



Green synthesis of nickel oxide nanoparticles using *Helianthus annuus* seed husks extract and their application for removal of Pb (II), Cd (II), and Cu (II) ions from contaminated water

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Abstract:

In the current investigation, nickel oxide nanoparticles (NiO-NPs) were prepared from *Helianthus annuus* seed husk extract via the green route. The title nanoparticle was examined using SEM-EDX analysis, IR spectroscopy, powder X-ray diffraction, and UV-visible spectroscopy. They were employed in batch adsorption tests to remove study ions under multiple variables, including the initial ion concentration, pH, impact time, and amount of adsorbent. The results indicated that Freundlich isotherm and the second-order rate model were better suited to the experimental data. The R^2 coefficients acquired through the pseudo-second-order are 0.820, 0.998, and 1, herein in the Freundlich model are 0.916, 0.997, and 0.995 for each of the lead, cadmium, and copper ions, respectively. Meanwhile, under ideal circumstances, getting rid rates were found to be 99.94%, 99.73%, and 97.52% over the ions Cu, Cd, and Pb, respectively. As a result, these synthesized nanoparticles may be a suitable option for efficiently removing harmful metals from wastewater via adsorption.

1. Introduction

Water contamination has become a global issue hence since industry, technology and human civilization have advanced quickly[1]. It is thought

that contamination with heavy metals constitutes one of the primary elements influencing water bodies due to that they are hazardous, not biodegradable and persistent while discharged into the ecosystem by both artificial (vehicle exhausts, industrial emissions, and mining) and natural

sources[2]. Among the polluted metals emitted through various industrial waste streams are lead, cadmium, and copper. Lead (Pb) is one of the more well-known hazardous elements because of its significant environmental impact. Prolonged exposure to elevated levels of Pb (II) can cause a variety of severe harm to the reproductive system, basic biological functions, nervous system, and body systems. In addition, anemia, insomnia, liver disease, bone cancer, headaches, dizziness, irritability, and muscle weakness, hallucinations, renal damage, coma, and finally death are all brought on by lead poisoning[3]. Cadmium is released into the environment during the production of steel, cement, Ni-Cd batteries, phosphate fertilizers, and cadmium electroplating, among other processes[4]. The World Health Organization (WHO) [5] established guidelines in 2010 that defined 0.003 mg L^{-1} as the highest cadmium content permitted in drinking water. Bivalent cadmium has been linked to several human malformations, including testicular shrinkage, renal deterioration, proteinuria, skeletal deformity, and muscle cramping [6]. Copper comes from a variety of industries, including mining, smelting, electroplating, surface finishing, electrolysis, and electrical components[7]. This is yet another necessary component for the creation of tissues, enzymes, and the growth of bones. It is hazardous and carcinogenic when consumed in abundance, and it can cause nausea, vomiting, headaches, liver and kidney failure, breathing problems, and stomach pain[8, 9]. According to USEPA guidelines, the maximum amount of copper that can be discharged into industrial waste water is 1.3 mg L^{-1} . For this reason, Wastewaters that are hazardous to living things because they contain toxic metals must be treated before being released back into the environment. The toxic metals have been removed using various techniques, such as adsorption, electrochemical, reverse osmosis, ozonation, photocatalysis, oxidation, UV radiation, bioremediation, and ion exchange[10]. However, the adsorbent is the best due to a large specific surface, excellent sorption sites, a variety of surface contacts, quick adsorption rates, and inexpensive cost, which are all desirable qualities in this method[11, 12]. Researchers have been concentrating on nanotechnology in this field for a few years now, nanomaterials are among the more widely utilized adsorbents[13]. On the other hand, because of their unique chemical and physical properties and enormous surface area, semiconductor metal oxide nanoparticles have attracted a lot of interest from bulk materials[14]. Nickel oxide (NiO) nanoparticles have drawn the attention of scientists as a well-known

semiconductor because of their wide range of energy (3.6 to 4.0 eV), multifunctionality, and higher exaction binding energy than other metal oxides[15]. NiO nanoparticles have been synthesized using a variety of physical and chemical techniques, including microwaves[16], solvothermal[17], precipitation [18], anodic arc plasma [19], and sol-gel [20]. Still, these techniques have several drawbacks, including the need for hazardous chemicals, a hefty price tag, and a lengthy processing time. As a result, we used the green synthesis method in this work since it is straightforward, inexpensive, environmentally benign, non-toxic, has quick reaction times, is naturally biodegradable, and produces large yields[21]. NiO NPs have been produced successfully employing a variety of plant component extracts, including extracts from *Ananas comosus* leaves[22], *Allium Cepa* peels (onion peels) [15] and *Citrullus lanatus* peels (watermelon rind)[23]. These plant parts provide a variety of beneficial bioactive substances, such as minerals, vitamins, alkaloids, polyphenols, flavonoids, and terpenoids, which are essential for the synthesis of nanoparticles because they act as capping, stabilizing, and reducing agents [24]. In this investigation, we chose *Helianthus annuus* seed husks (sunflower) for preparing the extract to use in a green method for preparing NiO nanoparticles. *Helianthus annuus* is a huge herbaceous crop belonging to the Asteraceae family, is extensively grown across the globe because of its ability to adapt to various soil conditions, great tolerance to drought, and relatively short growth cycle[25]. The goal of the current investigation is to evaluate the capability for NiO nanoparticles synthesized from *Helianthus annuus* (sunflower) seed husks extract as cheap adsorbent for elimination of Pb(II), Cd(II), and Cu(II) ions from watery solutions. Different instrumental techniques like XRD, SEM, EDX, UV-Vis, and FTIR, were employed to characterize NiO nanoparticles. The evaluation of the different variables influencing the adsorption process included the time of contact, starting metal concentration, pH of the solution, and dosage of the adsorbent. Moreover, kinetic and equilibrium research on the metal ion adsorption onto NiO NPs.

