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Eco-Friendly of Nickel oxide Nanoparticles (NiO): A Comparative Study of Different Precursors

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ABSTRACT

The nickel oxide (NiO) nanoparticles were synthesized using an eco-friendly approach involving *Lettuce* leaves extracts as a reducing and stabilizing agent, with two different precursors: $NiSO_4$ and $NiCl_2$. Initially, Ni $(OH)_2$ nanoparticles were obtained by the dropwise addition of sodium hydroxide solution in the presence of *lettuce* leaf extracts. These Ni $(OH)_2$ nanoparticles were then subjected to calcination at 500 °C to produce NiO nanoparticles. The synthesized nanoparticles were characterized by XRD, FE-SEM, and TEM, and their surface area was calculated using the Brunauer–Emmett–Teller (BET) method. XRD analysis confirmed the formation of nickel oxide nanoparticles, with average particle sizes of 32.35 nm and 43.16 nm for the $NiSO_4$ and $NiCl_2$ precursors, respectively. FE-SEM images revealed that Ni $(OH)_2$ nanoparticles exhibited a spherical morphology, whereas NiO nanoparticles displayed varying shapes. BET analysis confirmed differences in surface area between the samples. This study aimed to reduce and adsorb heavy metals from water pollution using the Langmuir and the Freundlich isotherm models, which may be used to simulate experimental data on the adsorption of sulfur compounds from crude oil.

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1. INTRODUCTION

A physical or chemical substance that has at least one dimension between 1 and 100 nm is called a nanomaterial. It has attracted considerable interest for its unique optical, electrical, and mechanical properties that set it apart from bulk equivalents. [1] [2] [3]. Nowadays, environmental pollution from organic pollutants and their effluents has become a vital problem to water bodies and makes it unfit for drinking due to the extracts of textile and printing mills. In addition, industrial waste from petroleum refineries, coal conversion processes, pesticides, and drug production pollute the environment with toxic, hazardous phenolic compounds [4]. Green or sustainable chemistry refers to the creation of chemical substances that reduce or eliminate the use and production of harmful materials [5] [6]. Furthermore, a reaction is referred to be a "green reaction" when it consists of three elements: a solvent, a catalyst, and an energy consumer. Nickel oxide (NiO) is a p-type transition metal oxide with a face-centered cubic (NaCl-type) crystal structure and a wide band gap ranging from 3 to 4 eV, characteristics that make it highly attractive for diverse applications, including photocatalysis, gas sensing, energy storage[7], and antimicrobial technologies. Its notable chemical stability, optoelectronic behavior, and biological activity have positioned NiO as a key material in the current research landscape on multifunctional nanomaterials [8]. Recently, various techniques have been used to synthesize NiO nanoparticles, including solvo-thermal process, solid-state chemical decomposition, reverse micro-emulsion method, chemical precipitation, thermal decomposition, and sol–gel combustion [9] [10]. However, these methods involve specialized equipment, high temperatures, complex procedures, and are even toxic.

Hence, there is great concern about developing eco-friendly methods that use biological systems such as fungi, bacteria, plants, and yeast, as well as naturally occurring compounds like vitamins and proteins. The extracts are widely used in the preparation of nanomaterials because they act as both reducing and capping agents, making them preferable to chemical and photochemical reduction methods. The objective of this work is to manufacture NiO nanoparticles utilizing two salts ($NiSO_4$ and $NiCl_2$) and extracts from *lettuce* leaves as a stabilizing agent and reduction factor. The generated nanoparticles' size, shape, and surface area will then be compared.[11]

2. Experimental Methodology

2.1. Materials:

Analytically pure chemicals (CDH) The NiO nanoparticles were created using deionized water, *lettuce* leaves, nickel (II) chloride (NiCl2) (98%), and nickel (II) sulfate (NiSO4) (99%).

