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Research Article

Statistical Approach for Removing Some Heavy Metals from Water Samples Using Natural Zeolite

Abdulsalam A. Matlob[1], Abbas F. M. Alkarkhi[2]

[1]Department of Medical Biochemical Analysis, Cihan University-Erbil, Kurdistan Region, Iraq, [2]Department of Anesthesia Technology, Cihan University-Erbil, Kurdistan Region, Iraq

ABSTRACT

The need for contaminant-free water in agriculture, industry, and medicine makes the removal of heavy metals from water a priority because of their high potential for toxicity and bioaccumulation. This study aimed to examine the effect of readily available and inexpensive, yet promising, natural zeolite, clinoptilolite, on removing heavy metals, specifically nickel (Ni) and chromium (Cr), from prepared water samples. Zeolite was used as an adsorbent. A two-level full factorial design was used to determine the main and interaction effects of three factors (amount of zeolite, pH, and metal concentration) on the removal percentage of Cr and Ni ions from the prepared samples. Batch processes were conducted using aqueous solutions under specific laboratory conditions. The results showed that the clinoptilolite zeolite can remove heavy metals from water samples. Highest removal percentages were 43% and 41% for Cr and Ni ions, respectively. The results show an interaction effect between zeolite amount and pH for Cr ion removal, and between metal concentration and pH for Ni ion removal. There was no main effect of pH on Cr ion removal. However, there was no main effect of zeolite amount on Ni removal.

Keywords: Factorial design, clinoptilolite natural zeolite, nickel, chromium, response surface methodology

INTRODUCTION

The presence of trace heavy metals, including lead, mercury, cadmium, and arsenic, in aqueous environments facilitates chronic daily exposure through routine utilization and ingestion. The contaminated water poses a significant risk to human health and the entire stability of the ecosystem. As such, metals can easily leach into the water through industrial waste, agricultural runoff, and sometimes illicit sources.[1]

Heavy metals are considered a high environmental risk due to their adverse effects on water quality.[2,3] Due to the high solubility of these metals in aquatic environments, they can be readily consumed by a variety of microorganisms,[4,5] which eventually enter the human food chain as larger organisms eat them, causing serious health complications.[6,7] The toxic effect of the contaminated water would reach humans through food consumption and cause major health issues that could be acute, chronic, mental, or even cancerous diseases.[8] These heavy metals can easily accumulate in the ecosystem, posing significant risks as they do not break down readily in the environment.[9]

Therefore, following effective methods to help remove these metal particles is an essential practice to maintain a healthy ecosystem and protect public health.[10] Several methods were introduced traditionally to remove large quantities of this metallic substance, such as chemical or physical methods, which could occur in high costs.[11] Hence, the use of zeolite has been introduced in recent studies to outperform the traditional methods in removing metal ions in the environment using absorption techniques.[12] One of the main possible solutions introduced in various research[13] is the removal of materials, specifically natural or synthetic crystalline aluminum silicate, which have properties that allow for quick adsorption and ion exchange. The original planted formula is efficient in water products due to its unique properties, for example, its large surface area and the cation exchange capacity, making it a good choice for preventing the recording of fixed minerals in the water.[3] The equipment features higher optical stability and resistance to adaptation in various environments.

Zeolites work in a unique procedure to remove heavy metals through ion exchange and absorption. Cations within

Corresponding Author:
Abdulsalam A. Matlob, Department of Medical Biochemical Analysis, Cihan University-Erbil, Kurdistan Region, Iraq.
E-mail: abdulsalam.atallah@cihanuniversity.edu.iq

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the zeolite structure exchange with heavy metal ions in the water, causing them to be absorbed into the zeolite structure, and thereby purifying the contaminated water.[14]

