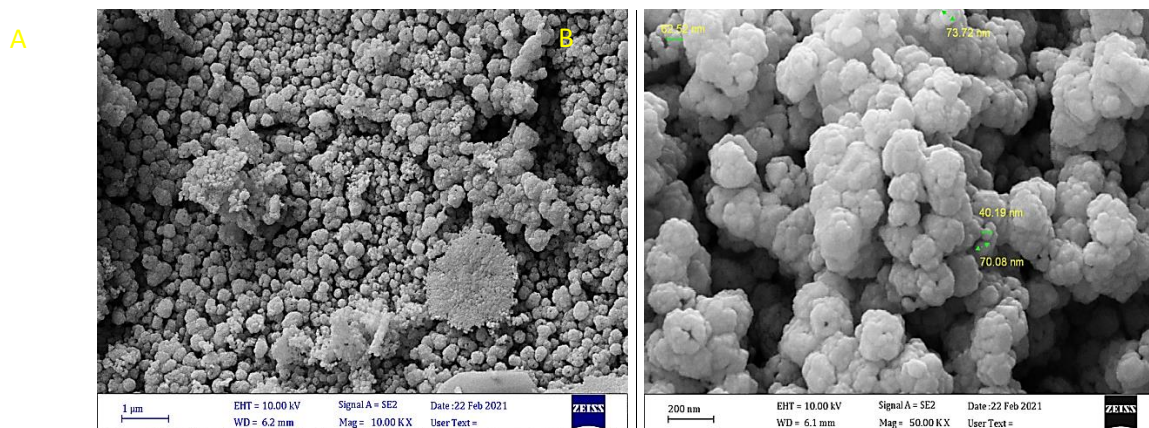


Figure 4 (A-D): FE-SEM analysis of NiO NPs from cellulose extract at 600 °C for 3 hours.

3.3 Photocatalytic activity of NiO NPs

Photocatalyst was calculated based on the degradation of the MB dye solutions by NiO NPs that prepared using ultrasonic method of cellulose extract. Figure 5 shows absorption spectra of MB solution that illuminated by UV light with wavelength of 338 nm for various time. The absorption peak is reduced with increasing illumination time indicating to degradation of MB dye. Figure 6 illustrates the degradation efficiency percentage for cellulose-based NiO NPs that synthesized at 10 gm/L under UV-light illumination for different time. From Fig. 6, the degradation ratio increased from 40% at illumination time of 15min to be 90% at 40min. The photocatalysts is due to the generation of numerous free holes in the valance band (VB) that lead to active oxygen species, such as OH^\cdot radicals and $\text{O}^{2-\cdot}$, being generated by electrons in the condition band (CB); these species are mainly responsible for dye degradation [22]. However, other nanometal oxide material such as ZnO nanorods showed high degradation ratio for MB dye under UV-light illumination [23].

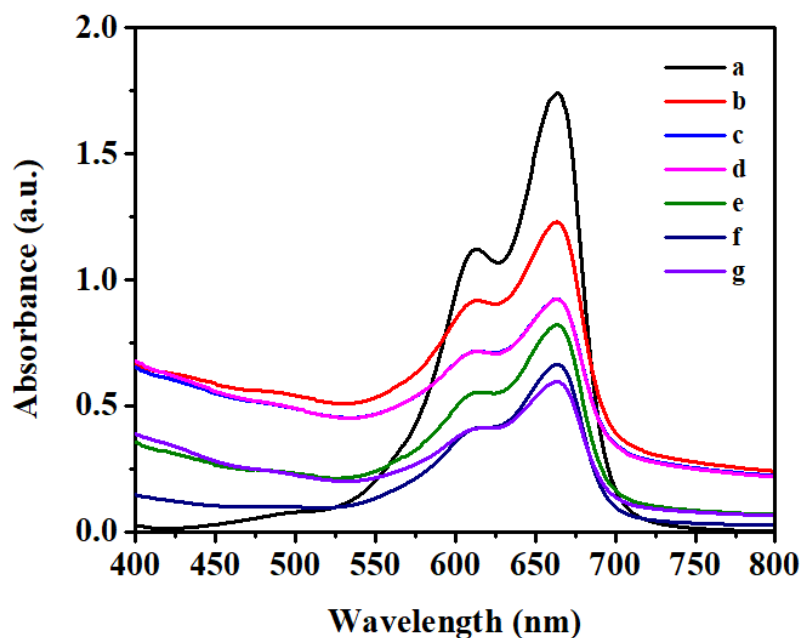


Figure 5: UV-Vis spectra of MB dye under the different time of irradiation of (a=0, b=15min, c=20min, d=25min, e=30min, f=35min, and g=40min).