2.2. Instruments and Apparatus:

Every sample analysis was carried out at the University of Tehran, Iran's College of Science. The Siemens model D500 is used to irradiate the samples to obtain the XRD pattern using the XPERT-Pro diffractometer, which operates at 30 mA and 40 kV, with scanning angles ranging from 20 to 80°. The Scanning Electron Microscope (FE-SEM) ZEISS Sigma VP and the Transmission-Scanning Electron Microscope (TEM) are utilized for surface investigations. TriStar 3000 from Micromeritics Inc. was used to determine the pore volume and specific surface area (SSABET).

2.3. Synthesis of NiO nanoparticles using *Lettuce* leaves extract:

Lettuce leaves from trees in the Diyala governorate were used to make the plant extract. They were thoroughly cleaned with ordinary water and then with distilled water. After two days of drying in the shade, the leaves were chopped and crushed finely. Ten grams were then added to 150 milliliters of distilled water, and the mixture was heated to 80°C for 30 minutes while stirred. After cooling and filtering, the extract is transferred to test tubes and centrifuged for ten minutes at 3000 rpm to remove any leftover live elements. It is then collected in a vial and stored at 25 °C. 50 milliliters of distilled water were mixed with 0.3 grams of salt and well swirled with a magnetic stirrer. Ten milliliters of the *Lettuce* leaves extracts are placed in a burette and added to the mixture, drop by drop, while it is at 25°C. We progressively added sodium hydroxide solution NaOH (0.1M) after raising the temperature to 80 °C until the solution turned basic and a precipitate developed. Pollutants and silt are washed away using ethanol and water. The precipitate is then dried for 3 hours at 70 °C to generate nano nickel hydroxide powder, and after 5 hours of burning at 500 °C to form nickel oxide nanoparticles, the powder is formed[12]. Figure (1) illustrates the steps for carrying out the experiment.

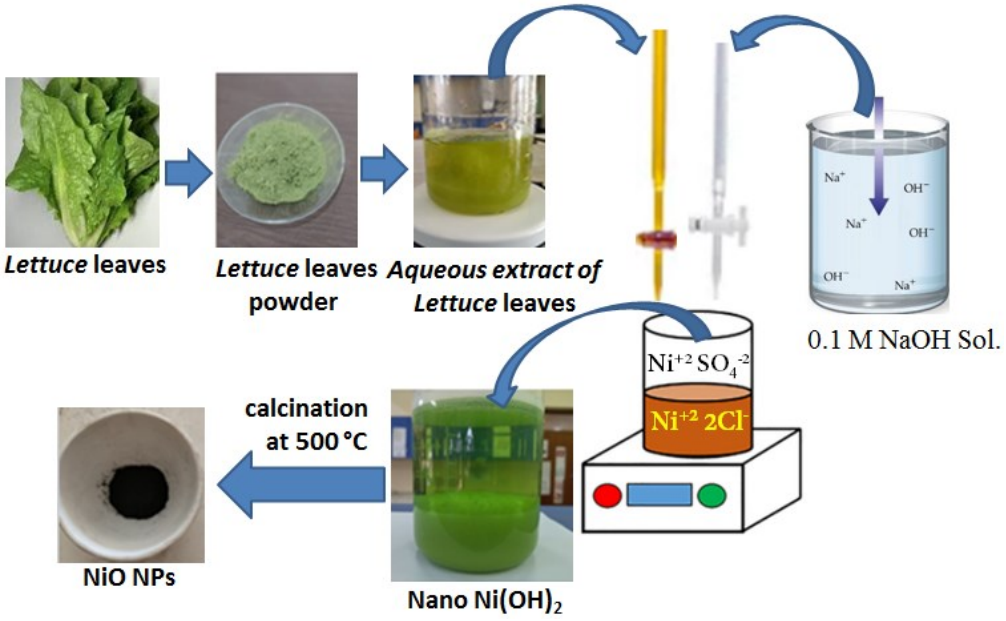

Figure 1. Synthesis of NiO NPs using *lettuce* leaf extract with $NiCl_2$ and $NiSO_4$ as sources.

