Optimization of Nickel Precipitation and Leaching Process from Simulated Industrial Waste: A Study on pH, Contact Time, and Sulfuric Acid Concentration

Nurul Zufarhana Zulkurnai, Umi Fazara Md Ali, Naimah Ibrahim, Mohd Irfan Hatim Mohamed Dzahir, Nor Ashikin Ahmad, and Fathiah Mohamed Zuki

Abstract The presence of nickel in industrial waste has emerged as a significant environmental concern, predominantly attributed to the plating industry. The significant objective of this study is to optimize the precipitation and leaching method to extract valuable nickel from the waste material. The high concentration of nickel found in the waste makes it a potentially valuable resource. To explore its potential extraction, the precipitation and leaching processes were optimized using a simulated Watts bath solution which is widely employed in the industry. This study focuses on examining the influence of pH, contact time, and sulfuric acid concentration on the

N. Z. Zulkurnai · U. F. M. Ali (✉) · M. I. H. M. Dzahir · N. A. Ahmad
Faculty of Chemical Engineering & Technology, Universiti Malaysia Perlis, 02600 Arau, Perlis, Malaysia
e-mail: umifazara@unimap.edu.my

M. I. H. M. Dzahir
e-mail: irfan@unimap.edu.my

N. A. Ahmad
e-mail: ashikinahmad@unimap.edu.my

U. F. M. Ali · M. I. H. M. Dzahir
Biomass Utilization Organization, Centre of Excellence (CoEBU), Universiti Malaysia Perlis, 02600 Arau, Perlis, Malaysia

N. Ibrahim
Faculty of Civil Engineering Technology, Universiti Malaysia Perlis, 02600 Arau, Perlis, Malaysia
e-mail: naimah@unimap.edu.my

F. M. Zuki
Center for Separation Science & Technology (CSST), Department of Chemical Engineering, Faculty of Engineering, Universiti Malaya, 50603 Kuala Lumpur, Malaysia
e-mail: fathiahmz@um.edu.my

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extraction of nickel concentration during the precipitation and leaching processes. To develop a prediction model for the process, three models were taken into consideration: quadratic, linear, and 2F1. The quadratic model exhibited the greatest adjusted R^2 value, suggesting a superior level of fit in comparison with the linear and 2F1 models. The predicted R^2 value of 0.8169 exhibits a satisfactory level of concordance with the adjusted R^2 value of 0.9737. The recommended optimal conditions proposed by response surface methodology (RSM) consisted of a pH value of 10.56, a contact time of 16.52 h, and a sulfuric acid concentration of 1.80 M in order to achieve a nickel concentration of 28,415 mg/L.

Keywords Optimization · Precipitation · Leaching · Nickel waste · Watts bath

1 Introduction

In the realm of industrial processes, there exists a notable focus on the extraction of valuable metals from waste materials, as it holds considerable potential for generating both economic and environmental advantages [1]. Among the several metals, nickel is well recognized as a highly coveted resource due to its extensive range of applications across multiple industries. Nevertheless, the inclusion of impurities in nickel industrial waste presents a significant obstacle in terms of recovery techniques and the resulting implications for both human health and the environment.

In the context of human health, nickel presents a notable hazard due to its capacity to elicit severe disease like cancer, lung fibrosis, cardiovascular disorders, and renal problems [2]. The inadequate disposal of nickel can result in severe environmental ramifications, leading to the contamination of water and soil [3, 4]. To avert these catastrophic incidents in Malaysia, the Department of Environment Malaysia (DOE) has implemented regulations to control the discharge concentration of nickel effluent into streams by set 1.0 mg/L as a discharge limit [5]. These measures aim to safeguard both the environment and the well-being of the population by mitigating the harmful effects of nickel contamination.

Plating industry has been identified as a major contributor to the nickel contamination found in industrial waste [6]. The predominant solution employed in this industry is the Watts bath solution. The composition of Watts bath solution is a combination of nickel sulfate, nickel chloride, and boric acid. Jaroslaw et al. [7] and Laokhen et al. [8] reported that the nickel waste contains a diverse range of contaminants, such as phosphorus, boric acid, lead, copper, zinc, and aluminum [7, 8]. One of the sustainable approaches to remove the contaminant is by precipitation process [9].