Moreover, the physical ability of adsorption of heavy metals increased due to the porous nature of the zeolite, which leads to enhancing its impact. Many advantages arise from using zeolitic materials, including ease of regeneration for more applications, low cost, and natural abundance. Zeolite materials can be used in many water treatment processes, such as in industry, for wastewater treatment, and water filtration for domestic purposes. Finally, environmental pollution problems of water can be addressed and solved sustainably and effectively using zeolite. To use technologies with significant effects to contribute to global efforts aimed at protecting and conserving our water resources, zeolite appears to be a promising approach and environmentally friendly material.[15,16] In diverse fields such as psychology, sociology, biology, and engineering, factorial design for response surface methodology is a commonly used experimental approach.[17]

The most widely used experimental method across fields such as psychology, sociology, biology, and engineering is the factorial design. This statistical approach enables researchers to evaluate the effects of multiple independent variables (factors) and their interactions on a response simultaneously. Factorial design can be used in two main types of studies: experimental and quasi-experimental, offering high efficiency and flexibility compared to other designs.[18]

In a factorial design, each independent variable is manipulated at different levels, and all possible combinations of these levels are tested. This results in a matrix of experimental conditions in which the effects of each independent variable and its interactions can be systematically analyzed.[19] Factorial designs can be classified into intergroup, intragroup, and mixed designs, depending on how participants are distributed across experimental conditions.[20]

In this study, the ability of natural zeolite to remove chromium (Cr) and nickel (Ni) ions from water samples was evaluated. Furthermore, the main and interactive effects of three factors (zeolite quantity, metal concentration, and pH) on heavy metal removal from water were determined using a two-level factorial design.

MATERIALS AND METHODS

All chemicals used were of reagent grade. A 2^3 factorial experiment was applied; it considers two levels (the base) for three factors (the superscript), producing $2^3 = 8$ factorial points. The design variables used in this study were the zeolite dosage as the adsorbent, the pH of the water samples, and the concentration of the treated metal samples. The response is the percent removal of Cr and Ni ions. The synthetic water samples were prepared using potassium dichromate ($K_2Cr_2O_7$) and nickel(II) chloride hexahydrate ($NiCl_2{\cdot}6H_2O$) as sources of Cr and Ni ions, respectively. The pH of the water samples was adjusted with synthesized hydrochloric acid and sodium hydroxide solutions. In this study, clinoptilolite – a natural zeolite – was employed to extract Cr and Ni ions from water samples. The clinoptilolite was of Turkish origin and obtained from the local market. The grain size was used as received from the company: 20 µm. Batch experiments were performed in eight runs at room temperature using 100 mL of the prepared metal solution. For each run, the solution was adjusted to a specific pH, amended with a predetermined mass of zeolite according to the experimental design, and agitated using a magnetic stirrer for 1 h. The mixtures were filtered to recover the filtrate for inductively coupled plasma (ICP) analysis.

The experimental runs were conducted at the laboratory of the College of Health Technology, Medical Biochemical Analysis Department, Cihan University - Erbil, from February 2023 to February 2024.

Description of Samples

The two levels of concentration for the two water samples, chosen and prepared according to the requirements of the factorial design, were 100 ppm and 400 ppm. The sources of Cr^{3+} and Ni^{2+} ions were $K_2Cr_2O_7$ and $NiCl_2{\cdot}6H_2O$, respectively. A Shimadzu ICPE-9820 simultaneous ICP atomic emission spectrometer was used to measure concentrations. The experimental design, including the factors and their levels used in the factorial design for Cr and Ni ions, is illustrated in Table 1.