3. RESULTS AND DISCUSSION

3.1. XRD analysis: The nickel oxide nanoparticles' XRD pattern was shown in figure 2.The only phase that was dominant was NiO NPs. According to the Debye-Sherrer equation, the average size of the NiO nanoparticles was around 32.35 nm for the $NiSO_4$ precursor and 43.16 nm for the $NiCl_2$ precursor. That is shown in Table1, and Figure 2.

Table 1. The indexing XRD of two types NiO nanoparticles.

NO.	2 Theta (degree)	FWHM	hkl	D, nm	D, nm Average
NiO NPs from $NiSO_4$	30.84	0.246	002	30.17	32.35
	36.18	0.3444	111	31.12	
	43.51	0.3444	200	35.15	
	54.17	0.3936	202	31.05	
	63.03	0.2952	220	30.13	
	75.13	0.2952	311	37.17	
NiO NPs from $NiCl_2$	31.71	0.246	002	35.17	43.16
	38.93	0.3444	111	45.56	
	43.93	0.3444	200	47.86	
	44.46	0.3936	111	41.95	
	55.93	0.2952	202	40.89	
	63.68	0.2952	200	48.52	

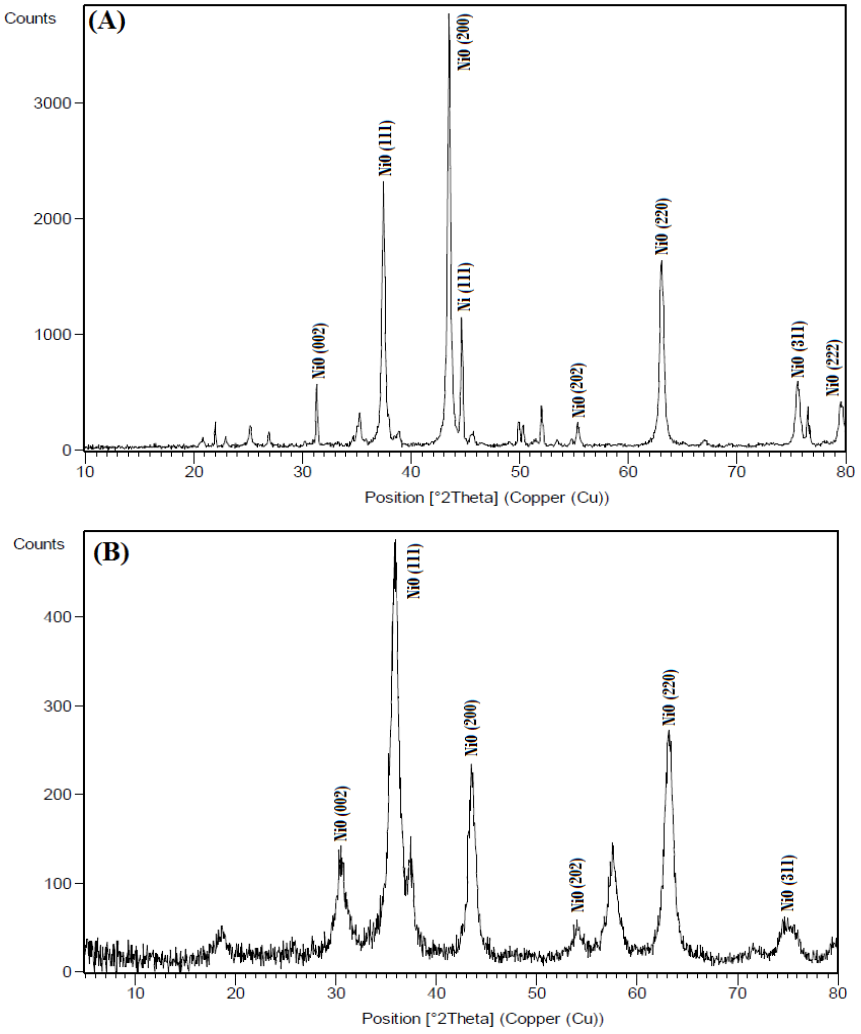

Figure 2. XRD pattern of NiO NPs (A) $NiCl_2$ as sources, and (B) $NiSO_4$ as sources

