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Walnut Shells as Sustainable Low-Cost Adsorbents for the Removal of Cadmium and Nickel from Aqueous Solutions

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ABSTRACT

The rapid development of industries has led to increased release of heavy metals into the environment. When released into the environment, metals have a tendency to linger eternally, build up in living tissues across the food chain, and endanger both the ecosystem and public health. Usually present in trace amounts in industrial wastewater, heavy metals are difficult to remove from water. In the current study, an adsorbent composed of walnut shells waste powder was manufactured and tested for the adsorption of heavy metals cadmium (Cd) and nickel (Ni) from synthetic aqueous solution. Several experimental conditions affecting the removal process were controlled, including pH, contact time, adsorbent dosage, temperature of solution, and initial concentration of Cd and Ni, to achieve optimal removal conditions. The adsorption technique was effectively implemented, attaining, under ideal circumstances, a removal efficiency of 95% for Cd and 99% for Ni. Additionally, the adsorption capacity (qe) of 14.25 mg/L for Cd and 14.85 mg/g for Ni. The optimum conditions for removing heavy metals (Cd and Ni) were: The initial metal concentrations are 30 mg/L, the bioadsorbent dosage is 2 g/L, the adsorbent particle size is between 0.3 and 0.075 mm, the agitation speed is 150 rpm, the contact period is 60 minutes, the pH is 5, and the solution temperature is 45°C. In summary, this study's suggested walnut shell material offers an economical, ecologically responsible, and alternative adsorbent for removing heavy metals from aqueous solutions by adsorption.

1. Introduction

Clean water is among the planet's most vital and valuable natural resources. It is essential to our survival and necessary for many households, agricultural, and industrial processes[1]. Rapid population expansion and industrialization have caused a sharp rise in water demand in recent decades, which has resulted in water contamination and the depletion of natural water supplies[2], [3]. During recent decades, the drainage of industrial wastewater loaded with organic pollutants has led to serious pollution in the surface water and groun

dwater[4]. Therefore, one of the biggest dangers to the existence of all living things is water pollution, including humans. Water pollution can affect the growth of living organisms, interfere with the food chain, or introduce toxic substances, harming human health and hindering its use in daily life[5], [6]. Most industries release industrial wastewater into nearby water bodies, especially rivers, without treating it, resulting in serious and undesirable impacts on the environment and ecosystems[7]. Natural water resources are severely strained by water-intensive industries, including pharmaceutical products[8], textiles[9],

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leather tanning[10], and refining of crude oil [11].

The high levels of heavy metals in industrial effluent are one of these issues. Because they are harmful at very low quantities and non-biodegradable, heavy metals are hazardous compounds that can harm the ecosystem. Metal poisoning causes many human diseases, such as cancer, neurological disorders, organ failure, stunted growth, abdominal pain, and other ailments[12]. Metal concentrations have been found in the environment as a result of the discharge of industrial wastewater from many industries, such as tanneries, textiles, electroplating, fertilizers, battery manufacturing processes, mining, and oil refineries, which cause metal toxicity[13]. Numerous wastewater types contain harmful metals that are ionic, including Pb (II), Fe (II), Cd (II), Cu (II), Ni (II), Zinc (II), Zn (II), Chromium (VI), Arsenic (II), As (III), Mercury (III), and Hg (II)[14]. They are released into the atmosphere in amounts that are harmful to people's health. The kind and concentration of metals, pH, wastewater type, and the solubility of the metal ion are some of the variables that affect metal toxicity[15]. Because metal ions cannot be biodegraded into less hazardous forms, heavy metal contamination is a worldwide issue. Compounds linger in the environment. Furthermore, because they impact living things' life through bioconcentration, in the food chain, bioaccumulation, and biomagnification, these ions are toxic and dangerous to living things. Because heavy metals can bioaccumulate, their presence in the environment has grown to be a serious concern. and their toxicity [14], [16]. Therefore, before releasing these metals into the environment, industrial wastewater must be cleaned of them.

There are several techniques for removing heavy metals from aquatic environments, including chemical precipitation, electrochemical treatment, chemical filtration, solvent extraction, membrane separation, and adsorption[17], [18]. Usually, these approaches are constrained by significant upfront expenses and/or their incapacity to satisfy strict effluent regulations[19]. Adsorption process is the stared as a talented technology among these methods due to its great removal efficiency, affordability, ease of use, simplicity, lack of sludge, and recyclability[20], [21]. One of the most effective approaches for removing contaminants from industrial wastewater is adsorption. Additionally, adsorption offers a compelling substitute therapy, particularly when the adsorbent is affordable and easily accessible[22]. This treatment technique extracts pollutant molecules (also referred to as adsorbates) from contaminated aqueous solutions using a Material that is either natural or artificial (also referred to as an adsorbent, biosorbent, or sorbent)[23]. Agricultural wastes are used as adsorbents in adsorption processes, particularly those derived from plant biomass (such as peels, roots, stems, fibers, etc.), which can typically be discarded after harvest. They are widely used as raw materials for adsorbent production[24], [25]. Research in recent years has focused on their use as adsorbents due to their low cost, large availability, and effectiveness in removing many organic and inorganic materials in adsorption systems[26], [27].