2. Experimental

2.1 Material and chemicals

Nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), sodium hydroxide (NaOH), Cadmium sulfat-hydrat ($3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$), Copper nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$), and lead nitrate ($\text{Pb}(\text{NO}_3)_2$) were purchased from Merck chemicals,

doubledistilled water, White Sun flower seeds husks(*Helianthus annuus* seeds shells) were obtained from local market, Ouargla ,Algeria.

2.2 Physical Measurements

X-rays pattern of the synthesized nickel oxide nanoparticles was recorded using Proto diffractometer using the radiation of Cu Ka (1.5406 Å) in the diffraction angles (2θ) from 30° to 80°.The crystallite size was determined by expert High Score Plus Software. IR spectra were measured on Cary 8000 Agilent technology). Elemental analyses for nickel and oxygen were performed using a EVA 15 (Zeiss) elemental analyzer. UV-Vis spectra were collected using a Cary 100 Agilent technology UV-Vis spectrometer from 200-800 nm wavelengths at room temperature. For Metal ions(Pb^{2+} , Cd^{2+} and Cu^{2+})concentration in an aqueous solution was mesuredusing (Analytic Jena Contra A 800 Atomic Absorption Spectrometer, Germany).

2.3 Preparation of extract

The white sunflower seed husks were cleaned, dried at 60 °C, and ground into a powder. To make the extract, 9.2 g of SFSH powder was combined with 100 ml of DD water and stirred to 70 °C for 30 minutes. The acquired brown extract was finally completely filtered with whatman filter paper N° 2, which was kept for further use at 4 °C.

2.4 Green synthesis of nickel oxide nanoparticles

20 ml of the extract of sunflower seed husks was added to a solution of 2 g of nickel nitrate in 100 ml DDW. The blend was heated to 80 °C while being constantly swirled to create a uniform mixture. Sodium hydroxide solution (1M) was gradually added to modify the pH to 12. The reaction continued for 2h under the same conditions; during this reaction,the colour of the mixture transitioned via dark green to pistachio green, which indicated the formation of NiO NPs. The precipitate formed was filtered, repeatedly washed with DD water, then driedovernight at 100 °C in the oven. Ultimately, the green powder was calcined for two hours at 400 °C, **figure 1** depicts the schematic of the nanoparticle's green production.

2.5 Metal ions treatment experiments

In order to evaluate elimination of Lead, Cadmium and Copper ions from an aqueous solution, batch mode adsorption tests were conducted under various experimental conditions, including pH

value (2–8), adsorbent dose (10 mg–100 mg), contact time (10 min–60 min), and initial aqueous concentration (5–25 mgL⁻¹).The pH solutions levels were changed using 0.1 M NaOH or HCl. Every experiment was done by adding 25 mL of solutions with varying amounts of Cd^{2+} and Pb^{2+} and sharking at 200 rpm at ambient temperature 25 °C [26]. After applying NiO-NPs, an atomic absorption spectrophotometer (AAS) was used to quantify the amounts of metallic ions in the solution. **Equation (1) and (2)** were utilized to calculate the adsorption efficiency (%) and capacity (mgg⁻¹) of NiO NPs[27].

$$R = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{C_0 - C_e}{W} \times V \quad (2)$$

Where: (R %) is the percentage removal of the two metals, (C_0) and (C_e) are the concentrations of metal ions before and after removal (mg L⁻¹), respectively. W stands for the NiONPs weight in g and V stands for the solution volume in L.

The experimental data from the metal removal investigation was fitted with [28] and Freundlich isotherm [29] models to determine the adsorption capacity of nanoparticles. **Equation (3)** provides the empirical equation for the final isotherm model.

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (3)$$

K_f and n are the Freundlich constants representing the adsorbent's adsorption capacity (mg g⁻¹) and adsorption intensity, respectively. The Freundlich isotherm constants (n) and (K_f) are determined by calculating the slope and intercept of the plot ($\log q_e$) versus ($\log C_e$).

The degree of nonlinearity with solution concentration and adsorption is indicated by the n value, it has the following expression: adsorption is a chemical process if $n > 1$, a physical process if $n < 1$, and a linear process if $n = 1$ [30]. The concepts underlying the Langmuir equation [28] are that there are a limited number of binding sites evenly spaced over the adsorbent's surface and that the adsorbate sticks to the surface to create a single molecular layer. There is no more sorption when the monolayer is produced, and there is no interplay of the materials that have been adsorbed. The following **equation(4)** can be used to provide more explanation.