As a result, the precipitation and leaching processes have garnered attention as a viable solution for recovering this valuable metal. The study done by Oustadakis et al. [10] employed magnesium oxide (MgO) as a reagent for precipitation [10]. According to his research, more refined nickel hydroxide can be acquired through precise regulation of pH levels and temperature conditions, but it has slower hydroxyl ion release compared to NaOH [10]. The study conducted by Lee [11] utilized

$Ca(OH)_2$ reagent to investigate the effects of increasing precipitation reagent on pH levels [11].

This study aims to address the gap in knowledge regarding the optimization of the nickel precipitation and leaching process from simulated Watts bath solution within the given setting. To yet, there has been a lack of study focused on optimizing the utilization of Watts bath solution. The implementation of Watts bath solution simulation provides a controlled environment for the examination and evaluation of the effectiveness of different parameters in enhancing nickel precipitation. The objective of this study is to determine the ideal circumstances for achieving the maximum concentration of nickel by methodically changing pH, contact time, and sulfuric acid concentration.

Moreover, a comprehensive mathematical model is necessary to comprehend the correlation between the process parameters and the concentration of nickel. Therefore, this study aims to evaluate various models, such as the quadratic, linear, and 2F1 models, to ensure the precipitation and leaching processes most precise and dependable depiction. The chosen model will offer a predictive framework for the estimation of nickel concentrations under varying operational situations, hence enabling the enhancement of process control and optimization.

2 Material and Method

2.1 Chemical and Material

The Watts bath solution was prepared by combining nickel sulfate ($NiSO_4$), nickel chloride ($NiCl_2$), and boric acid (H_3BO_3). Sodium hydroxide (NaOH) was utilized as the chemical for the precipitation process, and sulfuric acid (H_2SO_4) served as the leaching reagent.

2.2 Preparation of Simulated Watts Bath

A 1-L (L) beaker was filled with 1 L of distilled water. To this, 153 g of $NiSO_4$, 128 g of $NiCl_2$, and 30.9 g of H_3BO_3 were added. Beaker containing the solution was placed on a hot plate, and the temperature was adjusted to 55 °C. A magnetic stirrer was used to continuously stir the solution until all the solid compounds were completely homogenized.

2.3 Precipitation Process

The optimization process involved manipulating three key factors which is pH, contact time, and leaching reagent concentration. To achieve this, a Box–Behnken design was employed, which enabled a systematic exploration of the parameter space. The optimization of the nickel precipitation process was initiated by performing experiments at varying pH levels ranging from 8 to 13. The contact time also varied between 6 and 24 h. After each precipitation run, the obtained precipitate was carefully collected and subsequently dried in an oven at 120 °C for a period of 6 h.

2.4 Leaching Process

The dried nickel precipitate underwent the leaching process after the precipitation process. For this purpose, H_2SO_4 was employed as the leaching reagent, and its concentration was varied between 0.5 and 2.5 M. The leaching process was conducted at room temperature, and the ratio of the nickel precipitate to H_2SO_4 was maintained at 1:10. To ensure efficient dissolution of the precipitate, the mixture was subjected to agitation at 300 rpm, preventing sedimentation of the precipitate at the bottom of the beaker. The nickel concentration of each run will be determined during the optimization process by using inductively coupled plasma (ICP) machine.

2.5 Optimization Precipitation and Leaching Process

The design of experiment (DOE) methodology encompasses a set of statistical and mathematical techniques that include the fitting of polynomial equations to empirical data. In this study, a Box–Behnken design (BBD) of response surface methodology (RSM) with three levels and three factors was utilized to identify the optimal combination of precipitation and leaching process variables for the extraction of nickel from simulated Watts bath. pH, contact time, and H_2SO_4 concentration were the independent variables selected to be in this experimental design, the nickel concentration as the response for the combination of independent variables. For pH, the range varied from 8 to 13. For the contact time, the range is between 6 and 24 h. Meanwhile, for the H_2SO_4, it varied between 0.5 M and 2.5 M.

The variable levels in Table 1 are coded using the orthogonal balanced block design (BBD) method, resulting in equal spacing between the low (− 1), middle (0), and high (+ 1) levels. The inclusion of replicates at the center coordinates (0, 0, 0) allows the model to assess both the experimental error and the reproducibility of the data. The chosen experimental design comprises twelve experimental runs for combined 22 factorial designs. In addition, incomplete block designs are utilized,

Table 1 Variables and their levels for the BBD

Variable	Level		
	− 1	0	1
pH	8	10.5	13
Contact time	6	15	24
Sulfuric acid concentration	0.5	1.5	2.5

with the inclusion of five duplicated center points. This results in a total of seventeen experimental runs, as depicted in Table 1.