Walnut Shells are considered a high-value agricultural waste, as they contain high levels of cellulose, lignin, and hemisloseoseosene, which gives them good porphyle and effective sites for absorption. Studies have shown their excellent ability to remove many pollutants, such as organic dyes and heavy minerals, from water solutions. These walnut shells are characterized by low cost and availability locally, as well as being environmentally friendly by recycling agricultural waste rather than disposing of it[28], [29]. However, few studies have addressed the use of walnut shells for the removal of heavy

metals, representing a gap that this study aims to fill.

For the adsorption treatment of sewage or industrial wastewater, it is therefore essential to use walnut shell waste as a potential adsorbent. Wasted walnut shells (WS), an inexpensive, efficient, and environmentally friendly adsorbent, were used in the current work to examine the removal efficiency of cadmium (Cd) and nickel (Ni) from industrial wastewater through adsorption. Along with assessing the degree to which different factors impact the adsorption process, these factors include the impact of the solution's acidity function (impact of pH), the bioadsorbent dosage, the starting concentration of the nickel and cadmium contaminants, and the impact of temperature, particle size, and contact duration.

2. Materials and methods

2.1Chemicals

To create simulated heavy metal-contaminated wastewater, tap water was used to dissolve cadmium nitrate ($Cd(NO_3)_2.4H_2O$) and nickel chloride ($Ni(Cl)_2.6H_2O$) (Germany's Tetenal Photowerk GmbH), both of which had purities of more than 98%. The pH of the solution was adjusted using HCl and NaOH, which were acquired from Sigma-Aldrich, a chemical firm in Milan, Italy.

2.2 Equipment:

The following were used:

- To regulate the pH of the aqueous solution at room temperature throughout the tests, a pH meter (WTW, inoLab 720, Germany) was utilized as needed.
- A sensitive electric balance was used to weigh the materials used in the experimental laboratory work (Model: Sartorius ENTRIS124-1S Lab Balance, Germany).
- A thermometer was used to measure the temperature of the solution.
- Before and following treatment, the concentrations of heavy metals in the aqueous solution were determined by use of atomic absorption spectroscopy (Shimadzu AA-6200-Japan).
- Samples were stirred in a water bath shaker, varying from 50 to 300 rpm (During the separation phase, Grant Instruments Ltd., Serial No. Q72046001, Model OLS26, UK), at 150 rpm to lower surface mass transfer resistance.
- Glassware: Some glassware was used in these experiments, such as a volumetric flask, a pipette, and others.

2.3 Adsorbent preparation

Waste from discarded walnut shells was gathered from Baghdad's local marketplaces. After thoroughly cleaning the materials with distilled water, they were allowed to air dry for a full day. After that, they were cooked for 24 hours at 60°C. Particle sizes ranging from 0.075 to 0.3 mm were then separated by grinding and screening the dry samples. After being repeatedly cleaned with distilled water, they were dried for 48 hours at 48°C. The bioadsorbent was kept in airtight containers for use in the adsorption of water-based heavy metal solutions after it was fully dry.

2.4 Batch adsorption study

To create a 1000 ppm stock solution, one gram of dissolved cadmium or nickel was combined with one liter of distilled water. Additionally, distilled water was utilized to dilute the adsorbed heavy metals to the appropriate concentrations. An electronically calibrated balance (KERN & SOHN GmbH, ABS 220-4N balance, Serial No.: WB1310096) with a 220 g capacity and a 0.0001 g readability was also used to measure and calculate the weight. Additionally, to reach the target pH value for the sample, 0.1 mol/L of either HCl or NaOH was added, and the measurement was taken

with a pH meter that has been calibrated (InoLab pH 7110, WTW, Germany). Throughout a sequence of conical flasks, 100 ml of the heavy metal-contaminated solution was combined with varying volumes of adsorbent and set up to perform a batch adsorption study in a water bath shaker (Grant Instruments Ltd, Model: OLS26, Serial No.: Q72046001, UK) at a constant mixing rate of 150 rpm at 25 °C. Samples were then withdrawn after a specified period of time and filtered using a 0.45 μm membrane (Whatman, Germany). The sample was then filtered, and the heavy metal concentration was determined using an Atomic Absorption Spectrophotometer. Initial concentration (10–60 mg/L), adsorbent particle size (0.075–0.3), detention time (5–120 min), bioadsorbent dosage (0.5–3.0 g/L), solution temperature (15-45°C), and pH (2-7) were among the variables used in this study's tests. The removal efficiency (R%) was computed using the following formulas [18].

$$R(\%) = \frac{(C_o - C_e)}{C_o} \times 100 \qquad (1)$$

Using a mass–balance relationship, it was established that the heavy metal adsorbed onto WS had an equilibrium adsorption capacity of qe (mg/g)[30]:

$$q_e = \frac{(C_o - C_e)V}{m} \qquad (2)$$

where V is the size of the utilized solution (L), m is the mass of the utilized bioadsorbent (g), and Co and Ce are the initial and final (equilibrium) concentrations of the heavy metal (mg/L), respectively.

