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Original Research

Removal of Nickel Ion From synthesis wastewater Using Nanoparticles Under Ultrasonic Waves

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ABSTRACT

One of the most significant pollutants released into the environment by industrial processes is heavy metals. The adsorption technique has emerged as a leading strategy for metal ion removal in the last several years. The mass transfer process can be enhanced with the help of ultrasound. This study examined the effectiveness of high frequency ultrasonic waves in increasing the absorption of nickel (II) in aqueous solutions containing Fe_3O_4 nanoparticles. A higher initial amount of adsorbent was associated with a higher removal efficiency of nickel (II). When comparing the effectiveness of using shakers and an ultrasonic test vessel, the latter achieved a maximum efficiency of 84.3% at 60 minutes of contact time, 8 g of adsorbent at pH=5, while the former performed better. On the other hand, while using a shaker and 100 minutes of contact time with 10 g of adsorbent and pH=9, the greatest efficiency was 54.79%. Looking at how contact time affected the adsorption rate, we found that it was fastest at the beginning of the contact period. Increasing the contact duration and equilibrium time resulted in an increase in the amount of nickel (II) elimination from 20 minutes to 60 minutes when using either a shaker or a test vessel. But when using a shaker, the nickel (II) removal rate spiked again between the 60-minute and 80-minute contact times, reaching a peak of 79.54% in the latter. Actually, "cavitation" and high-frequency ultrasonic waves can produce microcurrents that remove a large amount of nickel (II) from water in a very short amount of time. A modest quantity of adsorbent was able to achieve a rapid increase in absorption rate when ultrasonic waves were introduced into the test vessel.

Keywords: Ultrasonic Waves, Fe_3O_4 nanoparticle, Nickel (II), Heavy Metals

1. Introduction

The presence of heavy metals in industrial effluents is a major concern for the environment. Metals and quasi-metals with densities more than 5 g/cm^3 are examples of these contaminants. One example of a metal is lead, but there are many others, including copper, zinc, molybdenum, manganese, iron, arsenic, chromium, mercury, magnesium, and cobalt [1].

The fact that heavy metals are inorganic contaminants rather than biodegradable is their primary drawback. For this reason, heavy metals are now considered a particularly hazardous class of environmental contaminants. Heavy metals have a place in plants, but at concentrations too high they can cause metabolic problems and stunted growth in the vast majority of plant species.

The electronics industry, landfills, military exercises, sludge and sediments from vehicle batteries, foundry industries, metal ore extraction and smelting, military exercises, and agricultural fertilizers are some of the sources of polluting heavy metals. Metals have far-reaching effects on many cellular processes in plants that aren't resistant to them. These include respiration, photosynthesis, mineral feeding, gene expression, and the structure and characteristics of cell membranes [2].

Problems with the nervous system, the digestive system, the bones, the kidneys, the hematopoietic centers, mental retardation, cancer, and other injuries can result from swallowing too much of these metals. Among the many harmful pollutants in our environment, heavy metals rank high. Air, water, construction materials, electronics, and even clothes can contain these harmful substances. While certain levels of some metals are necessary for bodily function, toxic exposure to even trace amounts can be fatal. The inability of the body to digest heavy metals is their primary drawback; this leads to a host of health problems. More so than that, they facilitate the development and dissemination of microbial, fungal, and viral infections. When heavy metals are consumed, they supplant other minerals and salts that the body needs. For instance, cadmium will substitute for zinc in meals if there is a zinc shortage, or heavy metals can accumulate in vascular, muscular, skeletal, and joint tissues [3].

The health impacts of certain metals in the water supply can be catastrophic. Heavy metal poisoning is most harmful when it happens when a person is still developing. A key factor in this effect is the fast development of bodily systems in developing children and fetuses [4]. Heavy metals can damage the brain in extreme situations, rendering it permanently impaired. Because children drink and eat more fluids relative to their body weight, they are more vulnerable to the harmful effects of heavy metals in food and water than adults are. Ultrasonic waves have numerous benefits when used in treatment processes, such as reducing suspended solubility and solubility, increasing biogas production, aiding dehydration, not producing secondary pollutants, and avoiding sludge creation [5]. They are also easy to utilize and may convert materials. These materials can be challenging to break down into less complex components [6]. When treating water and wastewater, ultrasonic vibrations typically speed either biological or chemical processes. Nanoparticles of iron oxide and bentonite are an effective adsorbent for the removal of heavy metals with dual capacities due to their high adsorption rate and specific surface area [7]. The mass transfer process, and by extension the efficiency and speed of reactions in biological processes, is greatly enhanced by ultrasonication [8]. Cavitation, another result of low-frequency ultrasound, changes the size of suspended particles and large molecules in a liquid through powerful mechanical and sonochemical processes. The outcome is enhanced biodegradation. When ultrasonic waves can't eliminate all impurities, they break them down into smaller pieces that can be eliminated biologically. In other words, it's not a capital letter [9].

Zorgani, et al. [7] stated that the prepared g-C_3N_4 was first studied as an adsorbent to remove copper and lead ions by traditional and ultrasonic methods for better dispersion. The pH, metal adsorbate concentration, and contact time were studied in this wide range of the adsorption study. The chemisorption adsorption mechanism is indicated by the pseudo-second-order model and the Langmuir isotherm. The maximum adsorption capacities of Cu^{2+} and Pb^{2+} were 285.7 mg/g and 238.09 mg/g, respectively, suggesting the good performances of this sorbent. After adsorption, the nanocomposites g-C_3N_4@CuNPs/g-C_3N_4@PbNPs was applied in a binary system for the photocatalytic reduction of anionic and cationic dyes with regenerating and recycling of heavy metals. Thermal, structural, textural, and morphological properties of each material were evaluated. Each type of nanocomposite showed a preference for either one, depending on their surface charge. High rate constants for the blue dye (methylene blue), 1.2881 min^{-1} for g-C_3N_4@CuNPs and 0.6406 min^{-1} $^{-1}$ for g-C_3N_4@PbNPs were achieved. This indicates that both organic and inorganic water pollutants were effectively removed.

Gökmen et al. [8] stated that the antidandruff treatments contain the antifungal climbazole (CBZ). In this study, the ketone group in the racemic CBZ molecule was reduced to synthesize CBZ-alcohol, 1-(4-chlorophenoxy)-1-(1H-imidazol-1-yl)-3,3-dimethylbutan-2-ol, and the optimal ultrasonic adsorption conditions were investigated for removing iron(II) (Fe^{2+}) ions from wastewater by adsorption from aqueous solutions. The study's parameters and levels were designed using response surface methods, and model equations optimized the results. The initial pH (1, 3, and 5), adsorption time (30, 45, and 60 min), adsorption temperature (40, 60, and 80°C), and wastewater Fe2+ ion consumption percentage were the independent variables. A wastewater sample from the Uşak Organized Industrial Zone was tested. Individually optimized dependent variable conditions were used for verification trials. Maximum Fe^{2+} consumption was 91.83%. Model R2 was 0.9598. It was found that CBZ-alcohol adsorbs Fe^{2+} from aqueous solution.