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad (4)$$

Where: C_e and q_e are the adsorbent's equilibrium concentration in (mg L⁻¹) and the amount adsorbed in (mg g⁻¹) at equilibrium, respectively. K_L is equilibrium constant for Langmuir adsorption,

which is correlated with the affinity of metal ions for nanosorbents ($L \text{ g}^{-1}$).

The essential part of the Langmuir model, which forecasts the affinity between the sorbent and sorbate, is the dimensionless separation factor R_L Equation (5).

$$R_L = \frac{1}{(1+K_L C_0)} \quad (5)$$

3. Results and discussion

3.1 X-ray diffractometer analysis

The X-ray diagram of green formation of NiO NPs is seen in **figure 2**. The diffraction peaks at 2θ values of 37.19° , 43.70° , 62.96° , 75.59° , and 79.54° relate to the miller indices (hkl) plane at (111), (200), (220), (311), and (222). The detected diffraction peaks, each getting a cubic structure, matched with JCPDS Card (No. 01-073-1519) **table(1)** which indicates the formation of NiO[31-33] The Strong diffraction peaks and the absence of indefinable peaks indicate the highest degree of crystallization and purity of synthesized NiO NPs. The size of the crystallite nickel oxide nanoparticles was calculated by applying Scherrer's formula **Equation(6)**[34].which was found to be 14.11 nm.

Where k is Scherrer's constant (typically from 0.9 to 1), λ is the wavelength, β signifies the full width at half maximum (FWHM), and θ is the Bragg's angle of diffraction, expressed in degrees[35].

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (6)$$

3.2 SEM and EDX analysis

The SEM picture of NiO nanoparticles annealed at 400°C is displayed in **figure (3a,b)**. It is visible in this image the irregular morphology of particles that agglomerated with each other, with different sizes ranging from 55 to 127 nm with an average value of 100 nm, shown in the histogram in **figure (3c)**. The agglomeration is possible account to the elevated surface tension and surface energy of NiO nanoparticles[36]. The stages of germination of the granules could be the reason for the non-homogeneous morphology. The EDX spectrum **figure(3d)** shows three clear peaks related to nickel and one of oxygen. This confirms these components' existence in the synthesized matter and the successful fabrication of NiO-NPs. The weight percentage was 56.44% of Ni and 43.56% of O. In addition, the atomic % of Ni and O were found to be 73.90 and 26.10, respectively. These results correspond to other research[37].

3.3 UV-Visible analysis

Figure (4) displays the gap band and UV-Vis spectra of the green synthesis to NiO-NPs. The output of this type of nanoparticles was validated by the appearance of a strong absorption peak (λ_{max}) at 298 nm, This outcome is in line with previous studies[38]. The strong band is caused by the electrical transition of the NiO semiconductor from the band of valence to the band of conduction [34]. The indirectly optical band gap energy (E_g) of NiO -NPs was ascertained using Tauc relation **Equation (7)**.

$$(ah\nu)^n = A(h\nu - E_g) \quad (7)$$

Which are: n is the indirect band gap transition, α is the absorption coefficient, E_g is the energy band gap and $h\nu$ is the photon energy. As a result, the band gap (E_g) was found to be 3.60 eV (figure 4); these results support that NiO is a semiconductor [39, 40].

3.4 IR analysis

The IR spectra of NiO-NPs that have been annealed at 400°C is shown in **figure (5)**. The functional group was identified in the region of $400\text{--}4000 \text{ cm}^{-1}$. The large absorption band that was seen at 3330 cm^{-1} is ascribed to O-H stretching vibrations from hydroxyl groups. Meanwhile, the second peak at 1626 cm^{-1} might be attributed to the bending of water molecules (H-O-H). The wide band at 1732 cm^{-1} corresponds to the carbonyl group (C=O), and the intense band observed at 1345 cm^{-1} fits the C-O extending vibration[41]. The other bands at 660 , 827 , and 981 cm^{-1} in the FTIR spectrum are related to Ni-O. The range of $1000\text{--}400 \text{ cm}^{-1}$ is known as the stretching frequency of metal-oxygen[22].

3.5 The effect of operating parameters on metal ion adsorption

3.5.a pH effect

pH is another significant factor affecting the adsorbent ability to adsorb heavy metal ions. The effect of pH to elimination efficiency of Cd^{2+} , Pb^{2+} and Cu^{2+} were investigated by adjusting pH from 2 to 8 without any other variables (the initial concentration of metal ion 20 mg L^{-1} , contact time 30 min at 200 rpm vibrating speed, with 20 mg of the adsorbent applied at room temperature). As shown in **figure(6a)** for all metal ions, the removal effectiveness by NiO nanoparticles was greater at basic pH compared to acidic pH; It was found that the elimination of pollutants including Pb(II), Cd(II), and Cu(II) was 97.52%, 99.73%, and 99.94 %, respectively, in alkaline conditions (pH = 8),

while acidic conditions ($\text{pH} = 2$) were found at 62.5%, 65.05%, and 99.90%.

Low adsorption of metals was seen at relatively pH values; this might occur related to the high concentration of hydrogen ions, which displace with M^{2+} at the active sorption/exchange locations and reduce the total of binding sites that are open to absorbing metals[42]. Meanwhile, the adsorbent area gets more negatively charged, and more OH groups are formed at a rise in pH, these metallic ions will be adsorbed more efficiently [43].