3. Results and Discussion

This work's primary goal is to investigate how well walnut shell waste biomass can eliminate the heavy metal pollutants Ni(II) and Cd(II) in industrial wastewater. The best conditions for removing nickel and cadmium ions from industrial effluent are found using a batch test. This test involves examining how the adsorption process is affected by pH, adsorbent dosage, adsorption period, starting contaminant concentration, adsorbent particle size, and temperature.

3.1 Impact of Adsorbent Dosage

From an economic perspective, determining the optimal adsorbent dosage is one of the critical factors that must be considered in an adsorption study. To ascertain the ideal dose for eliminating heavy metals (Cd and Ni) from industrial wastewater, an adsorption study using varying concentrations of adsorbent was carried out. The adsorption performance of the adsorbent was tested using amounts of adsorbent ranging from 0.5 to 3.0 g/L and under constant experimental conditions, including initial heavy metal concentration = 30 mg/L, adsorbent particle size = 0.075 – 0.3 mm, stirring speed = 150 rpm, pH = 5, temperature = 25 °C, and adsorption time = 60 min. The results are shown in Figure 1. The removal rate (R%) increased from 36% to 70% for Cd and from 45% to 79% for Ni as the adsorbent dosage improved from 0.5 to 2 g/L. The main reason for this is that a higher adsorbent dosage enhances the removal effectiveness by increasing the amount of active binding sites and the adsorbent surface area[31]. The clearance rate was unaffected by raising the adsorbent dosage above 2 g/L, though. The lack of heavy metal molecules in the water to bind to every active site on the adsorbent and cause surface saturation and equilibrium could be the cause of this[32].

As shown in Figure1 , the heavy metal removal rate decreased from 71% to 70% for Cd and from 80% to 79% for Ni when the dosage of Between 2.5 and 3 g/L, the adsorbent increased. The adsorption process may have left the adsorption sites unsaturated, which could explain this. The way the particles interact, such as aggregation, brought on by a high dosage of adsorbent, could be another factor. The total surface area accessible for adsorption may decrease as a result of this adsorbent aggregation, while the diffusion channel

length may rise[33]. Thus, for additional trials, the ideal adsorbent dose concentration of 2 g/L was selected.