2.6 Analytical Method

For the analysis of nickel concentration, 10 ml of each run of leaching solution was analyzed using the inductively coupled plasma mass spectrometry (ICP-OES) machine due to its robust and precise method.

3 Results and Discussion

3.1 Precipitation and Leaching Process

The treatment process optimization, focusing on pH, contact time, and leaching reagent concentration, was carried out using a Box–Behnken design. The results of the optimization runs are presented in Table 2. To achieve the desired pH for each run, the Watts bath solution was adjusted using NaOH. The solution was continuously stirred at 300 rpm using a magnetic stirrer to ensure the respective contact time was reached.

Following the treatment process, the precipitate was filtered using filter paper, as depicted in Fig. 1a. The image reveals the successful formation and separation of nickel (II) hydroxide ($Ni(OH)_2$) precipitate from the Watts bath solution through the reaction between the Watts bath solution and sodium hydroxide (NaOH) [12]. $Ni(OH)_2$ precipitate subsequently dried in an oven for 6 h at 120 °C aiming to eliminate any excess moisture. The precipitate of $Ni(OH)_2$, which has been dried, is depicted in Fig. 1b. The physical appearance of dried $Ni(OH)_2$ precipitate is green in color in a combination of solid flaky crystal and powdery textures. The dried $Ni(OH)_2$ precipitate then underwent the leaching process, as depicted in Fig. 1c. For the leaching process, H_2SO_4 was used as the leaching reagent. During this procedure, the solid $Ni(OH)_2$ has been effectively leached, resulting in the formation of a clear green solution of $NiSO_4$ [13].

Table 2 Optimized condition for electrowinning process

Run	Parameters		
	pH	Contact time (hr)	H_2SO_4 concentration (M)
1	8	24	1.5
2	10.5	24	2.5
3	13	24	1.5
4	10.5	15	1.5
5	8	15	2.5
6	8	6	1.5
7	10.5	24	0.5
8	10.5	15	1.5
9	13	15	0.5
10	10.5	6	2.5
11	8	15	0.5
12	10.5	6	0.5
13	13	6	1.5
14	10.5	15	1.5
15	10.5	15	1.5
16	10.5	15	1.5
17	13	15	2.5

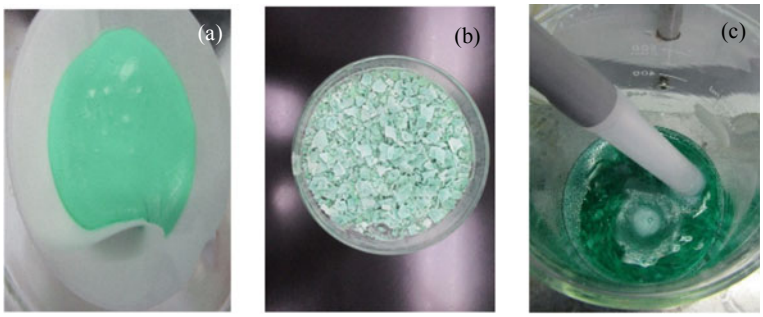

Fig. 1 Treatment process **a** filtration of precipitate, **b** dried precipitate, **c** leaching process

3.2 Optimization by Response Surface Methodology

The findings derived from the optimization of the treatment procedure, as depicted in Table 3, offer significant insights into the impact of pH, contact time, and H_2SO_4 concentration on the concentration of nickel in the treatment solution. Upon analysis of the data, significant patterns have been observed which provide confirmation of the influence that these parameters have on the process of nickel extraction.

Table 3 Result concentration of nickel for optimization process

Run	Parameters			Result
	pH	Contact time (hr)	H_2SO_4 concentration (M)	Concentration of nickel (mg/L)
1	8	24	1.5	28,986
2	10.5	24	2.5	28,385
3	13	24	1.5	28,997
4	10.5	15	1.5	27,829
5	8	15	2.5	27,275
6	8	6	1.5	24,416
7	10.5	24	0.5	28,149
8	10.5	15	1.5	27,825
9	13	15	0.5	26,627
10	10.5	6	2.5	25,140
11	8	15	0.5	26,271
12	10.5	6	0.5	21,809
13	13	6	1.5	24,648
14	10.5	15	1.5	27,906
15	10.5	15	1.5	27,839
16	10.5	15	1.5	27,853
17	13	15	2.5	26,993