3.5.b Adsorbent dosage effect

The amount of adsorbent was ranged from 10 mg to 100 mg under certain conditions (contact period of 30 min, 200 rpm shaking speed, room temperature, pH of 7 and initial ions concentration 20 mg L^{-1}). Nickel oxide nanoparticles (NiO NPs) showed an ideal elimination capacity of 92.50%, 99.91% for Pb(II) and Cu ions, respectively. These findings indicate that the rise of the adsorbent dosage also raised the effectiveness of metal elimination at an ideal dosage of nanosorbent (20 mg) for Pb(II) and Cu(II) (40 mg) **figure(6b)**. Additionally, the elimination efficiency of Cd(II) rose when the dose was increased, reaching a maximum clearance efficiency of 99.70%. It could be the result of more adsorption sites being accessible for the metal ion adsorption on the NiO-NPs surface.

Meanwhile, Pb(II) and Cu(II) metal ions have been removed at a higher rate, which is most likely the result of more adsorption sites on the surface of NiO-NPs[44]. After reaching the ideal value, it did, however, decline as the adsorbent dose increased. This results from the overpopulation of adsorbent particles, which causes the overlapping of adsorption sites. The main factor reducing the contact surface of nanoparticles to eliminate metal ions was decreased adsorption, which happened when a large dose of NiO NPs was scattered into synthetic wastewater[27].

3.5.c Contact time effect

Contact time significantly impacts the rate of metal sorption because there are numerous active sites on the adsorbed surfaces. The effect of contact time using nickel oxide nanoparticles on the elimination effectiveness of studied ions were investigated for 10-60 min during (room temperature, 200 rpm shaking speed, a dosage adsorbent 20 mg, pH of 7 and initial ions concentration of 20 mg L^{-1}). The results appear in **figure(6C)**.

Pb(II) adsorption showed a maximum removal efficiency of 74.73% during the first 10 minutes before slowing down. The absorption rate decreases when M^{2+} moves from the outer layer into the micropores as the areas of activity are progressively

occupied[45]. The repelling reaction is probably the cause of this slightly declining trend after ten minutes[46]. For Cd(II) and Cu(II) were removed with an effective removal rate during 30 minutes of 93.97% and 99.96%, respectively. It could be explained by an increase in the interaction duration between metal ions and the active sites on the nano adsorbent. Additionally, removability efficiency grows slowly in the later stages after being rapid in the first. This is mostly because there are initially larger vacant places on the adsorbent surface, although they eventually become less over time [47, 48].

3.5.d Initial concentration effect

Under ideal conditions, the adsorption of previous heavy metals from the artificially polluted water was investigated, with varying starting doses of (5, 10, 15, 20, and 25 mg L^{-1}) applied to each metal ion. **Figure (6d)** summarizes the results. The metal ions' adsorption at different concentrations, as seen in **figure (6d)**, reveals that the clearance percentage falls as the metal ion concentration increases. Where reduced from 92.30%, 99.83%, and 99.88% to 77.45%, 99.64%, and 99.74%, accordingly, to the removal of Pb(II), Cd(II), and Cu(II) ions.

This phenomenon shows how both adsorption efficiency and metal concentration are significantly correlated. The quantity of metal ions on the nanosorbent increases at low metal concentrations because there are more absorbable empty sites available. Additionally, higher concentrations of metal ions result in fewer accessible adsorption sites, which lower the pace at which these ions removed[49].

3.6 Adsorption models

In this study, the explanation of the adsorption process was provided by the Freundlich and Langmuir isotherm models. The linear equations for each of them are provided in **equations (3) and (4)**. According to the Freundlich model, the nanosorbent surface is heterogeneous. However, the Langmuir model proposes that the nanosorbent surface is uniform and that adsorption stops once the available adsorption sites are occupied[50]. **Figure 7 and table 2** show the isotherm variables for Pb(II), Cd(II), and Cu(II) adsorption. As can be observed, the R^2 values of Freundlich model are greater than the the Langmuir model. This suggests that the nanosorbent surface is made up of many heterogeneous layers and assumes the formation of a multilayer of NiO NPs. Also confirm that the adsorption of study metal ions be explained by the Freundlich model. In accordance, the theoretical maximum adsorption

capacities ($q_{\max}$) with the Langmuir model were in the following order: Cu(943.39)>Cd(684.93)>Pb(16.19).

These metals were absorbed per 20 mg of NiO nanoparticles. In addition, important details about the kind of isotherm can be found with the (R_L) value, which can be either unfavorable ($R_L > 1$), unstable ($R_L = 0$), linear ($R_L = 1$), or favorable ($0 < R_L < 1$) [51].

The results showed that the R_L for Pb (II), Cd (II), and Cu(II) were 0.04, 0.51, and 0.562, respectively. Their values range from 0 to 1, which indicates that good adsorption. Additionally, the magnitude of the sorption driving force is indicated by the n value in the Freundlich equation. Since n ranges from 1 to 10, this suggests that lead (II), cadmium (II), and copper (II) have chemically nanosorption onto NiO NPs.