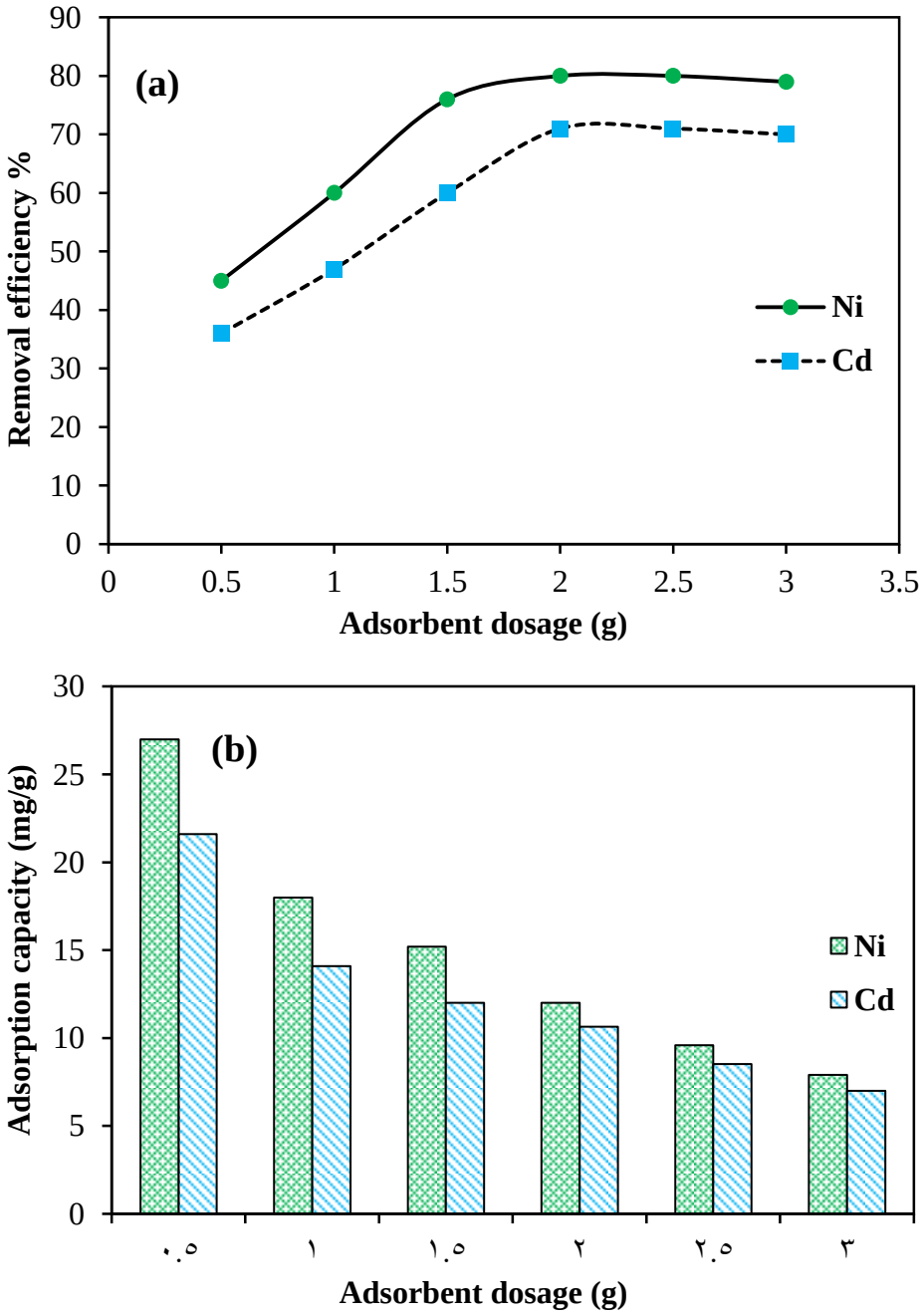


Figure 1: Impact of the adsorbent dosage on (a) the removal efficiency of heavy metals, (b) the adsorption capacity of heavy metals.

3.2 Impact of Initial Concentrations

Since the adsorption response is strongly correlated with the solute concentration, the concentration of the solution significantly affects the adsorption procedure. The initial concentration of pollutants offers a strong driving force in the adsorption process to get past any obstacle preventing the mass transfer of dissolved elements among the solution and solid phases [30]. While all other experimental parameters remained

constant, such this study examined the effects of different starting concentrations of heavy metals (cadmium Cd and nickel Ni) ranging from 10 to 60 mg/L on the adsorption capacity and removal efficiency using the following parameters: adsorbent dosage = 2 g/L, particle size = (0.075 – 0.3) mm, stirring speed = 150 rpm, pH = 5, temperature of solution = 25 °C, and detention time = 60 min. As illustrated in Figure 2, the findings demonstrated that as initial concentrations increased from 10 to 30 mg/L, the adsorption capacity (qe) increased from 3.25 to 12.00 mg/g for Cd and 3.85 to 13.65 mg/L for Ni, while the removal efficiency (R%) of heavy metals increased from 65% to 80% for Cd and77% to 91% for Ni. A higher concentration means more metal ions, so a possible explanation is that increasing the concentration of metal ions in the solution leads to increased mass transfer because of the increased driving force for ion transfer from the water to the surface of the adsorbent. Higher concentrations of Cd and Ni in the solution mean that more metal ions are surrounding the biomass's active sites, which makes the adsorption process more effective. Consequently, as the starting concentrations rose, so did the adsorption capacity[34]. When illustrated in Figure 2, the clearance rate starts to decline when the initial heavy metal content rises above 30 mg/L. This is because the adsorbent sample does not have enough binding sites to absorb metal ions at greater concentrations. The following explanation explains the impact of the initial metal concentration. At low adsorbate-to-adsorbent ratios, the adsorption of contaminants primarily occurs at higher-energy binding active sites. As the heavy metal-to-adsorbent ratio increases (i.e., at higher initial concentrations), adsorption initially occurs at lower-energy binding active sites, while the higher-energy active sites become saturated, leading to a decrease in overall adsorption efficiency. Additionally, observe that throughout the retention time, the adsorption capacity (qe) keeps rising as the original concentration does. The heavy metals' starting mass in the solution establishes the qe values; a higher initial concentration indicates a higher mass of metals that migrate well to the adsorbent's surface[35]. For subsequent trials, the ideal starting concentration of 30 mg/L was thus selected.