Table 1: The factors and levels used in the factorial design for Cr and Ni ions

Experiment	Metal concentration ppm	pH	Amount of zeolite g
1	400	10	0.5
2	100	4	0.5
3	100	10	3.0
4	400	10	3.0
5	100	10	0.5
6	400	4	0.5
7	100	4	3.0
8	400	4	3.0

Cr: Chromium, Ni: Nickel

RESULTS AND DISCUSSION

A non-poisonous, three-dimensionally sieve-like, crystalline, hydrated aluminosilicate with natural adsorption and ion-exchange properties, similar to those of zeolite, enables this material to remove heavy metals and soluble toxins from drinking water. Table 2 shows the outcome of the X-ray fluorescence test for analyzing the chemical structure of natural zeolite. The top oxide concentrations in the natural zeolite are silica and alumina, at 68.5% and 12.3%, respectively. The entirely crosslinked open framework tetrahedral structures in the zeolite come from silicon oxide and aluminum oxide, which consist of corner-sharing SiO_4 and AlO_4. This structure leads to the formation of small pores, 1–20 A in diameter, throughout the solid.[21]

Removal of Cr ions

The percentage of removed Cr ions based on ICP results showed the natural zeolite's capability to remove heavy metal ions from contaminated water. As shown in Table 3, the

Table 2: X-ray fluorescence test of the chemical structure of natural zeolite clinoptilolite

Compound/Element	Unit	AB1
SiO_2	%	68.5
Al_2O_3	%	12.3
Fe_2O_3	%	2.4
CaO	%	3.1
MgO	%	1.5
Na_2O	%	0.6
K_2O	%	3.1
TiO_2	%	0.1
MnO	%	0.1
P_2O_5	%	<0.02
Loss on ignition	%	8.2
S	ppm	91
CI	ppm	774
Sr	ppm	1992
V	ppm	27
Zn	ppm	14
Zr	ppm	20
Ce	ppm	29
La	Ppm	42

SiO_2: Silicon dioxide, Al_2O_3: Aluminum oxide, Fe_2O_3: Iron (III) oxide, CaO: Calcium oxide, MgO: Magnesium oxide, Na_2O: Sodium oxide, K_2O: Potassium oxide, TiO_2: Titanium dioxide, MnO: Manganese (II) oxide, P_2O_5: Phosphorus pentoxide, S: Sulfur, Cl: Chlorine, Sr: Strontium, V: Vanadium, Zn: Zinc, Zr: Zirconium, Ce: Cerium, La: Lanthanum

Table 3: Cr % removal with factors and levels used in factorial design

Experiment	Metal concentration ppm	pH	Amount of zeolite	Cr % removing
1	400	10	0.5	6.5
2	100	4	0.5	13.0
3	100	10	3.0	35.0
4	400	10	3.0	15.0
5	100	10	0.5	27.0
6	400	4	0.5	12.5
7	100	4	3.0	43.0
8	400	4	3.0	19.2

Cr: Chromium

highest Cr % removal was 43%. This result belongs to the run in the factorial design with metal concentration at 100 ppm, pH at 4, and zeolite amount at 3 g. The result indicates that the highest removal percentage was obtained at the maximum zeolite amount, whereas the metal ion concentration was lowest in the acidic solution. The low Cr removal efficiency is likely due to the presence of various oxides in the natural zeolite, which obstruct its metal-binding capacity. To improve future Cr removal, pre-treatment methods such as thermal or chemical modification could be employed to synthesize an industrial zeolite that removes these impurities and exposes more active sites.

Two-way analysis of variance (ANOVA) of Cr and Ni ions removal

A two-way ANOVA was conducted to examine the effects of metal concentration and zeolite quantity on the removal of Cr and Ni ions. As shown in Table 4, the analysis yielded a strong model with an adjusted R-squared of 75.06%, indicating that it explains over three-quarters of the variance in removal effectiveness. Results revealed that both the initial metal concentration ($P = 0.022$) and the zeolite quantity ($P = 0.040$) had statistically significant impacts on the percentage of Cr ions removal from water samples, since both P-values were below the significance threshold ($\alpha = 0.05$). This means that changing either factor significantly influences the removal outcome. However, the interaction between metal concentration and zeolite quantity was not significant ($P = 0.267$), suggesting that the effect of one factor does not depend on the level of the other. The lack of a significant interaction simplifies the interpretation of the main effects, indicating that the influence of zeolite amount is consistent across different mineral concentrations, and vice versa.