3.7 Adsorption Kinetic

A kinetic study of the adsorption of metal ions, along with additional variables, is required to determine the rate of adsorption. Additionally; it provides an explanation of how the adsorption process applies when batch parameters and adsorption techniques are used. [49]. In order to achieve that, we made a connection between the adsorption data of metallic ions and the generic solid-liquid adsorption system explanation models, including the pseudo-first, pseudo-second, and intraparticle diffusion kinetic models.

3.7.a First Order Rate model

According to this model, the amount of solid uptake over time and the difference in saturation concentration are directly related to the rate of change in solute uptake.

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (8)$$

In which k_1 is a constant adsorption rate (min^{-1}), q_e , q_t are the amounts of metal ions adsorbed per unit mass of the adsorbent (mg g^{-1}) at equilibrium and time, respectively.

3.7.b Second Order Rate model

The form of this kinetic model is as follows:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad (9)$$

Wherein: k_2 is the rate constant of pseudo-second order sorption ($\text{g mg}^{-1} \text{min}^{-1}$). If this model is suitable, the plotting, the plotting t/q_t versus time (t) would result in a line with a slope of $1/q_e$ and an intercept of $1/(k_2 q_e^2)$. The most appropriate kinetic model was determined to be the one with a better correlation value (R^2). Our findings would seem to show that, to get rid of ions (Pb, Cd, and Cu) on

fabricated nickel oxide nanoparticles, the employment of second-order kinetics yields a higher correlation value **figure (8a,b)** than first order. Additionally, by matching the results of experiments to **Equation (9)**, the second order rate equation's anticipated q_e value was found to be fairly similar to the actual q_e value, which we note that in the **table(3)**. This suggesting that the adsorption mechanism on NiO nanoparticles is pseudo-second order. These are consistent with previous results [43]. This implies that there is a relationship between the available active sites on nanosorbents and the adsorption capacity [50].

3.7.c Intraparticle diffusion model

This model is predicated on Weber and Morris' hypothesis [48] which explains the multistep adsorption process that involves the transfer of molecules into the interior of the solid pores after the movement of the adsorbate molecules from the solution phase to the adsorbent molecule surface. For this model, the linear equation is as **Equation(10)**.

$$q_t = K_{id} t^{0.5} + C \quad (10)$$

In this case, K_{id} ($\text{mg g}^{-1} \text{min}^{1/2}$) is the constant of intraparticle diffusion rate, can be determined from the slope of straight line **figure (8C)**, C is an intraparticle diffusion coefficient that may be found by finding the intercept of the plots between q_t and time (min), which is related to the boundary layer thickness (mg g^{-1}), $t^{0.5}$ is the time square root ($\text{min}^{1/2}$), and q_t is the time-dependent adsorption capacity. The values of C and K_{id} highlighted in **table(3)**. As shown the C values are larger due to effect of the boundary layer. Since every intra-particle diffusion rate constants calculated from this model in this work is non-zero, it is possible that this model is not the primary mechanism controlling the adsorption process [51].

3.8 Adsorption capacity compared via numerous adsorbents

Table (4) summarizes the results of a contrast highest capacity for adsorption of NiO nanoparticles with other adsorbents described in the literature to eliminate Pb(II), Cd(II), and Cu(II). The table is revealing evident that NiO NPs adsorption capacity is comparable with other nanomaterials. Taken together, these results suggest that synthesised nickel oxide nanoparticles it is effective for removing Pb(II), Cd(II), and Cu(II) ions. In general, anticipated to play the essential role in water purification applications.

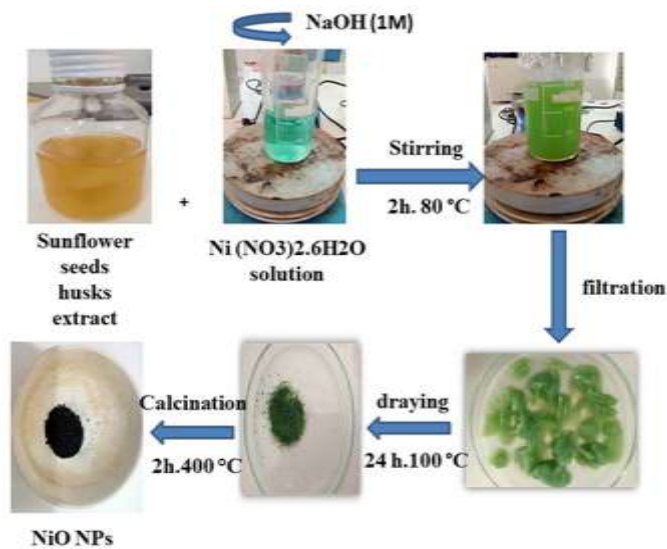


Figure 1.: Schematic of green synthesis of NiO nanoparticles

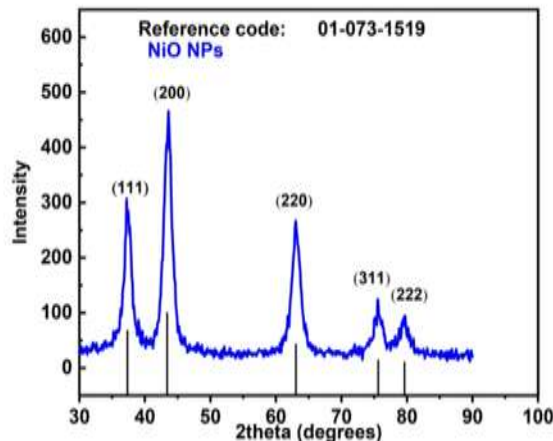


Figure 2: XRD pattern of biosynthesis of NiO- NPs at 400 °C.