3.2. FE-SEM image:

FE-SEM image: Figures (3 A-D) display the $Ni(OH)_2$ and NiO nanoparticles' FE-SEM images, which exhibit sheets surface cubic and oval nanoparticles [13], with average diameters of 45.43 and 109.46 nm, respectively, the spherical nanoparticles are part of the $Ni(OH)_2$ and NiO nanoparticles from the sulfate precursor. On the other hand, the average diameters of $Ni(OH)_2$ and NiO nanoparticles synthesis from chloride precursors in oval form are 63.55 and 266.34 nm, respectively. According to our findings, the nickel oxide nanoparticles are bigger than the hydroxide, and we think this is because the calcination procedure was carried out at a high temperature.

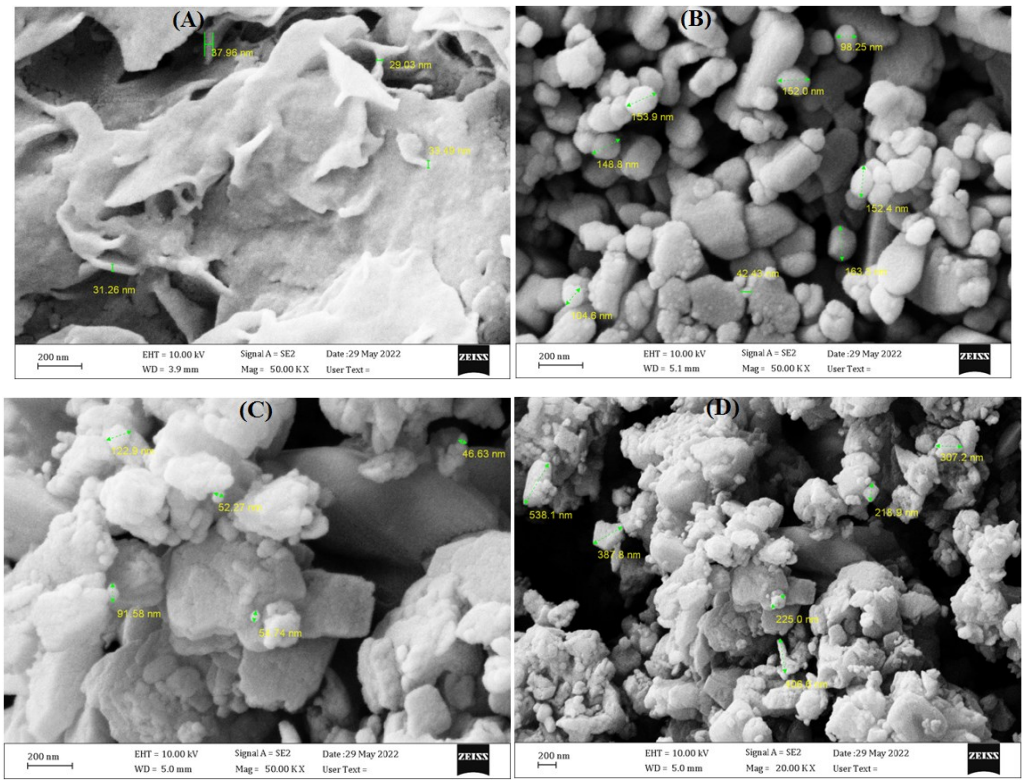

Figure 3. FE-SEM images of (A) $Ni(OH)_2$ NPs from $NiSO_4$,(B) $Ni(OH)_2$ NPs from $NiCl_2$, (C) NiO NPs from $NiSO_4$ and (D) NiO NPs from $NiCl_2$ precursor.

3.3. TEM images:

A TEM picture of nickel oxide nanoparticles (NiO NPs) from sulfate precursor at 100 nm magnification is shown in figure 4a. These particles are strongly clustered and have polyhedral with measured frequency, ranging from 47 to 69 nm. Nickel oxide nanoparticles (NiO NPs), which vary in size from 43 to 100 nm and have spherical and cubic morphologies, are seen in the TEM images in Figure 4 b which chloride as a precursor.