First, when keeping the contact time and H_2SO_4 concentration constant, it is noticeable that pH 13 results in the largest concentration of nickel in the treatment solution, as opposed to a pH of 8. This phenomenon can be observed by doing a comparative analysis between Run 1 and Run 3, as well as Run 6 and Run 13. The results validate that elevated pH levels possess an increment capacity to produce $Ni(OH)_2$ species in the course of the precipitation process. At the high pH condition, more hydroxyl ions dissociate from the NaOH and lead to the increase of supersaturation of $Ni(OH)_2$ [14]. This finding is consistent with prior studies conducted by other researchers in 2010, thus reinforcing the importance of pH in optimizing the process of nickel extraction [15].

Similarly, when pH and contact time remain constant, the highest concentration of H_2SO_4 which in turn yields a treatment solution with the largest concentration of nickel derived from the dried precipitate. This can be observed by the comparisons made between Run 2 and Run 7, Run 5 and Run 11, and Run 9 and Run 17. The observed relationship between the concentration of H_2SO_4 and the concentration of nickel aligns with the conclusions drawn by Jin Young L. et al. in their study conducted in 2010 [15].

Furthermore, under conditions of constant pH and H_2SO_4 concentration, increasing the contact time during the precipitation process leads to an elevated concentration of nickel in the treatment solution. The observations can be made by

comparing Run 1 with Run 6, Run 2 with Run 10, Run 3 with Run 13, and Run 7 with Run 12. This finding shows that increasing the contact time increases the precipitation of hydroxide ions from NaOH and Ni^{2+} ions from the Watts bath solution, leading to the creation of $Ni(OH)_2$ [12].

Upon analysis of the optimization data, it becomes noticeable that Run 3 demonstrates the highest nickel content at 28,997 mg/L. This is closely followed by Run 1, Run 2, and Run 7, which exhibit concentrations of 28,986 mg/L, 28,385 mg/L, and 28,149 mg/L, respectively. Specifically, the four runs that exhibit the greatest nickel concentrations all possess a contact time of 24 h, suggesting that contact time has a substantial impact on the precipitation process.

The reason for Run 3 having the largest precipitation rate compared to the other four runs with the same contact time can be attributed to the fact that Run 3 was conducted at a pH of 13. This high pH level resulted in an increased concentration of hydroxyl ions, which in turn facilitated a greater precipitation of nickel from the solution [14]. The central point runs exhibit nickel concentrations ranging from 27,853 mg/L to 26,993 mg/L. The minor differences in nickel concentration among the central point runs suggest minimal errors in the optimization model.

Table 4 displays the summary data related to the fitness of the optimization model. The models were selected based on the highest-order polynomials that were not aliased, and additional terms were included if they were deemed to be statistically significant. Based on the data analysis, the quadratic model emerged as the suggested model due to its highest adjusted R^2 value compared to the linear and 2F1 models. Moreover, the quadratic model was found to be non-aliased, suggesting its suitability for the optimization process [16].

The quadratic model exhibits a strong correlation with the experimental data, as evidenced by the high adjusted R^2 value of 0.9737. This indicates a favorable match between the model and the observed data. The obtained predicted R^2 value of 0.8169, which is less than 0.2 in comparison with the adjusted R^2 value [16], provides additional evidence for the reliability of the quadratic model in its capacity to predict the nickel concentration during the optimization process.

These findings indicate that the quadratic model accurately represents the correlation between pH, contact time, and H_2SO_4 concentration, as well as their influence on the nickel concentration in the treatment solution. The model offers a significant tool for forecasting the best conditions required to attain the highest concentration of nickel.

Table 4 Fit summary data

Source	Sequential *p*-value	Lack of Fit *p*-value	Adjust R^2	Predicted R^2	
Liner	0.0001	< 0.0001	0.7440	0.6432	
2FI	0.4853	< 0.0001	0.7365	0.4806	
Quadratic	0.0002	< 0.0001	0.9737	0.8169	Suggested
Cubic	< 0.0001		0.9997		Aliased

The high adjusted R^2 value signifies that the quadratic model explains a significant proportion of the variability in the data, suggesting that the model is a valid depiction of the system under investigation. The non-aliased nature of the model further enhances its validity, by precisely capturing and assessing the impacts of the variables.