1.1. Background

The removal of heavy metals (nickel) from wastewater is a challenging environmental issue, as these materials are toxic and persistent in water systems. Nickel, which is commonly present in industrial wastewater, may be harmful to the environment and human health; therefore, it should be efficiently removed [9]. It has also been documented in the recent studies that NPs [10] are excellent nano-adsorbents for sorption of Ni [II] ions due to

their high surface area and reactivity. Secondly, the as-induced ultrasonic wave can facilitate the adsorption of NPs by achieving higher dispersion and interaction between NPs and target ions. However, further research is necessary to optimize the parameters that enhance adsorptive processes (adsorbent dose and contact time) [11, 12].

Regarding the scope of this paper, it addresses the performance of nanoparticles for nickel ion removal from synthesis wastewater under ultrasound [13, 14]. The objective of the present work is to investigate other influencing parameters such as the adsorbent dose, pH, and contact time on the efficiency of nickel ion removal [15]. The performance of the nanoparticles under ultrasound is compared to that of nanoparticles during their operations via two methods, ultrasonic-assisted adsorption and shaker-based adsorption. The aim of the present study is to obtain theoretical data for optimizing adsorption conditions and improving efficiency.

1.2. Research objectives

- The influence of the concentration of iron oxide nanoparticles used as an adsorbent on nickel (II) adsorption from water in a study employing ultrasonic waves.
- Examination of how pH influences the adsorption of iron oxide nanoparticles onto nickel (II) from aqueous solutions using ultrasonic waves.
- The adsorption effectiveness of nickel (II) from an aqueous solution utilizing ultrasonic waves on the adsorbent of iron oxide nanoparticles was investigated, taking into account the influences of adsorbent dosage, contact time, and temperature.

To determine the concentrations of lead (II) and chrome (VI) ions in a salt solution, Fatehi et al. [44] utilized magnetic nanoparticles attached to activated carbon in 2017. XPS results were obtained before and after the adsorption of chromium and lead ions. The presence of the ions indicated that the adsorption process was effective. Chromium ions had minimal impact on lead ion adsorption in solution, according to the experimental data. The ions of lead (II) and iron (VI) have been removed from the system. Additionally, the research demonstrated the impact of salinity on the adsorption process.
In 2011, researchers examined how activated carbon and PVC-PPA resin absorbed and released chromium (VI) using ultrasound. The results demonstrated that ultrasonication accelerated the adsorption rate in resin and significantly shortened the equilibrium time in activated carbon, but only during the initial stages of adsorption. When tested in pure water, ultrasound did not influence the desorption of chromium (VI) from resin and activated carbon. Activated carbon absorbed more Cr (VI) when NaOH was added to the system, particularly when ultrasonication was present [45].
The simultaneous and competitive removal of (AO), (Er), and (MB) from an aqueous solution was studied by Ghaedi et al. [47] in a container with zinc sulfide nanoparticles placed on activated carbon and ultrasound. To learn more about nanoparticles, XRD, TEM, and FESEM were used. After optimizing the pH effect throughout time, the central composite design (CCD) under the RSM approach thoroughly evaluated the dependence of surface area on dye removal % as well as the magnitude of factors including initial dye concentration, adsorption dose, and ultrasonic time. This model is appropriate for the adsorption mode since it aligns with data gained via practical tests and those predicted through the experimental technique. This is because, with just 0.04 g of adsorbent, a high percentage of paint MB (100%), AO (99.6%), and Er (100%) could be removed in just 2.5 minutes, resulting in an increase.

1.3. The problem statement

The heavy metal, such as nickel ions, in the industrial wastewater can cause environmental and public health problems because of its toxic characteristic and natural water-holding period. Often, conventional wastewater treatment fails to achieve the required nickel removal efficiencies, especially when dealing with complex effluent compositions [20, 21]. Moreover, these techniques could be expensive and environmentally unfriendly. Nowadays, nanoparticles (NPs) have been explored as potentially useful adsorbent materials due to their large surface area and high density of active sites, whereas the optimization issues for real wastewater applications need to be further addressed. Furthermore, the role of ultrasonic waves in promoting the dispersion and adsorption behavior of adsorbents has not been fully developed yet for nanoparticle-assisted heavy metal removal [23]. The performance of sonic engineered nanoparticles for loading efficiency of nickel ions from synthesis wastewater with respect to adsorbent dose, pH, and contact time was examined. Filling this gap is

essential for improving the effectiveness, sustainability, and cost of treatment processes to remediate industrial wastewater and help counter environmental impact on nickel pollution [24].

2. Methodology

2.1. The adsorption of nickel (II) in an aqueous solution using nanoparticles

Industries and companies producing resins, petrochemicals, oil, paint, insecticides, plastics, leather, and pharmaceuticals are the primary producers of nickel (II) compounds. Workshops, smelters, foundries, textiles, ammonia air cleaners for paper, rubber, nitrogen work, nitrogen work, and aircraft repair and maintenance are some other places you might find nickel (II) [16]. Industry involving the weaving and manufacturing of socks. Organic fiberglass compounds, including nickel (II), have been found in groundwater, according to research. This suggests that it is essential to treat industrial effluent for nickel (II) before discharging it into waterways [17]. A number of techniques are available, including ion exchange in resins, ozone oxidation, aerobic and anaerobic biological reduction, and adsorption. Water and wastewater that have been polluted can have soluble organic materials, such nickel (II), removed through the use of activated carbon adsorption. When contrasted with biological treatment, the power of activated carbon to eliminate the vast majority of organic substances stands out [18]. Furthermore, toxicity and other factors that hinder biological therapy do not reduce or cease upon adsorption by activated carbon in terms of removal efficiencies. Nevertheless, there has been substantial advancement in the field of nickel (II) biologicontacty removal research with the goal of improving wastewater treatment processes [19]. As a result, reports have emerged indicating that this technology can remove nickel (II) up to high concentrations. Activated carbon is used in a variety of industrial adsorption procedures, both continuous and discontinuous, or in the form of adsorption columns. The continuous technique was employed in this study. The primary objective of this research is to examine how Fe3O4 nanoparticle-enhanced nickel (II) adsorption in aqueous solution is affected by high-frequency ultrasonic waves. The experimental parameters, such as starting sample pH, starting adsorbent quantity, and contact time, have been adjusted to align with the study's aims. Next, we examined the adsorbent's nickel (II) removal effectiveness under various settings and looked into how well the experimental data matched up with the Friendlich and Langmuir isotherms [20].

2.2. Construction of test container and description of laboratory equipment

The test vessel's primary body is a rectangular cube formed by adhering five 30 cm long, 15 cm wide, and 12 cm high sheets of glass. Five 2.5 cm diameter piezoelectric converters operating at 1.7 MHz are mounted on the base plate of this glass container. Figure (1) shows the real picture of the test vessel.

Figure 1. An image of a test vessel equipped with piezoelectrics.

To ensure that the piezoelectric converter currents have a full impact on the inside space of the test vessel, their placement is carefully considered. There are five piezoelectric MHz 1.7 plates at the base of the test vessel, which can generate efficient mixing. Placement of the piezoelectrics is done via glands. Figure 2 displays the real picture of a piezoelectric and a gland. Two ring rubber gaskets are used to keep fluid from leaking out of the gland when the piezoelectric is placed within. It renders piezoelectric vibration ineffective.

Figure 2. Picture of a sample of piezoelectric, plastic gland and washer.

Since they are in close proximity to the fluid and generate audible currents within it, 1.7 MHz piezoelectric converters are an excellent choice for efficient mixing. Consequently, the fluid within the test vessel receives their energy. A trigger is utilized to activate the piezoelectrics placed in the test vessel, allowing for unique operation of each piezo. Figure (3) shows the real picture of this circuit.