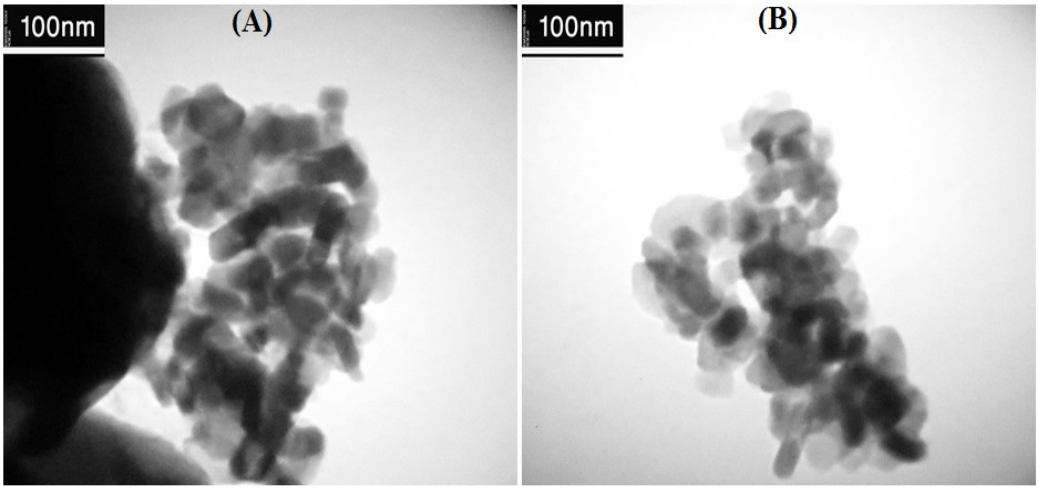

Figure 4. TEM images of (A) NiO NPs from $NiSO_4$,(B) NiO NPs from $NiCl_2$.

3.4. BET Analysis:

The distribution of nanopores and surface area can be better understood by using BET and BJH experiments. Figure 5(A-D), which was created, shows the BJH nanopore size distribution scheme as well as the adsorption and desorption isotherms of NiO NPs. The existence of medium pores (diameter 2-80 nm) [14] was shown by the BJH diagram, and the surface area of NiO NPs, which were formed from sulfate precursor, was 49.83 m^2/g. Nonetheless, the pore size distribution was irregular, with most nanopores ranging from 20 to 40 nm (Figure 5A).

Even though the surface area of NiO NPs generated from chloride precursor was less at 24.11 m^2/g (Figure 5C), as illustrated in Figure 5B, the BJH diagram demonstrated the existence of medium holes (diameter 2-80 nm). The bulk of the nanopores were between 10 and 30 nm in size, indicating that pore size was not uniform. As seen in figure 5D, the existence of medium pores (width 1-100 nm) was shown by the BJH diagram.[15]