Main effects plot (data means) for Cr ions removal

The main effect is the effect of a single independent variable (factor) on the dependent variable (response), while ignoring the effects of other independent variables. Figure 1 shows the plot of the main effect of the three factors, amount of zeolite, pH, and metal concentration, on the removal percent. As shown in Figure 1, the main effect of the amount of zeolite on the removal percentage increased when going from a low level (0.5 g) to a higher level (3 g). For metal concentration, the removal percent for Cr ions was increased when going from a high level (400 ppm) to a low level (100 ppm). For pH, the removal percentage remained almost constant at both high and low levels. This means no effect on the response when the pH level was changed. Therefore, the pH was ignored from the two-way ANOVA analysis. Besides, the selected two factors, the amount of zeolite and metal concentration, for the ANOVA analysis, are most central to the research question. In addition, the study focuses specifically on the interaction of the two most impactful selected factors. Adding pH might be part of a "future study" rather than the current objective.

Interaction plot (data means) for Cr% removing

In general, the interaction plot was used to show how the relationship between one factor and a continuous response depends on the value of the second factor. Interaction plots were generated and discussed to highlight the interaction effect observed during Cr ion removal using natural zeolite. The more non-parallel the lines are, the greater the strength of the interaction. The interaction plot of zeolite amount versus pH [Figure 2] shows a significant interaction effect. This is due to the non-parallel plot lines, which means that the percentage of removed values (y-axis) at any zeolite level is not steady but varies with pH. The highest removal rate occurred with 3 g of zeolite at a lower pH of 4, but this conclusion will change when the highest mean removal value is obtained at the higher pH of 10. This change in the removal mean value is due to the interaction effect between the two factors, although

Table 4: Two-way analysis of variance of Cr ion removal versus metal concentration and amount of zeolite

Source	Degrees of freedom	Sum of squares	Mean square	F	*P*-value
Metal concentration	1	524.88	524.88	13.39	0.022
Amount of zeolite	1	353.78	353.78	9.02	0.040
Interaction	1	64.98	64.98	1.66	0.267
Error	4	156.82	39.205		
Total	7	1100.46			

S=6.261 R-Sq=85.75% R-Sq (adj)=75.06%

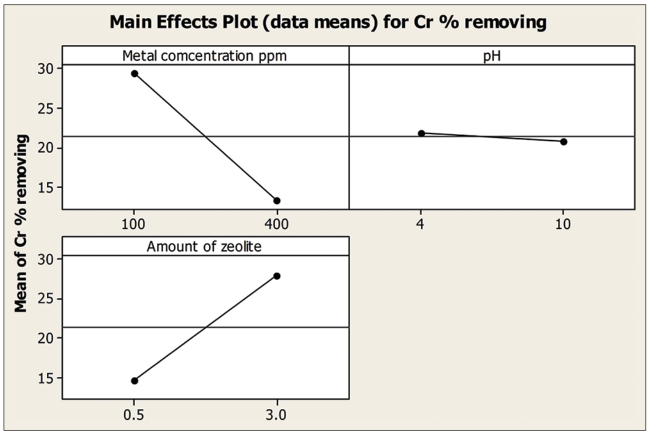

Figure 1: The main effect plot of the amount of zeolite, metal concentration ppm, and pH on the Cr ions percentage removal