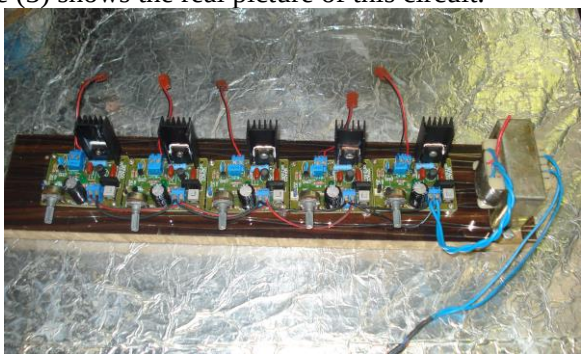


Figure 3. Real photo of a circuit designed to drive piezoelectrics.

2.3. Materials

To make things better, double-distilled water was utilized in every trial. Lab Tech Company's model GS-1330 distilled water apparatus was utilized. Adsorbents manufactured by Asia Research Company, namely Fe3O4 nanoparticles, were utilized to carry out the intended studies. The identified Fe3O4 nanoparticles are shown in Figure (4). Ni (II) solutions were produced for all experiments by dissolving Ni (NO3) 2.6H2O in distilled water and then diluted to the concentration needed for the experiment. At the outset, a 100 mg/L initial concentration of 2.6H2O Ni (NO3) powder was dissolved in 1 L of distilled water in a separate container. Next, the solution was vigorously stirred at room temperature (25 ° C) using a shaker set at 150 rpm. Using a pH meter (Model: Eutech pH 700, Singapore), we tested the pH of solutions containing 0.1 mol% NaOH and HCL to find out how pH affected the results. This study examined the removal of Ni (II) ions from the base solution as a function of adsorbent dose (Fe_3O_4 nanoparticles). A variable ranging from 2 to 8 grams of adsorbent was considered for the evaluation. The adsorbents were dissolved in solutions containing Ni (II) ions in specific amounts to create each solution. Currently, we are investigating four distinct pH values for each solution containing Ni (II) ions and adsorbent. Asia Research Company's Fe3O4 nanoparticles have found adsorption application.

Figure 4. Real photo of Fe_3O_4 nanoparticles made by Asia Research Company used in this research.

2.4. Method of preparing nickel-contaminated samples (II)

Samples of water contaminated with nickel (II) were prepared by dissolving 2.6H2O Ni (NO_3) powder, manufactured by the German company Merck, in distilled water. One liter of distilled water is mixed with 100 mg of 2.6H_2O Ni (NO_3) powder in a separate container. The next step is to thoroughly mix the solution of choice at room temperature (25 ° C) with a shaker set to 150 rpm. Due to the flammability of nickel (II), the test stock solution was prepared in the specified amounts for each set of tests, and the samples were monitored for up to four hours following the adsorption test to ascertain the ultimate concentration of nickel (II).
In the beginning, depending on the desired usage, a specific number of Fe_3O_4 nanoparticles were introduced to 300 ml of water containing nickel (II) with an initial concentration of 100 mg / L. This was done to investigate the influence of ultrasonic waves or shakers on the removal of nickel (II). The device collects samples and then switches off when a certain amount of time has passed. Before smoothing the samples with an English-made standard Watman's filter, we measure the final pH of each sample.

2.5. Preparation of suspension containing nickel (II) and Fe_3O_4 nanoparticles

This study examines the elimination of nickel (II) from the base solution as a function of adsorbent concentration (Fe_3O_4 nanoparticles). With 2, 4, 6, 8, and 10 g of Fe_3O_4 nanoparticles in each of five 1 liter solutions, a range of concentrations may be achieved to create a suspension. The adsorbent is dissolved in a nickel (II) solution in specific amounts to create each solution. Now that we know that the tests work well at three distinct pH levels, we evaluated five different solutions containing nickel (II) and adsorbent at 1, 3, 5, 7, and 9 pH. The medium can be changed or made acidic by adding solutions of sodium hydroxide (0.1 M) or hydrochloric acid (HCl), respectively.

2.6. Laboratory examination of the test container and performing experiments

Two phases of experimentation were carried out in this study:

1- The first step is to transfer the solution containing the nickel (II) ions and adsorbent to a comparable human container. Then, set the container on a shaker and set it to spin at 150 rpm for a specific amount of time. This speed is said to be effective for removing nickel (II) ions, according to the references. After one hour of operation at the specified pace, the stirring was turned off. We investigated the Fe_3O_4 nanoparticles free solution for nickel (II) adsorption recovery by pipetting it into a tiny ionized plastic container.

2-The procedures from the previous stage were replicated in the test vessel to examine the impact of 1.7 MHz ultrasonic waves on the absorption of nickel (II). Before ultrasonic waves are sent into the solution, the driver activates the piezoelectrics, and 1 liter of the nickel (II) and adsorbent suspension is poured into the test vessel. For some while, the solution was subjected to ultrasonic waves. No mechanical stirrer is required, even though the ultrasonic waves' high frequencies produce extremely high mixing. The solution was removed from the test tube after a specific amount of time had passed, and in order to isolate the nanosorbents, it was transferred to a 1 liter beaker and filtered through Whatman filter paper. The recovery rate of nickel (II) from adsorption was studied. We reported the final amount of nickel (II) adsorbed by averaging the three results obtained from each experiment, which were conducted three times at room temperature. Equation (2) was used to compute the percentage of nickel (II) removal and Equation (1) was used to get the adsorption capacity:

$$q_e = \frac{C_0 - C_e}{m} V \quad (1)$$

$$Removal\ \text{efficiency} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

Where qe is the adsorption capacity of nickel (II) in equilibrium, CO and Ce are the initial and final concentrations of nickel (II) in solution, V is the volume of solution and m is the amount of adsorbent used.

2.7. Experimental design

To find out how different nickel removal settings affected the results, the authors used the DOE approach. Parameter optimization in wastewater treatment is a significant economic, technical, and practical issue. An excellent and practical mathematical tool for improving cooling procedures is the response surface methodology (RSM), a statistical and mathematical approach to experiment design. Using RSM, one can optimize connected activities, ascertain the relationship between independent and dependent variables (responses), and so on, up to

the point where various system factors impact outputs. Advanced Composite Design (ACCD) Among RSM's most beloved uses is the remarkable design approach to developing quadratic models. This research was out to optimize the suggested treatment method for nickel removal by using RSM in the planning stages. Specifically, in Design Expert software, version 11/1, the RSM method is utilized to maximize the effectiveness of nickel removal utilizing ultrasonic and shaker. There were three separate parameters whose designs corresponded to four different CCD ranges. The procedure of extracting nickel from aqueous solution was carried out with three independent variables: pH A), contact time B), and adsorbent dosage C). Based on the preliminary research reported in Table 1, these three variables were chosen along with their respective ranges.

Table 1. Coded and reviewed levels of experimental variables.

Name	Units	Low	High	-alpha	+alpha
pH	-	1	9	3	7
Contact time	min	20	100	40	80
Adsorbent mass	g	2	10	4	8

Two central thighs, two axial thighs, and one central thigh form the basis of the central composite design, with n representing the number of elements designed. This study used a three-variable model with eight factorial points, three axial points, and four central point repeats. As a result, 18 tests should be conducted (4+(3)2+8=18). Here we may demonstrate the second-order generic model in the CCD model:

$$Y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j + e_i \quad (3)$$

Y represents the solution. Consider the variables xi and xj. Other parameters for setting include b0 and b, whereas ei stands for an error. The variables and answers related to the procedure were derived by analysis of variance. Coefficients like R2 and Adj-R2 were calculated to evaluate the second-order model's quality.