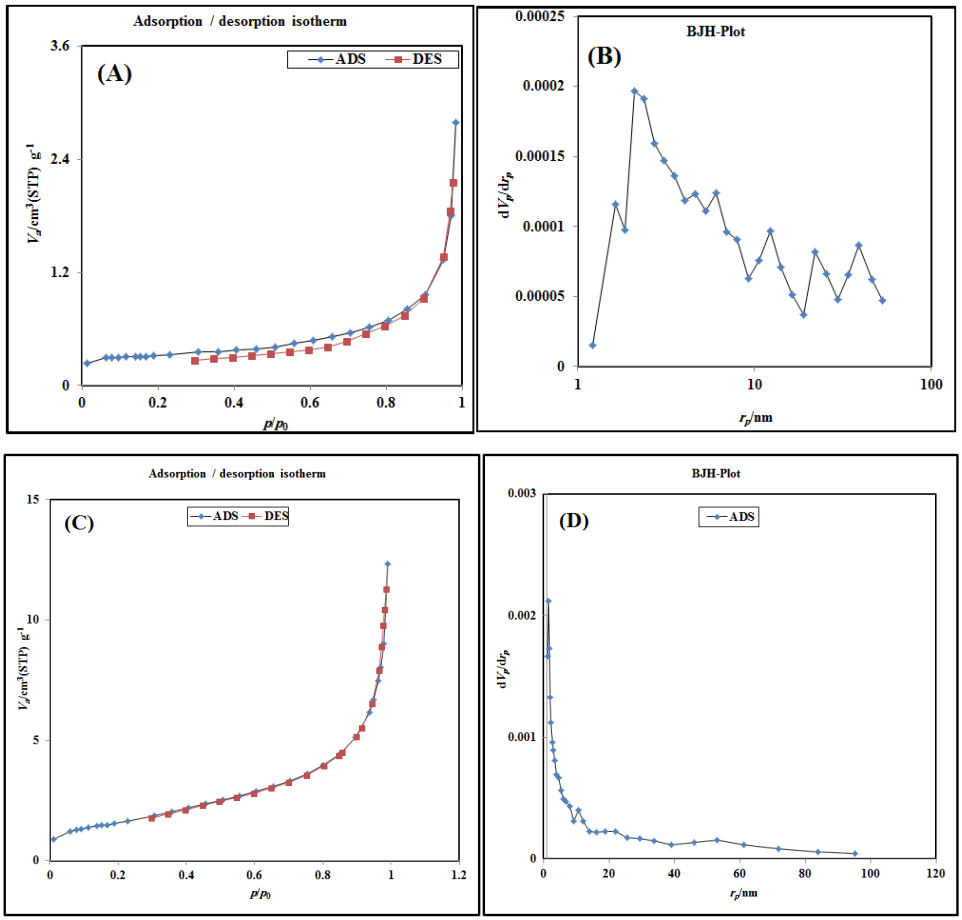

Figure 5. BET and BJH plot of (A) and (B) NiO NPs from $NiSO_4$ precursor, (C) and (D) NiO NPs from $NiCl_2$ precursor.

4. Adsorption of pollutants on NiO nanoparticles

The Langmuir and Freundlich isothermal models may be used to simulate experimental data on the adsorption of sulfur compounds from crude oil. Equation (1) provides the Langmuir isotherm [16]:

$$\frac{C_t}{q_t} = \frac{1}{bqmax} + \frac{C_t}{qmax} \qquad (1)$$

Qt (mg/g) is the quantity of solute adsorbed. qmax (mg/g) is the maximum monolayer on the adsorbent. The equilibrium concentration is Ct (mg/L), and the Langmuir isotherm constant is b(L/mg). The specific adsorption (Ct/qt) vs. equilibrium concentration (Ct) is linear. qt(mg/g) is the quantity of solute adsorbed. qmax (mg/g) is the maximum monolayer on the adsorbent.

The Freundlich equation, Equation (2)

$$\ln q = \ln kF + \left(\frac{1}{n}\right) \ln Ct \qquad (2)$$

Where k_F is adsorption capacity (L/mg) and 1/n is adsorption intensity; it also indicates the relative distribution of the energy and the heterogeneity of the adsorbate sites.

As demonstrated by the relationship between (Ct) versus (Ct/qt) in Figure 6A and C, the adsorption curves of sulfur compounds in their crude oil were measured using the Langmuir equation. According to the results, the Langmuir adsorption equation, which is well-suited to surface monolayer sorption with a few matching sites and consistent adsorption energies, may be used for them. Although the Freundlich equation was developed for adsorption on heterogeneous surfaces, it does not account for monolayer adsorption.

As demonstrated by the relationship between (ln qt) vs (ln Ct), the adsorption curves of sulfur compounds (figure 6 B and D) in crode oil dilute were adjusted to the Freundlich equation, and the findings indicate that they do not undergo adsorption on this Equation (2).