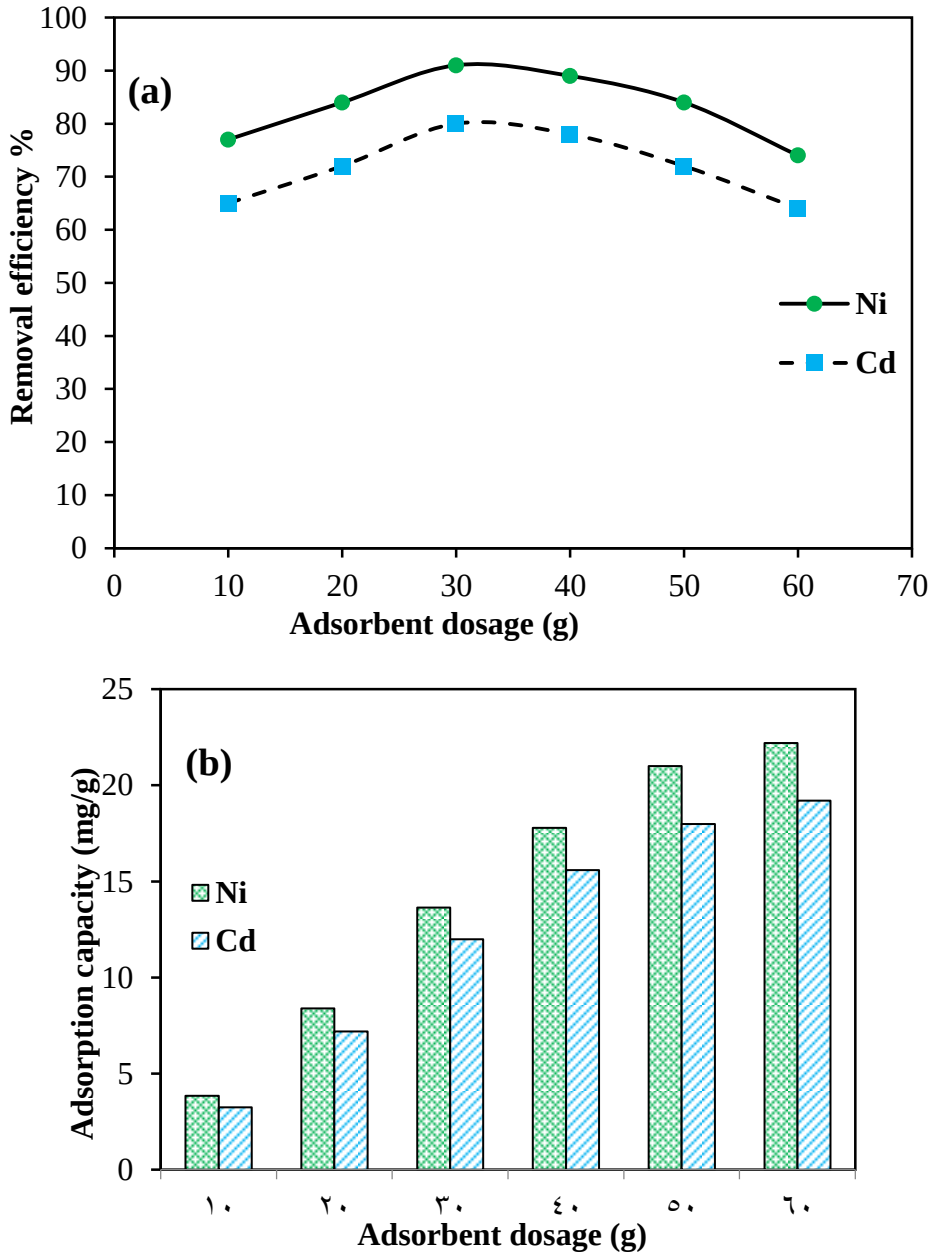


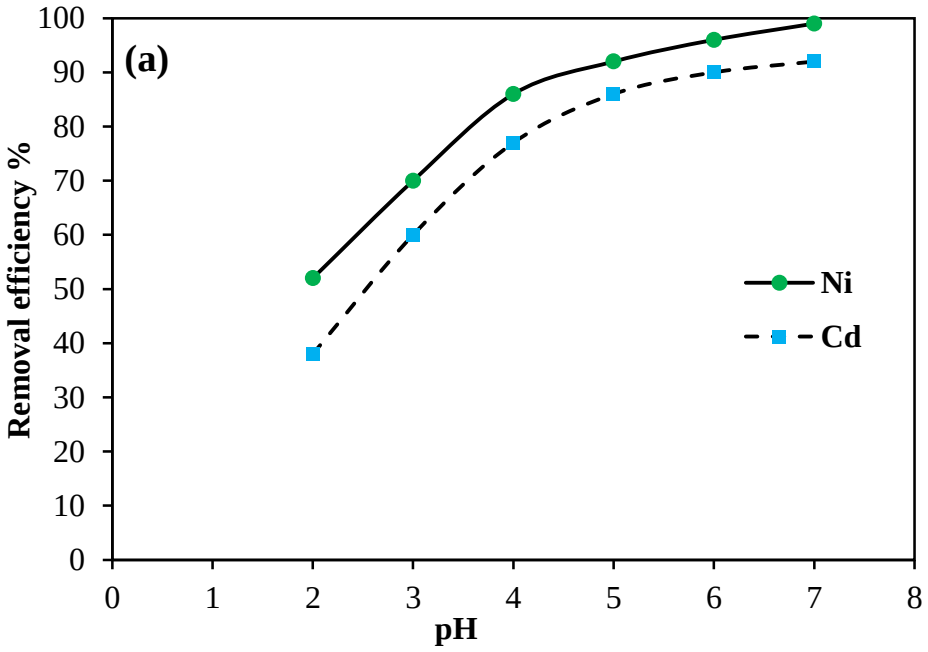
Figure 2: Impact of initial heavy metal concentration on (a) heavy metal removal efficiency, (b) heavy metal adsorption capacity.

3.3 Effect of pH Level

Because it influences both the surface charge of the adsorbent and the type, one important control parameter is the pH of the solution. Both the adsorbent's surface charge and the distribution of pollutants related to their dissociation constants will be impacted by the starting solution's pH. The pH levels usually differ for industrial wastewaters containing heavy metals. The structure of the contaminant and the adsorbent determines how the adsorbed metals interact. As a result, their varying levels of ionization at various pH values alter the metal molecules' net charge. However, the adsorbed molecules may have varying surface charges at changed pH, which has a substantial influence on their affinity for the adsorbent particles[10]. With all other parameters held constant, such as adsorbent dose = 2 g/L, particle size = (0.075 - 0.3) mm,

stirring speed = 150 rpm, initial metal concentrations = 30 mg/L, solution temperature = 25 °C, and detention time = 60 min, the impact of changing the pH of the water from 2 to 7 on the effectiveness of heavy metal removal was investigated. The impact of different pH values on the rate at which heavy metals (nickel Ni and cadmium Cd) adsorb onto the surface of the adsorbent is depicted in Figure 3. When illustrated in Figure 3, it was evident that when pH increased from 2 to 7, the metal ion removal efficiency (R%) rose from 38% to 92% for Cd and 52% to 99% for Ni. Additionally, the adsorption capacity (qe) rose from 5.7 to 13.8 mg/L for Cd and from 7.8 to 14.85 mg/g for Ni.