3. Paying attention to the adequacy of the model

In certain cases, the experimental data of the independent variable cannot be satisfactorily explained by mathematical models that are suitable to the data. One helpful way to think about the right model is analysis of variance (ANOVA). In Table (2), we can see the combined values of the two answers that correspond to a whole design matrix. We have looked into the removal efficiencies of ultrasonics (Y1) and shakers (Y2) as potential response parameters.

Table 2. Experimental ranges Independent parameters applied to CCD design.

		Factor 1	Factor 2	Factor 3	Response 1	Response 2
Std	Run	A:pH	B:Contact time	C:Adsorbent mass	Removal efficiency-US	Removal efficiency-SH
		-	min	g	(%)	(%)
14	1	5	60	4	76.67	70.23
8	2	9	100	10	62.5	79.54
6	3	1	20	2	37.4	34.23
17	4	7	60	6	82.8	78.12
7	5	5	60	6	80.12	74.35
4	6	9	20	2	45.8	42.98
16	7	1	100	10	52.9	71.26
3	8	5	80	6	72.2	78.34
5	9	9	100	2	53.23	74.04
15	10	5	40	6	72.49	60.22
1	11	5	60	6	82.91	73.03
11	12	3	60	6	75.17	69.45
12	13	5	60	6	80.34	73.38
9	14	1	100	2	41.18	65.59
10	15	5	60	8	84.3	75.45
13	16	9	20	10	48.34	43.88
2	17	1	20	10	43.24	40.45

The results of the analysis of variance (ANOVA) test for response functions for the efficiencies of ultrasonic and shaker nickel removal are shown in Tables (3) and (4), respectively.

Table 3. ANOVA test for response functions for nickel removal efficiencies with ultrasonic.

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	4520.89	9	502.32	116.61	< 0.0001	significant
A-pH	178.62	1	178.62	41.46	0.0004	
B-Contact time	143.17	1	143.17	33.24	0.0007	
C-Adsorbent mass	129.56	1	129.56	30.07	0.0009	
AB	8.30	1	8.30	1.93	0.2076	
AC	4.13	1	4.13	0.9594	0.3600	
BC	19.88	1	19.88	4.61	0.0688	
A^2	2.87	1	2.87	0.6658	0.4414	
B^2	173.26	1	173.26	40.22	0.0004	
C^2	0.8052	1	0.8052	0.1869	0.6785	
Residual	30.15	7	4.31			
Lack of Fit	25.34	5	5.07	2.11	0.3525	not significant
Pure Error	4.81	2	2.41			
Cor Total	4551.05	16				

Table 4. ANOVA test for response functions for nickel removal efficiency with shaker.

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	3542.20	9	393.58	123.46	< 0.0001	significant
A-pH	130.03	1	130.03	40.79	0.0004	
B-Contact time	2238.85	1	2238.85	702.31	< 0.0001	
C-Adsorbent mass	51.39	1	51.39	16.12	0.0051	
AB	2.59	1	2.59	0.8118	0.3975	
AC	3.77	1	3.77	1.18	0.3130	
BC	2.05	1	2.05	0.6432	0.4489	
A^2	0.4849	1	0.4849	0.1521	0.7081	
B^2	50.20	1	50.20	15.75	0.0054	
C^2	0.8760	1	0.8760	0.2748	0.6163	
Residual	22.31	7	3.19			
Lack of Fit	21.38	5	4.28	9.14	0.1015	not significant
Pure Error	0.9353	2	0.4676			
Cor Total	3564.51	16				

Both model equations have F values of 116.61 and 123.46, and the fact that the p values are low (<0.0001) indicates that the model is statistically accurate. Adjusted R^2 and correlation coefficient reveal the circumstances under which answers can be influenced by independent variables and their interactions. Indeed, in this case, the values of R2 indicate changes in the model-justifiable responses. R2 values of 0.9934 for Y1 and 0.9937 for Y2 show that the model can explain the variations in the answers. The corrected coefficients (R^2) for Y1 are 0.9849 and for Y2 they are 0.9857. The models' great significance is confirmed by large quantities of adj. R2. Furthermore, it is worth noting that "sufficient accuracy" (Y1: 28.59 and Y2: 33.006) is an additional parameter that takes into account the model's correctness. Both parameters are well-defined enough to show the model clearly. Additionally, in order to find contradicting data and to determine whether the regression model is adequate, a special method is applied to the ANOVA computations. Figure 5 shows that the experimental data for the shaker removal efficiency parameters and the ultrasonic removal efficiency parameters both follow nearly straight distributions, indicating a strong correlation between the two sets of values. This demonstrates a strong grasp of empirical fact. To illustrate the connection between experimental data and expected values from the model, this covered scheme type is helpful. Nevertheless, it may mask critical aspects of the model. But you can see these characteristics clearly in the other natural diagrams, so you can use them to make sure the fit is

good. One of the most useful ways to see if the model needs adjusting or if the analysis's basic assumptions have been met is to look at the residuals graphically.

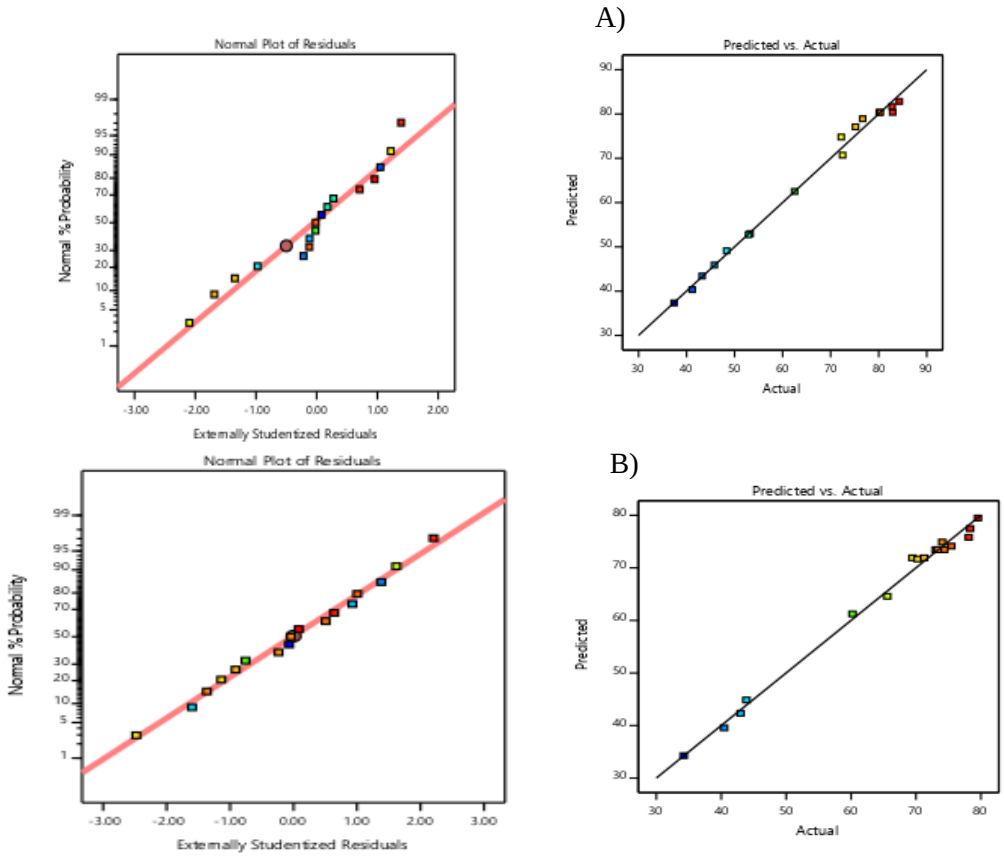


Figure 5. Natural diagram of residual and predicted values versus observed: a) Ultrasonic removal efficiency b) Shaker removal efficiency.