The significance and model fitness were analyzed using the analysis of variance (ANOVA). The impact of each individual variable and their interactions with one another on the response were also evaluated by this approach. Analysis of variance (ANOVA) was used to further evaluate the proposed model's dependability at a 5% significant level. Therefore, the *p*-value must be less than 0.05 in order to evaluate the model's significance [17].

The model's significance was confirmed by a model F-value of 66.77 and a *p*-value < 0.0001. The *p*-value for model *A*, *B*, and *C* are 0.7272, < 0.0001, and 0.0008, respectively (Table 5). This shows that the *B* and *C* models are significant. According to ANOVA table, the concentration of nickel is mostly affected by contact time and sulfuric acid concentration (*BC*). Similar to this, it was possible to infer that the B^2 and C^2 were important model terms for the response based on the *p*-values of the linear and quadratic terms.

Additionally, it was found that the independent variables' linear term effects on the answer were in the following order: ($B = 2313.00$) > ($C = 617.13$) > ($A = 39.63A$). This order shows contact time has the largest impact, followed by sulfuric acid concentration and pH. Equation 1 represents the final empirical model for the nickel concentration in terms of coded components.

Table 5 ANOVA (partial sum of squares—Type III) for the developed response surface model

Source	Sum of squares	df	Mean square	F-value	*p*-value	
Model	57,240,000	9	6,360,000	66.77	< 0.0001	Significant
A-pH	12,561.13	1	12,561.13	0.1319	0.7272	
B-contact time	42,800,000	1	42,800,000	449.38	< 0.0001	Significant
C-sulfuric acid concentration	3,047,000	1	3,047,000	31.99	0.0008	Significant
AB	12,210.25	1	12,210.25	0.1282	0.7309	
AC	101,800	1	101,800	1.07	0.3357	
BC	2,395,000	1	2,395,000	25.14	0.0015	Significant
A^2	29,674.12	1	29,674.12	0.3116	0.5941	
B^2	4,250,000	1	4,250,000	44.63	0.0003	Significant
C^2	4,002,000	1	4,002,000	42.02	0.0003	Significant
Residual	666,700	7	95,242.42			
Lack of fit	662,400	3	220,800	203.91	< 0.0001	Significant

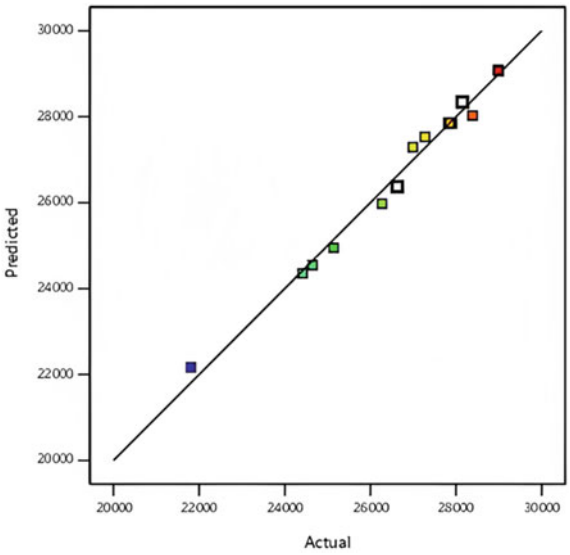

Fig. 2 Predicted versus actual value of nickel concentration (mg/L)

$$\begin{aligned}\text{Nickel Concentration} = {} & 27850.40 + 39.63A + 2313.00B \\ & + 617.31C - 55.25AB - 159.50AC - 773.75BC \\ & - 83.95A^2 - 1004.70B^2 - 974.95C^2 \end{aligned} \quad (1)$$

Figure 2 illustrates that the data points exhibit a uniform distribution and are closely matched with the linear trend. This finding suggests a strong correlation between the predicted response values and the actual observed values [18]. The close proximity of the data points to the regression line indicates that the constructed model effectively predicts the dependent variable by considering the independent variables of pH, contact time, and H_2SO_4 concentration. This alignment seen in this study demonstrates the high validity of the model for response value prediction.

Additionally, Fig. 3 shows the normal probability plot of residuals, which offers valuable insights into the distribution of the data points. The plots demonstrate that the residuals are closely aligned with the linear line, indicating a normal distribution of the data [19]. This implies that the observed data points are aligned with the assumptions of normality. A normal distribution of residuals suggests that the model is less sensitive to external noise or factors that could potentially induce deviations in the data. Therefore, the developed model is reliable for predicting the response variable.