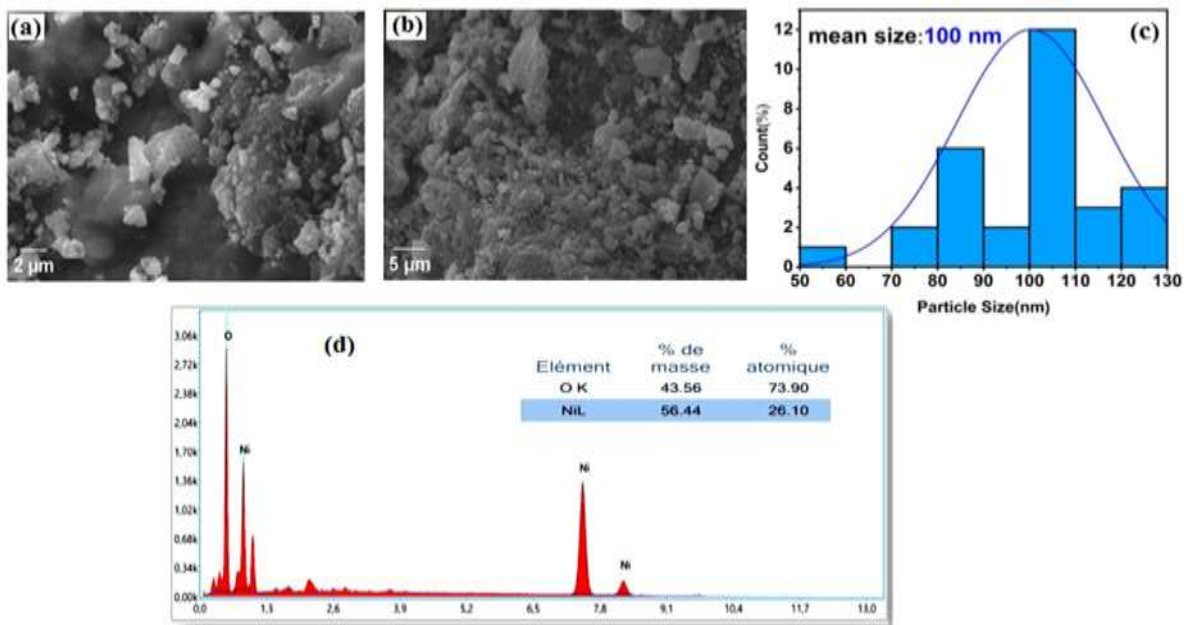


Figure 3 : (a,b) SEM pictures of NiO nanoparticles. (C) particle size distribution of the manufactured NiO- NPs. (d) EDX spectra of NiO nanoparticles

Table 1.: XRD peaks of nickel oxide nanoparticles at different diffraction angles

Miller Indices (hkl)	2 θ (degree)	FWHM	Particle size D(nm)
(111)	37.1993	0.48	17.46
(200)	43.7004	0.6	14.26
(220)	62.9625	0.36	25.87
(311)	75.5972	0.36	27.92
(222)	79.5419	0.96	10.76

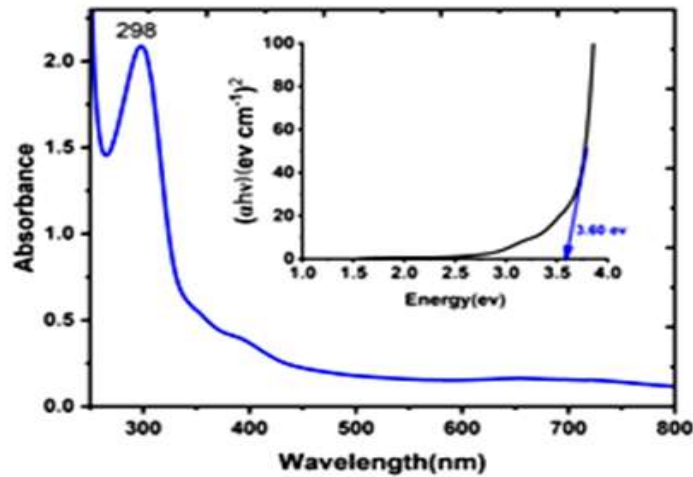


Figure 4:UV-Visible of green synthesis of NiO-NPs.