4. Investigation of the effect of ultrasonic on the removal efficiency of nickel (II)

Figure 6 shows that the adsorption speed is highest at the beginning of the contact times by looking at the effect of contact time. Contact and equilibrium times ranging from 20 to 60 minutes led to an increase in the amount of nickel (II) removal in both the shaker and test vessel modes of operation. But when using a shaker, the nickel (II) removal rate spiked again between the 60-minute and 80-minute contact times, reaching a peak of 79.54% in the latter. Since the nanoparticles contain more adsorption sites, the initial adsorption rate for nickel (II) ions is extremely quick. With an ultrasonic-equipped test container, the nickel (II) removal rate is rapid in the first 20-60 minutes, reaching a peak of 84.3% at 30 minutes after contact. However, after 60 minutes, the rate begins to decline. Interestingly, at first, the adsorbent's surface area and number of sites were enhanced through ultrasonic dispersion in the solution, leading to substantial nickel (II) adsorption. However, after 60 minutes, this effect seemed to be attenuated, likely as a result of ion depletion from the adsorbent's surface. Desorption did occur, and the nanoparticles' surfaces did indeed release nickel (II) into the watery solution. Using an ultrasonic test vessel, the impact of pH on the extraction of nickel (II) ions from water was examined in the subsequent section. At pH=9, the amount of nickel (II) removal is at its highest, while at pH=2, it is at its lowest. The trend of changes in nickel (II) removal efficiency has been increasing from 2 to 9.

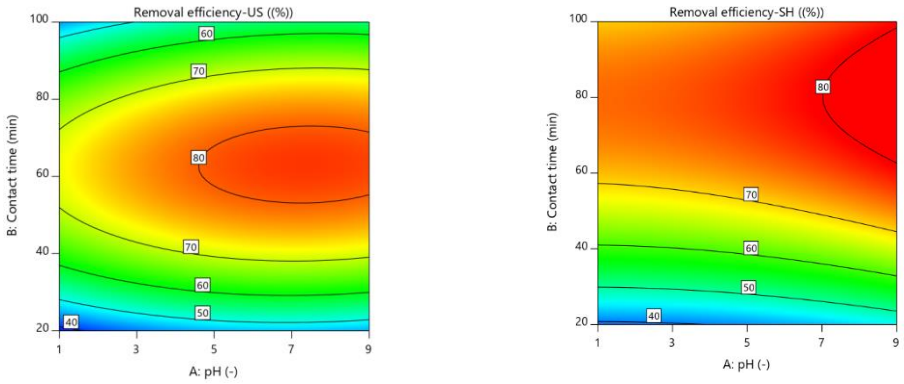


Figure 6. Investigation of the effect of contact time and pH on removal efficiency with a) ultrasonic, b) shaker (adsorbent amount equal to 6 g).

Increasing the initial amount of adsorbent increases the removal effectiveness of nickel (II), and the test vessel equipped with ultrasonic performs better than the shaker in removing nickel (II), as shown in Figure (7), which evaluates the effect of the adsorbent dose. At pH 5 and 9, the maximal adsorption capacities were 84.3 percent and 79.5 percent, respectively, after 60 and 100 minutes of contact with 10 g of adsorbent. Note that in both situations, the optimal amount of adsorbent was 8 to 10 g and the pH range was 7 to 9, according to the study of the effect of the adsorbent dose. In contrast, using an ultrasonic test container with an extended contact time resulted in a decrease in the efficiency of nickel (II) removal, whereas a longer contact time with a shaker improved performance and increased removal efficiency by nearly 7%.

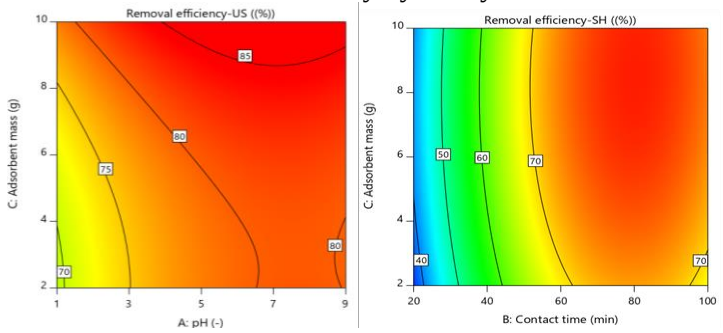


Figure 7. Investigation of the effect of adsorbent dose and pH on removal efficiency with a) ultrasonic, b) shaker (contact time equal to 60 minutes).

Figure 8 shows that at a constant pH of 5, the use of ultrasonic in the test vessel has a superior performance in removing nickel (II) than the shaker, when looking at the simultaneous effect of adsorbent dose and contact duration. An 84.3% maximum efficiency with 60 minutes of contact and 10 g of adsorbent was achieved using ultrasonic technology, whereas 79.54% maximum removal efficiency with 80 minutes of contact and 10 g of adsorbent was achieved using shaker technology. Figure 8 shows that the ideal amount of adsorbent, which was 8 to 10 g in both situations, had the optimum performance.

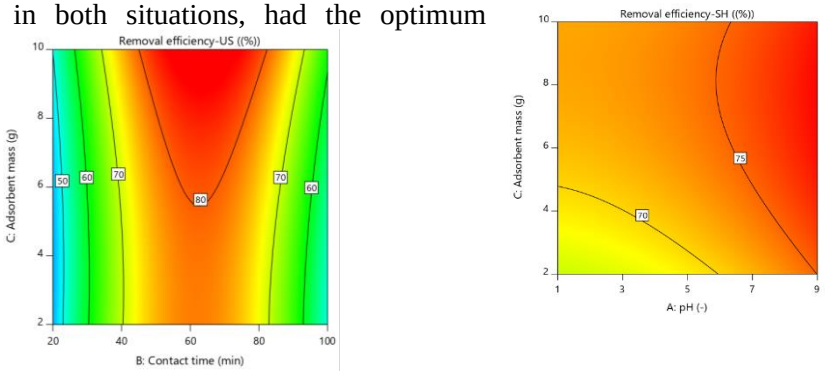
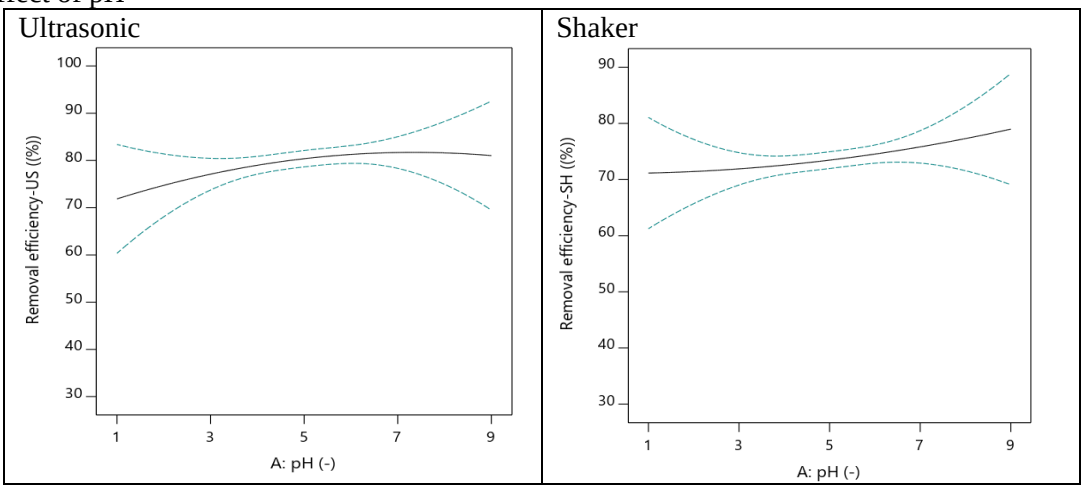