Figures 4, 5 and 6 depict three-dimensional response surface plots that demonstrate the interaction effects of two optimization variables on the concentration of nickel. In these plots, the third variable is held constant at a zero level. These plots provide valuable insights into the impact of variation on nickel concentration during the treatment process.

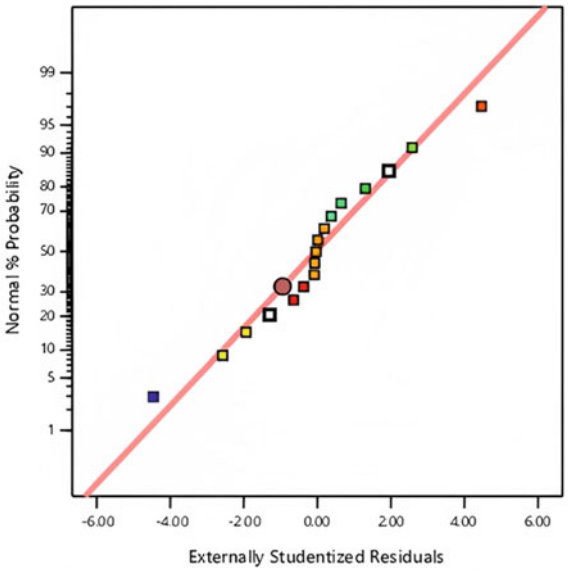

Fig. 3 Normal plot of residuals

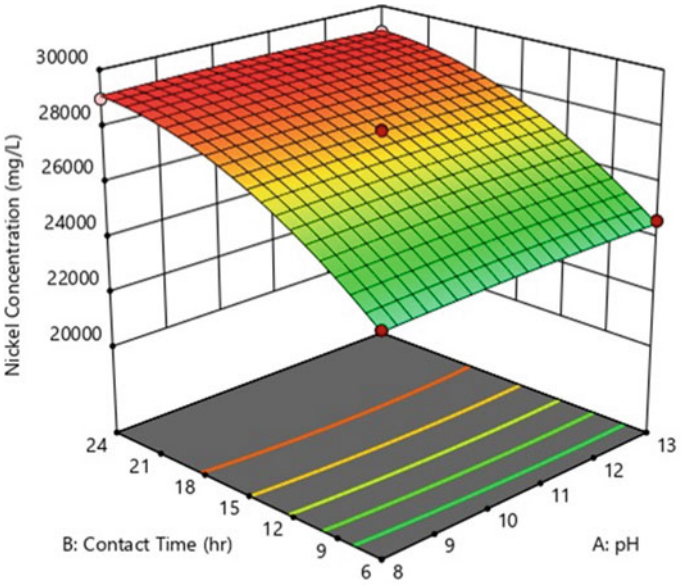

Fig. 4 Three-dimensional plot of nickel concentration with the combine effect of pH and contact time

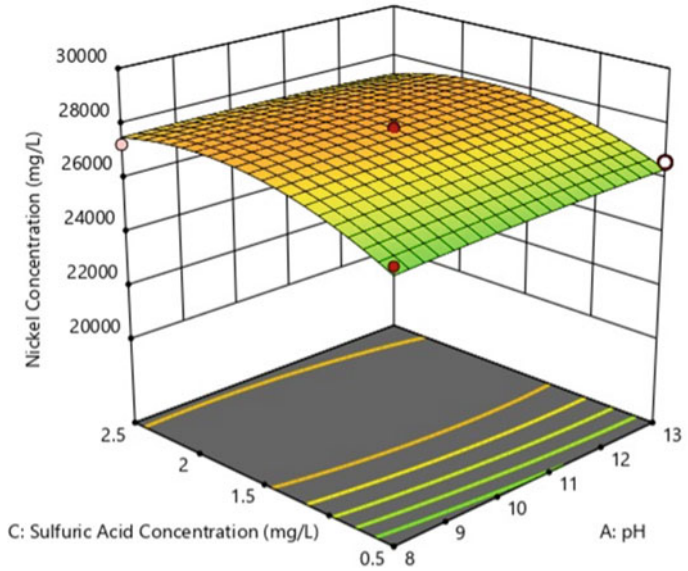

Fig. 5 Three-dimensional plot of nickel concentration with the combine effect pH and sulfuric acid concentration