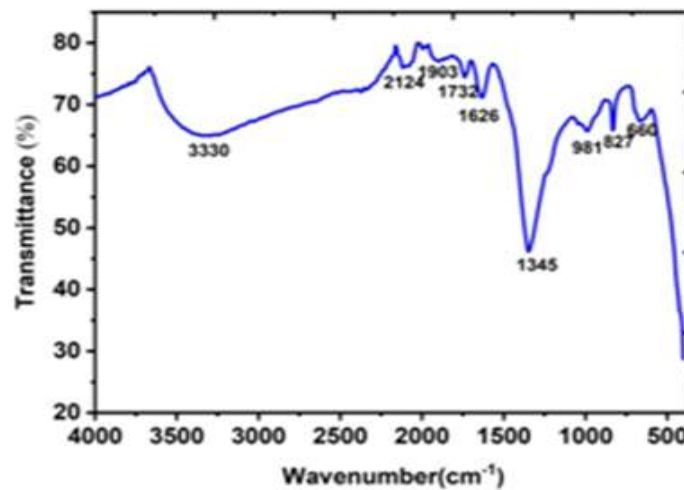


Figure 5: FTIR spectra of NiO nanoparticles annealed at 400 °C.

Table 2.Parameters of Isotherm models.

Metals	Langmuir				Freundlich		
	$q_{\max}(\text{mg/g})$	$k_L(\text{L/mg})$	R_L	R^2	$K_f(\text{mg/g})$	n	R^2
Pb	16.19	1.91	0.04	0.90	10.18	1.91	0.91
					161.75	1.45	0.99
					241.00	1.37	0.97
Cd	684.93	0.08	0.51	0.89			
Cu	943.39	0.07	0.56	0.58			

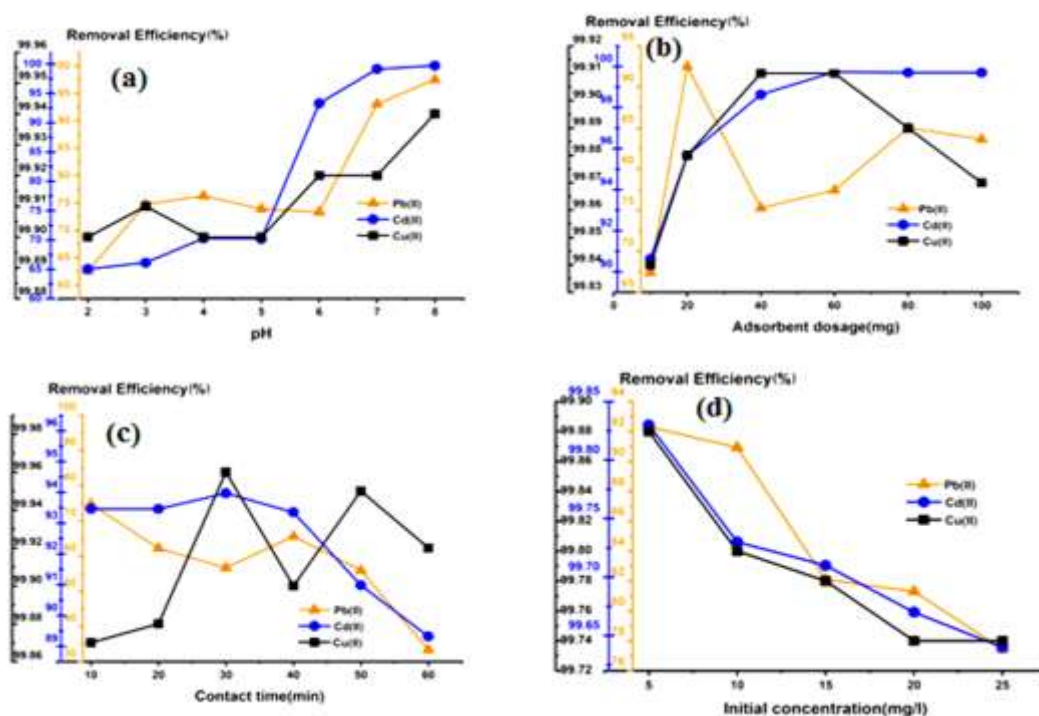


Figure 6: Selective adsorption of Pb(II) , Cd(II) and Cu(II) ionss into NiO-NPs, (a) effect of pH, (b) effect of adsorbent dosage, (c) effect of contact time, (d) effect of initial concentration.