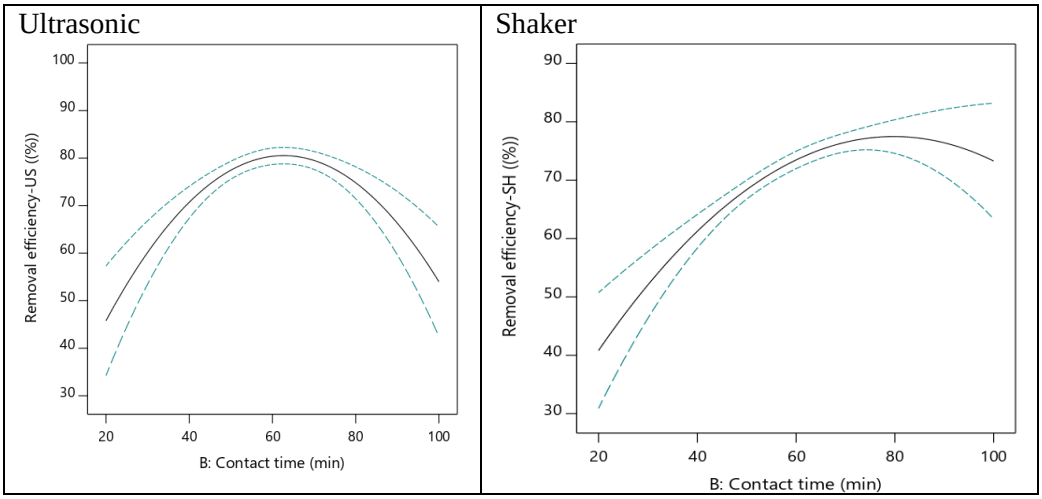
Figure 8. Investigation of the effect of adsorbent dose and contact time on removal efficiency with a) ultrasonic, b) shaker (pH 5).

In Figure (9) all the results of the previous three figures are observed as a single factor to investigate the effect of the parameters and the previous results are confirmed again.

A) The effect of pH



B) Contact time effect



C) Adsorbent dose effect

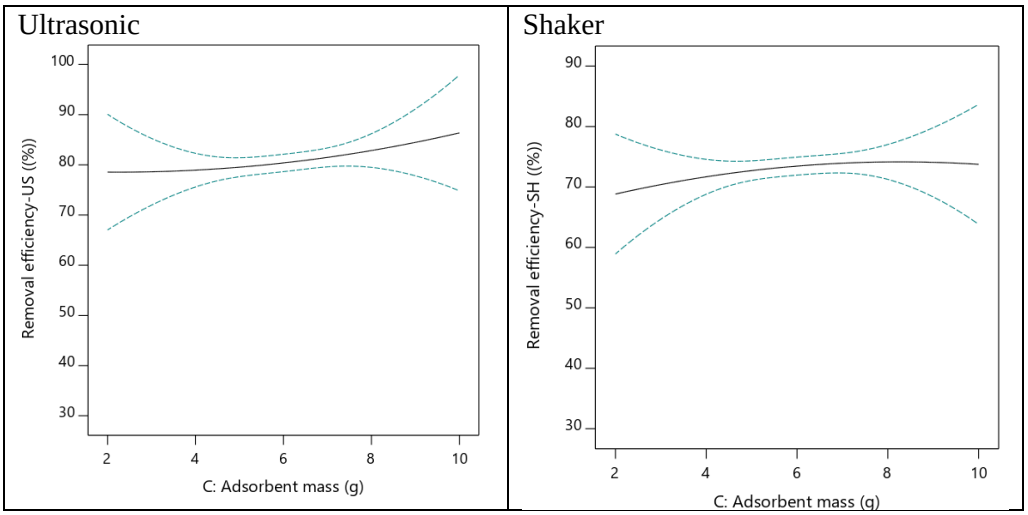
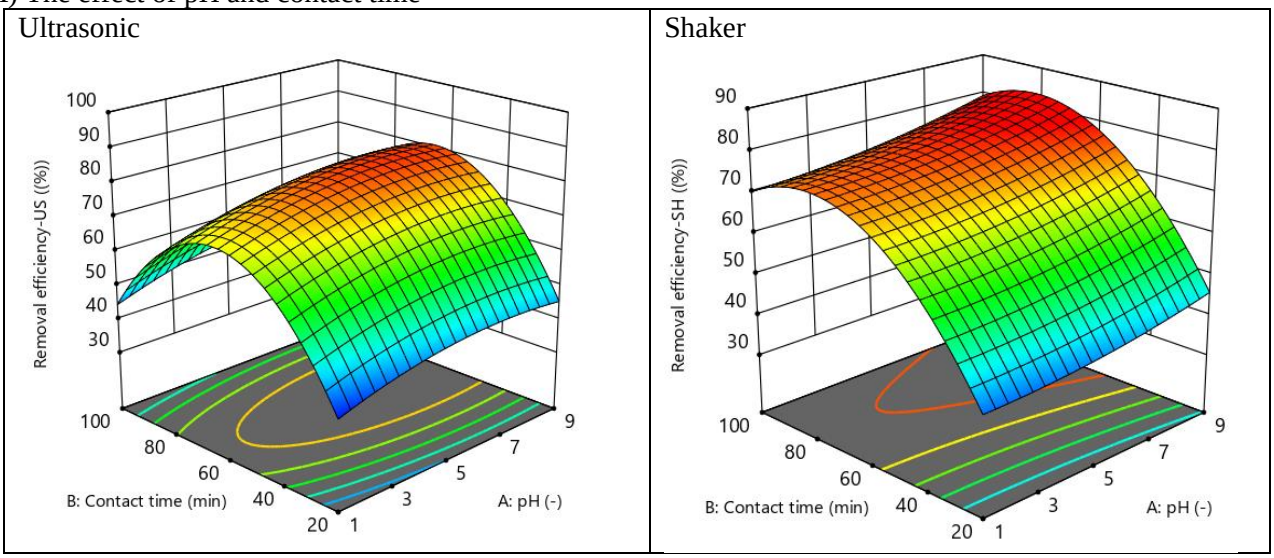


Figure 9. Two-dimensional diagram investigating the effect of parameters on removal efficiencies with ultrasonic and shaker.