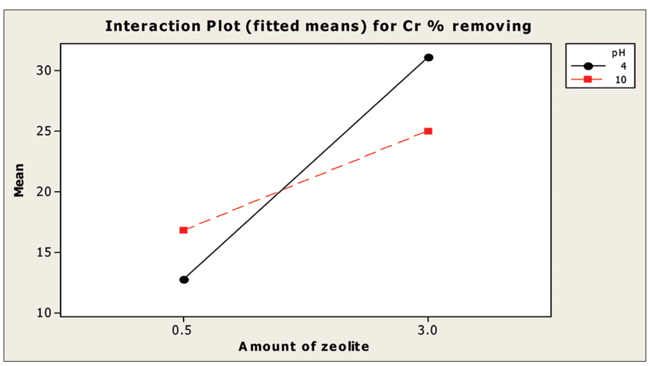

Figure 2: The interaction effect plot between the amount of zeolite and the pH

the mean removal value was higher at pH 4 than at pH 10. The percentage removal of Cr ions depends not only on the amount of zeolite but also on pH.As shown in Figure 3, there is no interaction effect between the amount of zeolite and the metal concentration. This is due to the parallelism between the lines representing the two factors.

Figure 4 shows that the same result applies to the relationship between the pH and metal concentration. It means the highest mean removal value was stable when evaluating the interaction effect between zeolite amount and metal concentration. The same conclusion is observed when evaluating the interaction effect between pH and metal concentration. Although the two lines that represent the amount of zeolite and the metal concentration in Figure 3 do not seem to be parallel, there is no intersection between the

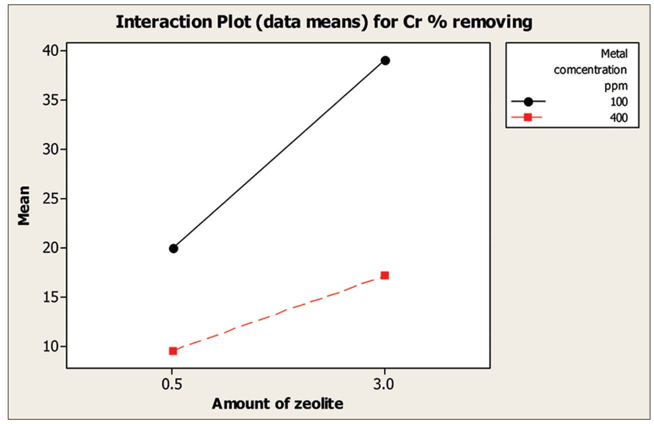

Figure 3: The interaction effect plot between the amount of zeolite and the metal concentration ppm

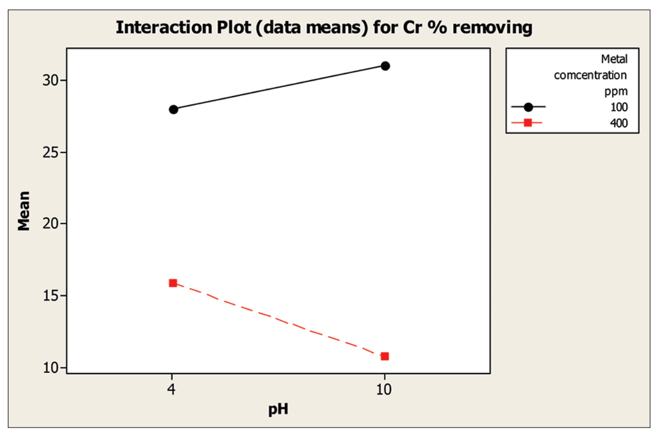

Figure 4: The interaction effect plot between the pH and the metal concentration ppm

two lines at the two levels adopted in this work. Accordingly, there is no interaction between the amount of zeolite and the metal concentration. The same conclusion regarding metal concentration and pH is shown in Figure 4.