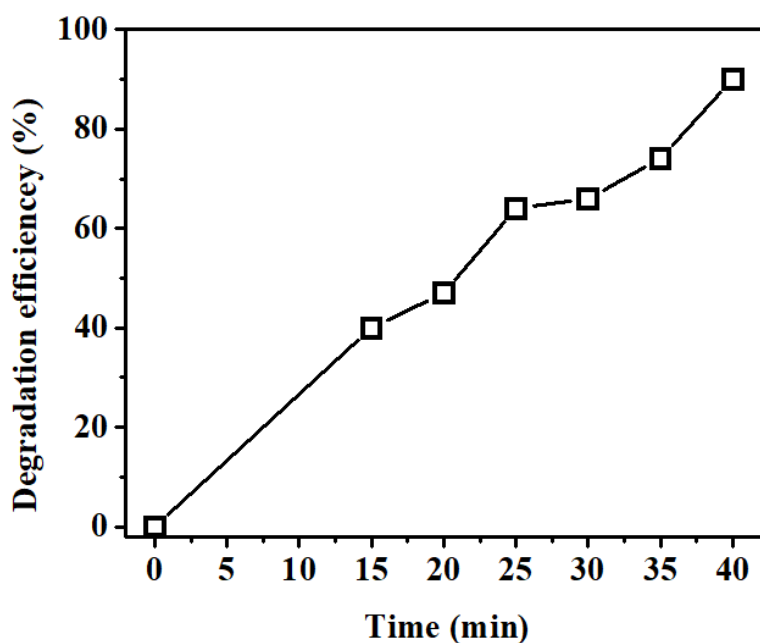


Figure 6: The degradation efficiency of MB dye by NiO nanoparticles under different time of UV light irradiation

4. Conclusions

NiO NPs were synthesis from cellulose extract with NiCH₃ salt by ultrasonic method 1 M at 600°C for 3 hours. In this research, NiO NPs at 1 M have greater structure properties than at 0.5 M. This method is new, simple, inexpensive, and environmentally friendly. The XRD results show the NiO NPs is clear and crystallite but that the diffraction peaks possess can be well indexed as the face center cubic (FCC) structure of NiO NPs at 1 M with a crystallite size of 9 nm. In addition, the FE-SEM result revealed the existence the NiO NPs prepared by the ultrasonic method at 600°C for 3 hours with a 40 nm grain size with nano-balls structure. Photocatalytic was studied in the degradation of the MB dye by use NiO NPs. The rate of MB dye degradation efficiency was determined to be 90% at a time 40min.



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دراسة الخصائص التركيبية للحبيبات النانوية اوكسيد النيكل المحضرة بطريقة المايكرويف للتطبيقات في المحفزات الضوئية

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المستخلص

تم تحضير جسيمات اوكسيد النيكل النانوية (NiO NPs) من مستخلص السليلوز مع 1 مولاري من ملح خلات النيكل (NiCH₃) النيكل بواسطة طريقة الترددات فوق الصوتية عند درجة 600 سيليزية. تم تشخيص جسيمات النيكل النانوية بواسطة حيود الاشعة السينية (XRD) ومجهر الإلكتروني الماسح (FE-SEM). أكدت نتائج XRD أن NiO NPs المحضرة هي بلورية بشكل مكعب متمركز الوجه (FCC) والحجم البلوري لها 9 نانومتر. كما اظهرت قياسات FE-SEM أن حجم جسيمات NiO NPs تتراوح من 40,19 نانومتر إلى 73 نانومتر مع تركيب كرات نانوية. تمت دراسة النشاط التحفيزي الضوئي لـ NiO NPs من خلال نسبة تحلل صبغة الميثيلين الزرقاء (MB) تحت زمن إضاءة مختلفة بواسطة الأشعة فوق البنفسجية حيث زادت نسبة تحلل صبغة MB من 40% إلى 90% عند زيادة وقت الإضاءة من 15 إلى 40 دقيقة.