The increased quantity of metal ion ligands is responsible for the enhanced adsorption of metal ions with rising pH. Additionally, hydronium and positively charged metal ions compete with one another at low pH, which decreases metal ion biosorption[44]. The high removal efficiency at pH values of 5 or higher may result from the precipitation of heavy metals rather than from adsorption efficiency because it has been demonstrated that these metals clump and precipitate in alkaline solutions. As a result, 4 was thought to be the ideal pH number.



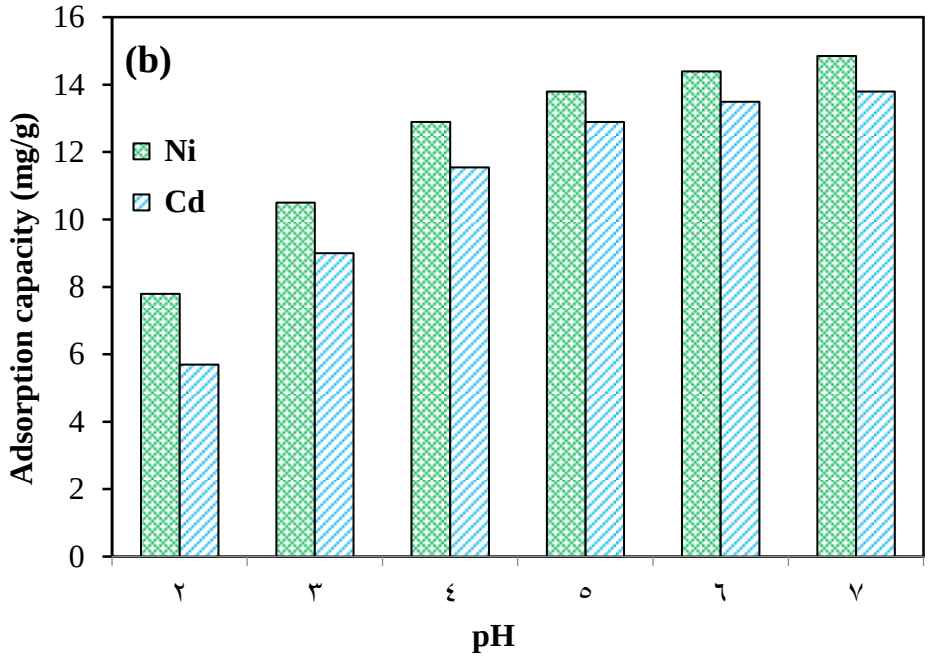


Figure 3: Impact of pH level on (a) heavy metals removal efficiency, (b) heavy metals adsorption capacity.

3.4 Impact of detention time

In the process of heavy metal adsorption, detention time is crucial. The necessary adsorption equilibrium time was determined by examining the impact of detention time on the removal efficiency of heavy metals. While maintaining constant other experimental parameters, including initial heavy metal concentrations = 30 mg/L, pH = 5, solution temperature = 25 °C, particle size = 0.075 – 0.3 mm, speed of agitation = 150 rpm, and adsorbent dosage = 2 g/L, the adsorption time was varied between 5 and 120 minutes to investigate its impact on the adsorption process. The impact of detention time on the removal rate (R%) and adsorption capacity (qe) is depicted in Figure 4. The process of adsorption is done very quickly in the early stages, as long as the surface of the wipe is not saturated with mineral ions. Throughout the first 60 minutes, the clearance rate of heavy metals (Cd and Ni) was extremely quick. As shown in Figure 4, the duration extended from 5 to 60 minutes, and the removal efficiency (R%) rose from 32% to 80% for Cd and 38% to 85% for Ni. Additionally, the qe improved from 4.8 to 12 mg/g for Cd and 5.7 to 12.75 mg/L for Ni. From 60 to 120 min, the removal rate slightly increased and reached equilibrium after 120 min of operation. The removal rate of Cd and Ni was initially rapid because of the large amount of available adsorption sites on the adsorbent surface. After that, the adsorption rate gradually rose, which might be because the dissolved ions took a while to diffuse into the adsorbent's bulk pores[40].