Ni ions Removal

The percentage of Ni ions removed, based on the ICPE results, demonstrated the natural zeolite's capability to remove heavy metal ions from contaminated water. As shown in Table 5, the highest Ni% removal was 41%. This result corresponds to the run in the factorial design with a metal concentration of 100 ppm, a pH of 10, and a zeolite mass of 0.5 g. The result indicates that the highest removal percentage was obtained

Table 5: Ni% removing with factors and levels used in factorial design

Experiments	Metal concentration ppm	pH	Amount of zeolite g	Ni % removing
1	400	10	0.5	18.7
2	100	4	0.5	16
3	100	10	3.0	33
4	400	10	3.0	30
5	100	10	0.5	41
6	400	4	0.5	26.7
7	100	4	3.0	20
8	400	4	3.0	11.5

Ni: Nickel

at the lowest metal concentration and zeolite amount. Based on factorial design analyses, Cr (specifically Cr [VI]) and Ni (specifically Ni [II]) behave differently due to their chemical properties.[22,23] That is why the optimal removal was achieved at pH 4 with 3 g of zeolite for Cr ions, and at pH 10 with 0.5 g for Ni ions.

Main effects plot (Data Means) for Ni% removal

The main effect is the effect of a single independent variable (factor) on the dependent variable (response), while ignoring the effects of other independent variables. Figure 5 shows the plot of the main effects of the three factors, amount of zeolite, pH, and metal concentration, on the removal percent. As shown in Figure 5, the effect of the amount of zeolite on the removal percent remained nearly constant when going from a low level (0.5 g) to a higher level (3 g). This indicates a very small effect of changing the level of zeolite on the response. For Ni concentration, the removal percentage increased from a high level (400 ppm) to a low level (100 ppm). For pH, the removal percentage increased from a low pH of 4 to a higher pH of 10. This indicates a strong main effect of pH and metal concentration on the response.

Interaction effect of zeolite on Ni ions removal

In general, an interaction plot was used to show how the relationship between one factor and a continuous response depends on the value of the second factor. Interaction plots were generated and discussed to highlight the interaction effect observed in the removal of Ni ions when using natural zeolite. The more non-parallel the lines are, the stronger the interaction. The interaction plot of metal concentration versus pH [Figure 6] shows a significant interaction effect. This is due to non-parallel plot lines, meaning that the percentage of removal (y-axis) varies with pH at any metal concentration. It seems that the highest removal percentage occurs at a metal concentration of 100 ppm and a pH of 10. However, this conclusion changes when the highest mean removal value is reached at the lower pH of 4. This change in the mean removal value is due to the interaction effect between the two factors, although the mean removal value was higher at pH 4 than at pH 10. The percentage removal of Ni ions depends not only on the metal concentration but also on pH.As shown in Figure 7, there is no interaction effect between the amount of zeolite

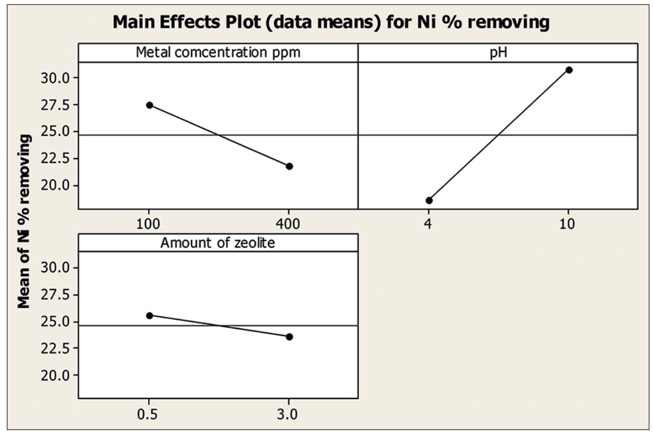

Figure 5: The main effect plot of the amount of zeolite, metal concentration ppm, and pH on the Ni ions percentage removal