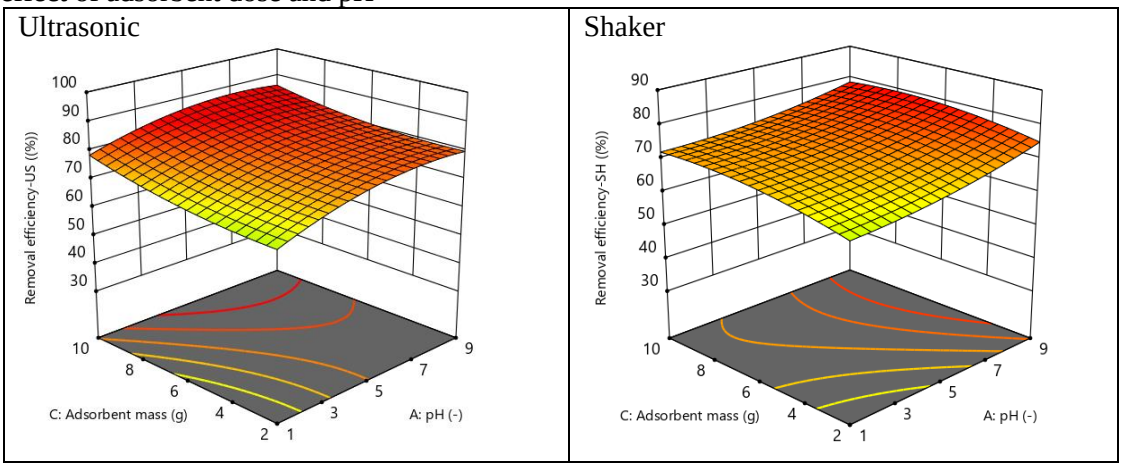
Two parameters on the effectiveness of nickel (II) removal using ultrasonic and shaker can be examined simultaneously in Figure (10) which exhibits three-dimensional graphs. Figure (4-6) a demonstrates that the adsorption rate was greatest during the first contact times. Increasing the contact duration and equilibrium time resulted in an increase in the amount of nickel (II) elimination from 20 minutes to 60 minutes when using either

a shaker or a test vessel. But when using a shaker, the nickel (II) removal rate spiked again between the 60-minute and 80-minute contact times, reaching a peak of 79.54% in the latter. Since the nanoparticles contain more adsorption sites, the initial adsorption rate for nickel (II) ions is extremely quick. With an ultrasonic-equipped test container, the nickel (II) removal rate is rapid in the first 20-60 minutes, reaching a peak of 84.3% at 30 minutes after contact. However, after 60 minutes, the rate begins to decline. An ultrasonic and shaker test vessel study on the removal of nickel (II) ions from aqueous solutions reveals that the removal efficiency of these ions increases as the pH increases from 2 to 9, reaching a maximum at pH 9. At pH 2, the rate of nickel (II) elimination is the lowest, while at pH 4, it is the highest.

A) The effect of pH and contact time



B) The effect of adsorbent dose and pH



C) Effect of absorbent dose and contact time

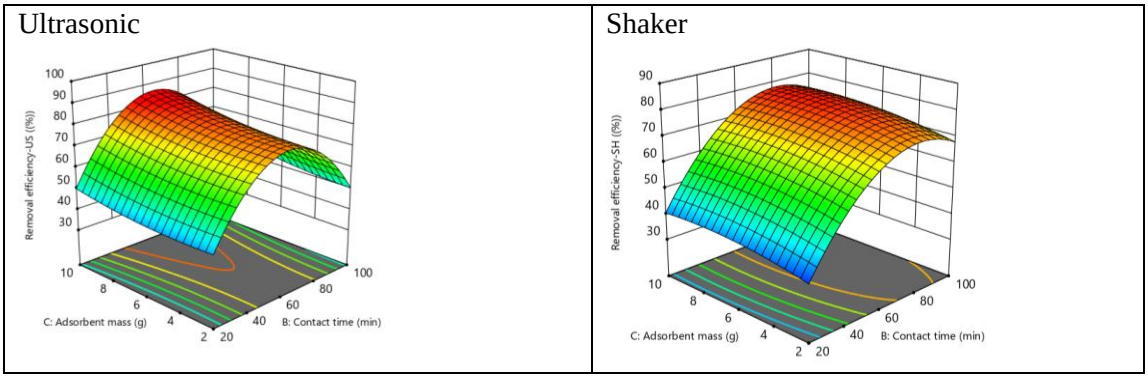


Figure 10.Three-dimensional diagrams to investigate the simultaneous effect of two parameters on the removal efficiency with ultrasonic and shaker.

The results demonstrate that the initial amount of adsorbent has a direct correlation to the removal effectiveness of nickel (II), as shown in Figure (10) b. Interestingly, the test vessel equipped with ultrasonic performed better than the shaker in terms of nickel (II) removal, namely possessing At pH 9, the maximum efficiency was 79.54% after 100 minutes of exposure, while it was 84.3% before. The research of the effect of the adsorbent dose found that 8 to 10 g of adsorbent and a pH between 7 and 9 produced the greatest outcomes in both cases. However, when using an ultrasonic test container, increasing the contact time results in decreased efficiency of nickel (II) removal, whereas increasing the contact time from 60 minutes to 80 minutes improves shaker performance and increases removal efficiency by almost 7%. The findings in Figure (5) c are likewise a composite of those in Parts A and B of the same figure.

The results from the present work also demonstrated that the effect of determining an optimized initial dose (8 to 10 g) was with a percentage efficiency up to ~100% for pH range values of 7-9, in line with prior studies on adsorbent dosages as well as Ni(II) removal. For instance, Karakaya et al. (2025) indicated consistency in the findings that high doses of adsorbents were able to enhance heavy metal removal and were optimized at the highest doses. Besides, the ultrasonic system performance was better than that of the shaker in this study, with a maximum efficiency of 79.54% after 100 min and at a pH of 9 compared to 84.3% as was reported. This is in line with Gökmen et al. (2025), who reported an increased metal ion removal, in terms of a better dispersion and contact between the surface of the adsorbent and metal ions, when applying an ultrasonic system.

The degree of influence of its contact time was also found to be significant in both the IUP and U-UT, as the loss % and removal capacities (relative to 60 min) were increased up to approximately 7% and decreased as shaker operation extended its contact time from 60 min to 80 min for all tests. This result is contradictory to the work of Karakaya et al. (2025), in which contact time is more likely to result in the effective removal of metal ions since both ultrasound and mechanical methods were used; thus, the optimization of the ultrasonic system may have to be reconsidered.

The results obtained in this study are consistent with previous works indicating that adsorbent dose and contact time are controlling parameters of the biosorption process. However, the difference between the shaker experiment and the ultrasonic method indicates the necessity for a better understanding of ultrasound optimization to attain better removal efficiency. This comparison demonstrates that there is much potential to further enhance ultrasonic-assisted adsorption systems in the future.

Applications of the results in environmental detoxification and industrial waste treatment are very attractive. Nanoparticles as adsorbents are of tremendous advantage in the removal of toxic metals, especially nickel ions from industrial wastes. Due to its frequent presence in synthesis wastewater, nickel represents severe ecological risks with impact on water bodies, soil, and diverse aquatic organisms. Utilizing nanoparticles combined with ultrasonication, this work introduces a novel technology that could open the door for better and less expensive treatment techniques than classic chemical methods.