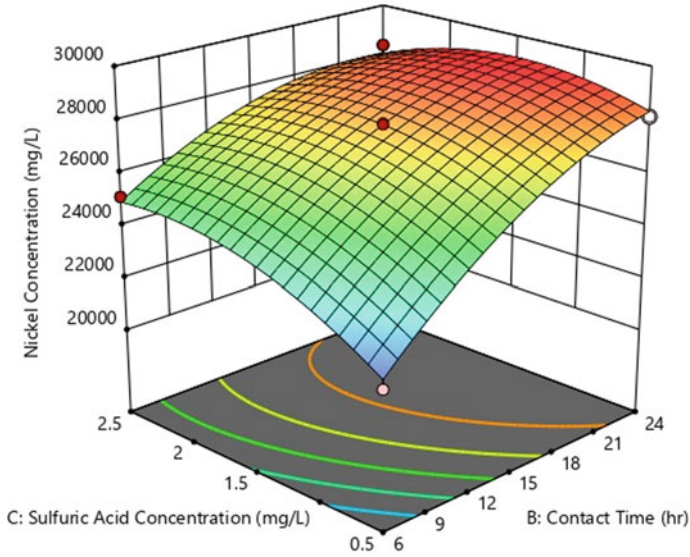

Fig. 6 Three-dimensional plot of nickel concentration with the combine effect contact time and sulfuric acid concentration

Figure 4 examines the impact of contact time and pH on the nickel concentration. The plot shows that increasing the contact time leads to higher nickel concentrations across all pH levels. This implies that a prolonged duration of contact time enhances the precipitation process, leading to higher yield extraction of nickel. The graph illustrates the significant of optimizing contact time in order to maximize the nickel concentration in the treatment solution.

In Fig. 5, it illustrates the impact of pH and sulfuric acid concentration on the nickel concentration. The data plot suggested that the optimal sulfuric acid concentration during the leaching process and pH during precipitation located near the midpoint values. This observation implies that extreme values of pH or sulfuric acid concentration may not lead to the highest nickel concentration. However, it should be noted that there exists an optimal range in which the process of nickel extraction demonstrates its highest level of effectiveness. These findings emphasize the significant of maintaining an optimum pH and sulfuric acid concentration to attain the desired nickel concentration in the treatment solution.

Figure 6 examines the impact of varying contact time and sulfuric acid concentration on the nickel concentration. The plot demonstrates that manipulating the contact time, while keeping the sulfuric acid concentration at an intermediate level, leads to elevated nickel concentrations. This suggests that a longer contact time, in combination with an optimal sulfuric acid concentration, enhances the efficiency of the leaching process and leads to an increased nickel concentration in the treatment solution. The findings of Kurama [20] and Zhanyoung et al. [21] have yielded similar outcomes [20, 21].

To evaluate the precision of the optimization results obtained through Box–Behnken design, three new batches of samples were prepared based on the suggested optimum conditions derived from the RSM software. The optimized conditions included a pH of 10.56, a contact time of 16.52 h, and an H_2SO_4 concentration of 1.80 M.

The nickel concentrations obtained from the three batches were recorded as follows: 28,405 mg/L for the first batch, 28,391 mg/L for the second batch, and 28,449 mg/L for the third batch. The overall average nickel concentration for the three replicate samples was determined to be 28,415 mg/L, as presented in Table 6.

Table 6 Experimental and predicted value of nickel concentration by using optimum preparation parameters

Replicate	Nickel concentration (mg/L)	
	Experimental	RSM predicted
1	28,405	28,270
2	28,391	
3	28,449	
Average	28,415	

By comparing the predicted nickel concentration from the RSM optimization with the experimental values obtained from the new samples, a slight difference can be observed. The predicted value from the optimization was 28,415 mg/L, which closely matches the average experimental value obtained from the three batches. This small discrepancy, with an error margin about 0.51%, suggests that the RSM optimization results are relatively accurate and reliable.

4 Conclusion

In conclusion, the optimization process of RSM by using Box–Behnken design effectively resulted in the determination of the optimal circumstances, which include a pH value of 10.56, a contact time of 16.52 h, and an H_2SO_4 concentration of 1.80 M. To validate the accuracy of these optimized results, three new sample batches were carefully run in strict accordance with the specified settings. The average concentration of nickel in these samples was determined to be 28,415 mg/L, exhibiting a minor deviation of 0.51% from the anticipated value.

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