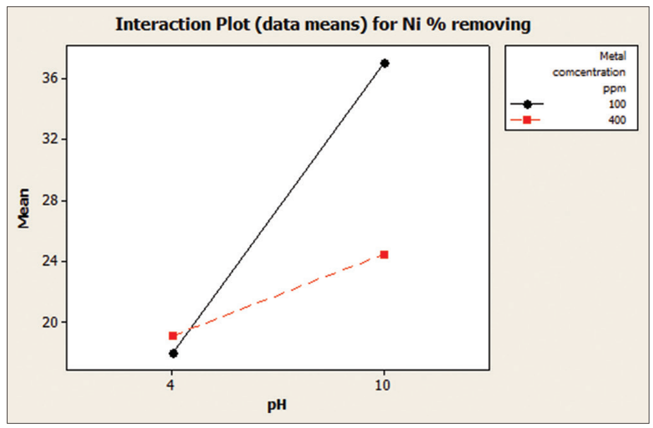

Figure 6: The interaction effect plot between metal concentration ppm and pH

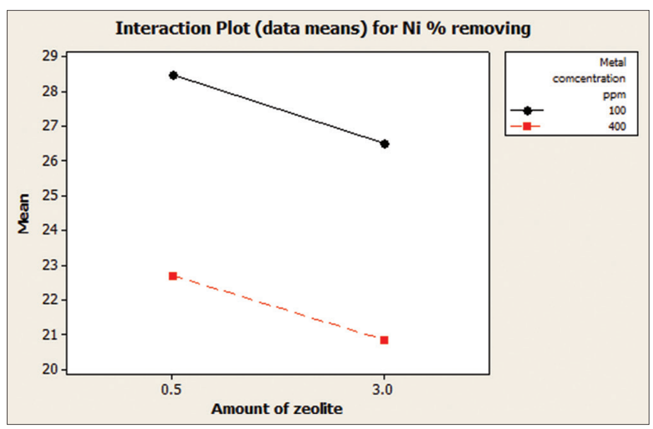

Figure 7: The interaction effect plot between the amount of zeolite and the metal concentration ppm

and the metal concentration. This is due to the parallel lines that represent the two factors. It means the highest mean removal value was stable when evaluating the interaction effect between zeolite amount and metal concentration.

Figure 8 shows that the same result applies to the relationship between pH and zeolite amount. The same conclusion is observed when evaluating the interaction effect

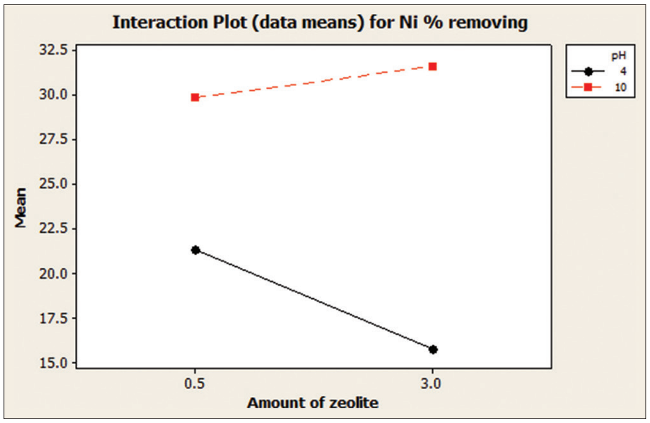

Figure 8: The interaction effect plot between the pH and the amount of zeolite

between pH and zeolite amount. Although the two lines that represent the amount of zeolite and the pH in Figure 8 do not seem to be parallel, there is no intercept between the two lines at the two levels adopted in this work. Accordingly, there is no interaction between the amount of zeolite and the metal concentration. The same conclusion regarding the amount of zeolite and pH is shown in Figure 8.

CONCLUSION

In this study, the research objectives were examined to provide a summary of the study's findings and to conduct a critical assessment. The investigation examines the use of natural zeolite as an inexpensive adsorbent for removing Cr and Ni from synthetic industrial metal waste solutions.

The results showed that zeolite successfully removed a significant amount of Cr and Ni ions from the prepared water samples. The results showed that the strongest effect was the zeolite amount in grams on Cr and Ni ions, followed by pH. The results also showed interaction effects between zeolite amount and pH for Cr removal, as well as between metal concentration and pH.

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