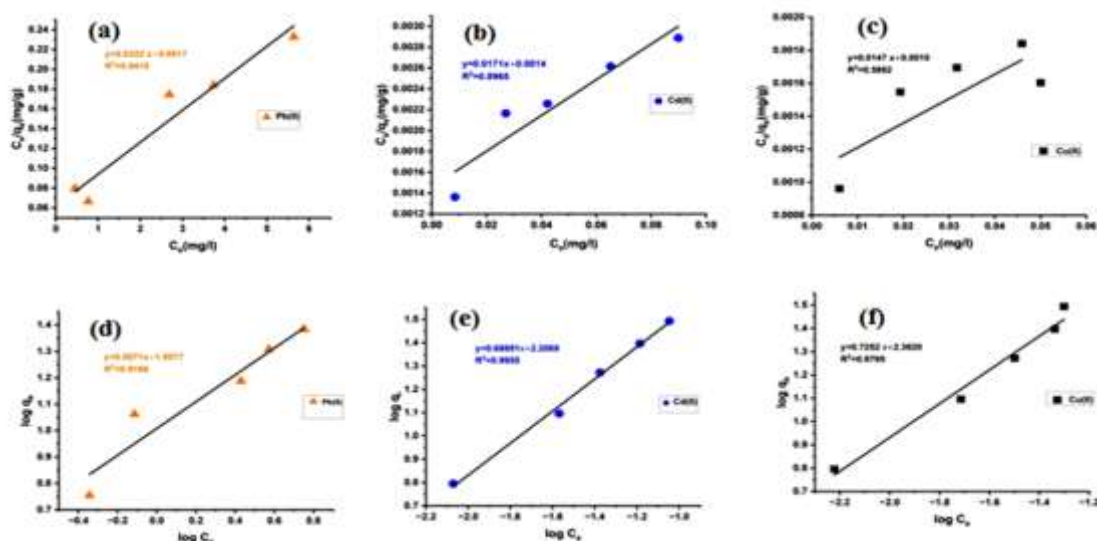


Figure 7:(a,b,c) Langmuir graphs .(d,e,f) Freundlich diagram for the adsorption of Pb(II), Cd(II), and Cu(II) through NiO NPs at interaction period: 30 min,amount dosage: 0.02 g and pH:7.

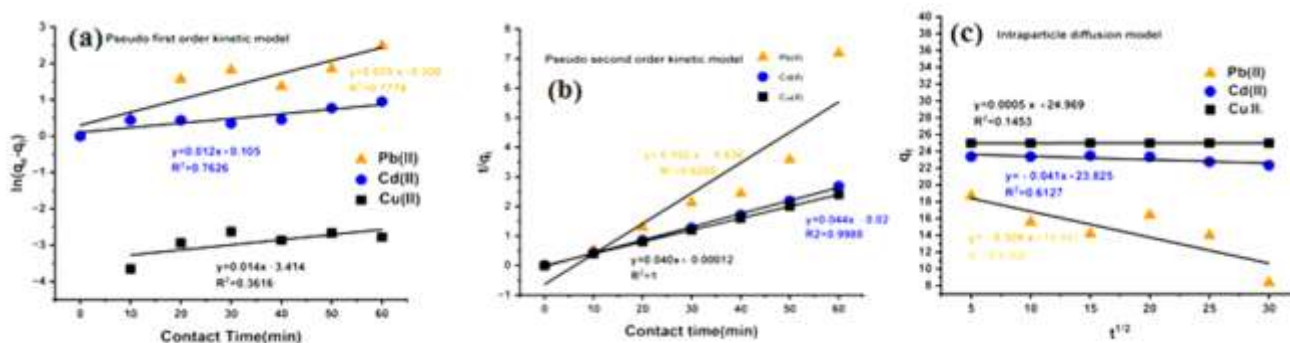


Figure 8:(a) Pseudo-first order, (b) Pseudo-second order and (C) Intraparticle diffusion models for the adsorption metal ions using NiO- NPs at initial concentration: 20 mg/ L, dose: 0.02 g, and pH:7.

Table 3.Parameters of adsorption kinetic models.

Metal ion	Pseudo1 st order				Pseudo2 nd order				Intraparticle Diffusion		
	Q _{exp} (mg/g)	K ₁	Q _{ecal} R ²		K ₂	Q _{ecal} R ²			K _{diff} C	R ²	
Pb	18.68	-0.03	51.35	0.77	-0.016	9.72	0.82		0.309	19.94	0.61
Cd	23.49	-0.01	21.11	0.76	-0.076	22.47	0.99		0.041	23.82	0.61
Cu	24.99	-0.01	40.032	0.36	-0.040	24.98	1.00		0.0005	24.96	0.14

Table 4. An overview of a few more known adsorbents applied to eliminate Cd²⁺, Pb²⁺ or Cu²⁺.

Adsorbent	q _{max} (mg/g)		References
	PbCd	Cu	
<u>CuO</u> -NPs	88.80	15.60	[1]
	-		
<u>ZnO</u> -NPs	7.66	2.02	[26]
	-		
<u>IO</u> -NPs	118.76	105.93	[52]
	-		
<u>TiO₂</u> -NPs	-	15.2	[53]
<u>TiO₂</u> -NPs	-	-	[54]
<u>Al₂O₃</u> -NPs	-	-	[55]
		47.2	[55]
<u>NiO</u> -NPs	148.6	135	This study
	149.1		
	41.2	118.9	
	47.9		
	16.19	684.93	
	943.39		

4. Conclusions

In this research, we have successfully synthesized nickel oxide nanoparticles from Helianthus annuus seed husk extract, which is considered agricultural waste. Its efficacy as an adsorbent for taking out Pb, Cd, and Cu ions from tainted water has been verified. NiO NPs' sorption capacity depended on the adsorbent dosage, starting metal contentions, pH, and time. The findings of this study indicate that ideal removal efficiencies for Pb(II), Cd(II), and Cu(II) were found to be 97.52%, 99.52%, and 99.54%, respectively. We think that our findings are good. In addition, the experimental results showed that the pseudo-second-order and the Freundlich isotherm model matched the adsorption process. According to how well these models fit together, the adsorption process was heterogeneous and chemisorption. Based on the Langmuir model, the maximal adsorption capacities for Pb(II), Cd, and Cu were 16.19, 684.93, and 943.39 mg/g, in that order. The evidence from this study suggests the possibility that synthesized nickel

oxide nanoparticles might help to remove harmful heavy metals from polluted water.

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