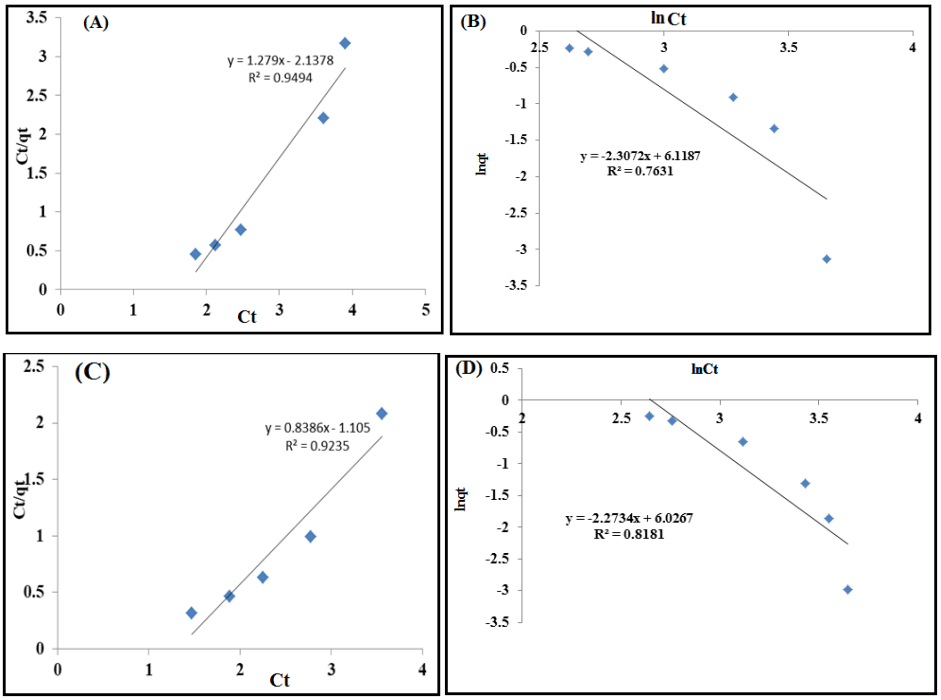

Figure 6. Adsorption equation of (A) Langmuir isotherm by NiO NPs from $NiSO_4$ as precursor, (B) the Freundlich isotherm by NiO NPs from NiSO4 as precursor, (C) the Langmuir isotherm by NiO NPs from NiCl2 as precursor, and (D) the Freundlich isotherm by NiO NPs from $NiCl_2$ as precursor at 298 K.

5. Conclusion

The properties of nanoparticles generated using diverse plant extracts varied with the type of phytochemicals obtained. The pH, nickel ion concentration, solution stirring speed, and plant extract concentration were also used to determine the size and other distinguishing features of the resulting nanomaterial. The size and shape of the particles can be seen using tools such as a scanning electron microscope (SEM) or a transmission electron microscope (TEM). By analyzing the graphical representation of the NiO, the average size of the NiO nanoparticles for the NiSO4 and NiCl2 precursors, as determined by XRD, was 32.35 and 43.16 nm, respectively. With average sizes of 45.43 and 109.46 nm, respectively, the spherical nanoparticles constitute a component of the Ni $(OH)_2$ and NiO nanoparticles from the sulfate precursor. A variety of particle sizes, with cubic and oval shapes and different average diameters, is revealed by FE-SEM analysis. However, Ni(OH)2 and NiO nanoparticles prepared from chloride precursors had typical diameters of 63.55 and 266.34 nm, respectively. The TEM images displayed are highly crowded and range in size and shape from 47 to 69 nm. The spherical and cubic morphologies of nickel oxide nanoparticles (NiO NPs) range in size from 43 to 100 nm. The surface area of NiO nanoparticles is 24.11 m^2/g for the chloride precursor and 49.83 m^2/g for the sulfate precursor, as determined by the BET method. Based on the correlation coefficient values, the Langmuir isotherm was ultimately determined to be the most effective for describing sulfur adsorption.

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