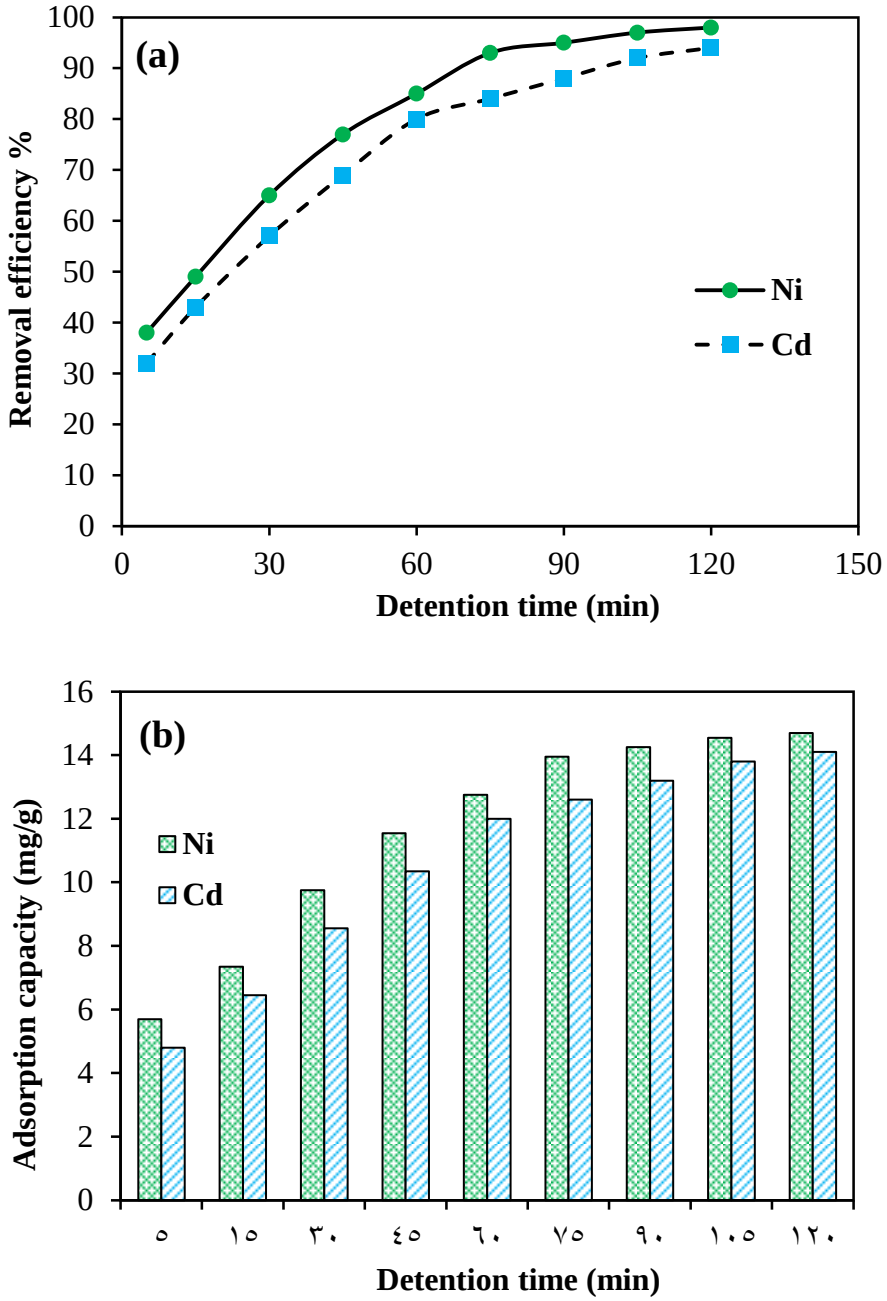


Figure 4: Impact of detention time on (a) heavy metals removal efficiency, (b) heavy metals adsorption capacity.

3.5 Impact of solution temperature

One crucial element in the adsorption process is temperature. A higher temperature accelerates the pace at which adsorbed molecules diffuse through the adsorbent molecules' interior pores and outer boundary layer. Heavy and trace metal adsorption has been significantly influenced by temperature in recent years. The majority of tests were carried out at room temperature, according to data analyses, and often, there were only a few degrees Celsius of temperature difference across studies[36]. With the following ideal parameters: Initial heavy metals concentration = 30 mg/L, pH = 5, detention time = 60 min, adsorbent dose

= 2 g/L, adsorbent particle size = (0.075 – 0.3) mm, speed of agitation 150 rpm, and solution temperature was varied between 15 and 45°C to examine its effect on the adsorption process. As illustrated in Figure 5, the removal efficiency (R%) rose from 60% to 95% for Cd and 68% to 99% for Ni. Additionally, the qe improved from 9.0 to 14.25 mg/g for Cd and 10.2 to 14.85 mg/L for Ni. The results showed that warmth boosts the removal efficiency, suggesting that the metal removal process is endothermic. Because of the internal diffusion-controlled adsorption mechanism, the adsorption capacity rises with increasing temperature. The reason for the resulting shift in the adsorbents' adsorption capacity is that rising temperatures cause the aqueous solution containing the contaminant molecules to become less viscous. This enables faster diffusion of the adsorbent across the adsorbent particles' bulk (external) and porous (internal) limits. Furthermore, when the temperature increases, the adsorbent's porosity and total pore volume increase, increasing the number of active sites accessible for adsorption [37], [38].