The contribution to ecologically based wastewater treatment technology is considered. The recovery of heavy metals (Ni, Cu) from wastewaters via a green synthesis protocol with minimal usage of hazardous chemicals is essential for the prevention of environmental pollution. The optimization of the influencing factors (the adsorbent dose, contact time, and pH) is also studied by this work, giving useful guidance for the promotion of using nanoparticle-adsorption systems on a large scale in practice. The incorporation of ultrasonic waves promotes the dispersion and interaction of nanoparticles, and as such, this approach may prove useful in emerging water purification technologies.

The findings could have wider implications for the public health community by presenting measures to minimize exposure risks related to toxic contamination of drinking water supplies. The increase of the Ni^{2+} removal efficiency facilitated by this study could help to achieve environmental protection standards and promote environmental regulations in terms of industrial pollution reductions.

In brief, the paper is a promising alternative in terms of efficiency for nickel removal from industrial wastewater (economic and environmental perspectives) and opens new prospects for research on nanomaterials and advanced water treatment.

5. Optimal values

The three numerical factors involved in the process of nickel (II) elimination by iron oxide nanoparticles are being optimized in the experiments. When calculating the effectiveness of ultrasonic and shaker removal, three-factor interactions in polynomial model equations are taken into account. Using analysis of variance (ANOVA),

we checked the responses for quality and found that the variables are taken into account to improve the efficiency of nickel removal. In RSM, the numerical optimization section takes into account the best possible circumstances for critical parameters in order to achieve the targeted values for the shaker removal efficiency and ultrasonic removal efficiency. Table 4-4 compiles the goal category, the range of values for the variables and responses, and the intended values for the variables and responses. The goal of this optimization is to find the best possible values for the variables and output parameters because these factors have a significant impact on the effluent heavy metal removal efficiency when utilizing adsorbents. The verification assessment and data gathered from the utility optimization using the CCD show that the responses (ultrasonic removal efficiency and shaker removal efficiency) are within the permissible agreement.

Table 5. Limitations of the three variables studied and both responses for optimization.

Number	**pH**	**Contact time**	**Adsorbent mass**	**Removal efficiency-US**	**Removal efficiency-SH**	**Desirability**	
1	**8.133**	**65.881**	**8.577**	**84.785**	**79.990**	**1.000**	**Selected**
2	7.988	67.949	8.585	84.620	80.355	1.000	
3	8.155	67.730	9.344	85.856	80.322	1.000	
4	8.117	69.499	8.969	84.913	80.839	1.000	
5	6.614	76.500	9.893	84.379	79.566	1.000	
6	8.921	63.958	9.511	85.851	80.280	1.000	
7	8.812	68.397	9.756	86.068	81.327	1.000	
8	8.827	62.680	9.676	86.181	79.579	1.000	
9	7.798	67.270	8.348	84.411	79.928	1.000	
10	7.363	70.473	8.917	84.800	79.982	1.000	
11	8.455	70.024	9.117	84.871	81.432	1.000	
12	8.425	63.150	8.397	84.418	79.566	1.000	
13	7.059	74.839	9.787	84.978	79.994	1.000	
14	8.900	64.934	9.885	86.541	80.375	1.000	
15	7.710	66.447	8.437	84.668	79.555	1.000	
16	8.316	68.065	8.503	84.321	80.903	1.000	
17	6.753	74.581	9.854	85.178	79.550	1.000	
18	8.447	71.673	9.592	85.309	81.565	1.000	
19	7.525	68.486	8.400	84.360	79.846	1.000	
20	7.357	68.354	8.407	84.412	79.577	1.000	

6. Conclusions

In this study, Fe_3O_4 nanoparticles were used to test how high frequency ultrasonic waves intensified nickel (II) adsorption in aqueous solution. Investigating the impact of adsorbent dose, it was noted that the initial amount of adsorbent had a direct correlation to the removal efficiency of nickel (II). Interestingly, when comparing the two methods, the test vessel equipped with ultrasonic performed better in terms of nickel (II) removal (maximum efficiency of 3) compared to the shakers. With a pH of 5 and 9, 84% and 79.54%, respectively, were achieved after 60 minutes of contact with 8 g of adsorbent, and after 100 minutes of contact with 10 g of adsorbent, respectively. The research of the effect of the adsorbent dose found that 8 to 10 g of adsorbent and a pH between 7 and 9 produced the greatest outcomes in both cases. However, when using an ultrasonic test container, increasing the contact time results in decreased efficiency of nickel (II) removal, whereas increasing the contact time from 60 minutes to 80 minutes improves shaker performance and increases removal efficiency by almost 7%.

Looking at how contact time affected the adsorption rate, we found that it was fastest at the beginning of the contact period. Increasing the contact duration and equilibrium time resulted in an increase in the amount of nickel (II) elimination from 20 minutes to 60 minutes when using either a shaker or a test vessel. But when using a shaker, the nickel (II) removal rate spiked again between the 60-minute and 80-minute contact times, reaching a peak of 79.54% in the latter. Since the nanoparticles contain more adsorption sites, the initial adsorption rate for nickel (II) ions is extremely quick. The removal rate of nickel (II) in the test container

equipped with ultrasonics is extremely quick in the first 20-60 minutes, reaching a high of 84.3% at 30 minutes after contact. After 60 minutes, however, the removal rate begins to decline. Interestingly, at first, the adsorbent's surface area and number of sites were enhanced through ultrasonic dispersion in the solution, leading to substantial nickel (II) adsorption. However, after 60 minutes, this effect seemed to be attenuated, likely as a result of ion depletion from the adsorbent's surface. Desorption did occur, and the nanoparticles' surfaces did indeed release nickel (II) into the watery solution. As a result of the cavitacin phenomenon and the generation of micro-dimensional sound currents by high-frequency ultrasonic waves, it is possible to remove a large percentage of nickel (II) from aqueous solutions in a relatively short amount of time. The adsorption rate was quickly boosted with a tiny amount of adsorbent when ultrasonic vibrations were applied to the test vessel. Using an ultrasonic test vessel, the impact of pH on the extraction of nickel (II) ions from water was examined in the subsequent section. At pH=9, the amount of nickel (II) removal is at its highest, while at pH=2, it is at its lowest. The trend of changes in nickel (II) removal efficiency has been increasing from 2 to 9. In the future work, further exploration should be conducted to optimize the ultrasonic parameters (frequency, intensity, and time of exposure) for improved efficiency in removing nickel ions. In this respect, it is important to study the regeneration and reusability of the particles, which could ascertain cost-effective and sustainable applications for prolonged periods. Moreover, the application of this process at an industrial plant should be assessed in pilot-scale studies. Extending the work for multicomponent metal ion removal might open up new opportunities, and further ecotoxicity studies of nanoparticles are also required to evaluate their safety and environmental impact.

Declaration of Competing Interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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Author Contributions

Ms. Walaa Abdulkhaleq Zghair led the formulation of the research problem, collected recent articles, and organized them into clear, accessible formats. She also designed and proposed the work, and discussed the proposed design. Ms. Mannal Muhammed and Asst. Prof. Dr. Nuralhuda Aladdin Jasim verified the recommendations presented in the proposed work and contributed to the design and discussion of the results. All authors contributed to the final version of the paper, discussing the results and refining the manuscript.

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