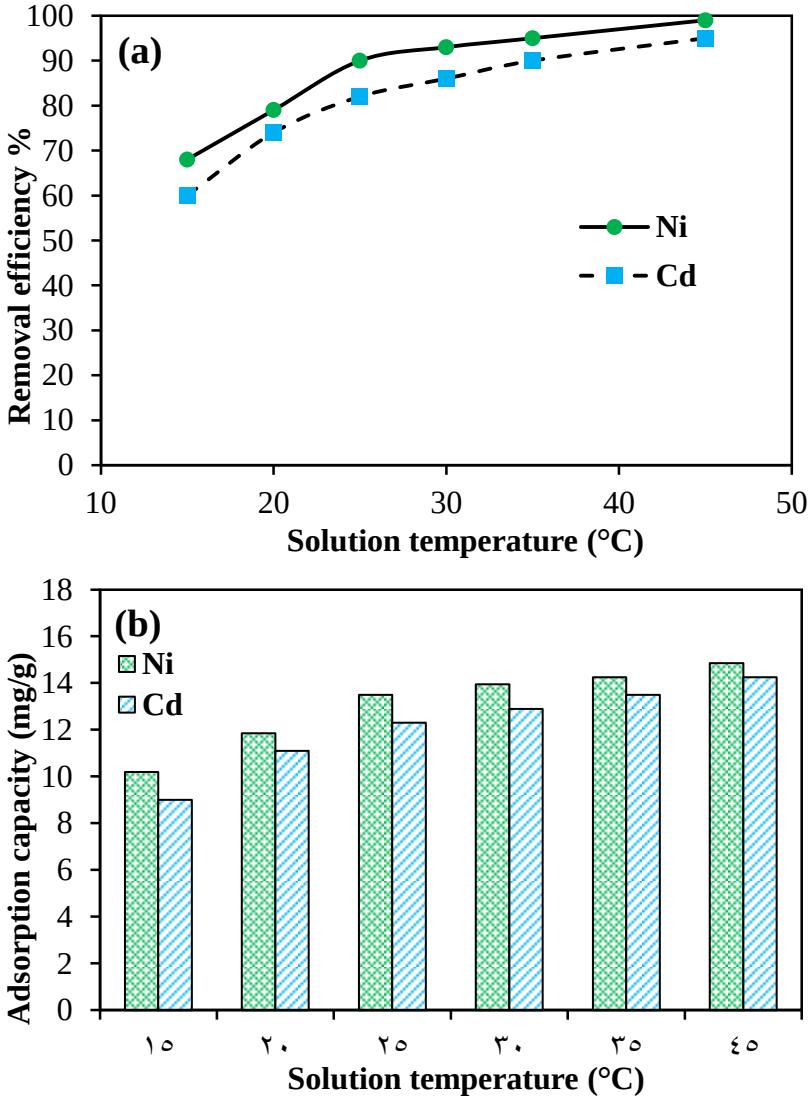


Figure 5: Impact of solution temperature on (a) heavy metals removal efficiency, (b) heavy metals adsorption capacity.

4. Conclusions

Heavy metal (Cd and Ni) adsorption from industrial effluent using batch adsorption techniques was investigated in this study using an adsorbent made from powdered walnut shell waste. To identify the ideal adsorption conditions, a number of investigations were carried out. Several findings emerged from the study's batch experiments: Under ideal circumstances, A removal efficiency of 95% for Cd and 99% for Ni was attained with the successful execution of the adsorption procedure. The optimal parameters for the removal of Cd and Ni were as follows: adsorbent dose = 2 g/L, particle size = (0.075 – 0.3) mm, speed of agitation = 150 rpm, initial Cd and Ni concentration = 30 mg/L, pH = 5, detention time = 60 minutes, and solution temperature = 45°C. The adsorption process and removal efficiency were greatly impacted by the adsorption conditions. Before reaching a certain point, where the removal efficiency tended to stabilize, the removal rate rose in tandem with the dosage. The adsorbent's total surface area may have decreased as a result of active site overlap, which could account for this steady-state removal efficiency. As the starting concentration rose, so did the removal efficiency. A concentration of 30 mg/L was ideal. The adsorbent surface became saturated at increasing starting concentrations, and the energy of the adsorption binding sites started to decrease. Heavy metals have been found to aggregate and precipitate in alkaline environments. Consequently, the precipitation of these metals rather than the adsorption effectiveness may be the cause of the high removal efficiency at pH values of 6 or higher. Consequently, pH 5 was selected. At first, the heavy metal adsorption process was quick. The adsorbent's hollow active sites frequently contribute to the fast adsorption at the first contact time, which subsequently slows down as a result of binding site saturation. It was found that 60 minutes was the ideal equilibrium time between the adsorbent and adsorbent surfaces. The findings indicated that the metal removal process is endothermic since the removal efficiency increased as the temperature rose. The internal diffusion-controlled adsorption mechanism resulted in an increase in the adsorption capacity at higher temperatures. The results indicate that farm waste can be applied as an inexpensive material to remove heavy metals from water. In conclusion, the suggested biosorbent in this work is an economic, effective, and environmentally friendly means to remove heavy metals from industrial wastewater.

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