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Modeling design parameters with Taguchi experimental method for obtaining operating conditions for Cu(II) removal through adsorption process

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ABSTRACT

One of the environmental concerns deals with the removal process of pollutants in water and wastewater. In this paper, a removal process was used to eliminate pollutants in water and wastewater. The aim of this paper was to obtain an optimum adsorption condition for the highest metal ions adsorbed (MIA) mg/g. For this particular purpose, a Taguchi L_{18} design was conducted. In addition, the signal-to-noise ratio was analyzed for optimum adsorption levels. The analysis of variance was also performed to evaluate the effect of each adsorption condition on MIA (mg/g) values. Moreover, an optimization model was also proposed to find the best optimal setting of adsorption levels. Then, the confirmation tests were performed using optimum coded levels of the adsorption parameters for the verification purpose. The results of the experimental study showed a good performance that Cu(II) removal capacity was found to be 43.66 mg/g. To the best of our knowledge, this research is the first to study natural stone as an efficient, inexpensive and cheap adsorbent for the removal process.

Keywords: Modeling; Taguchi design; Optimization; Adsorption

1. Introduction

Heavy metals are highly concerned for major environmental conditions recently. In practice, there are many techniques in the literature for the removal of the metals. However, these techniques may not be appropriate for a number of situations. For example, chemical precipitation technique is not appropriate when the pollutants are present in trace amounts. Similarly, ion exchange method is not inexpensive, easy and uncomplicated. Along the same lines, solvent extraction technique is expensive for less-concentrated solutions. Similarly, reverse osmosis method is a costly process when the membranes get so readily spoiled.

To overcome these limitations, adsorption technique is a preferred method for the removal process of toxic contamination from water. Particularly, Tran et al. [1] reported that the adsorption technique could be highly economical, effective, flexible, and uncomplicated. Moreover, Mohanty et al. [2] investigated that the adsorption technique could be applicable at low concentrations and appropriate for batch processes and little sludge production. Along the same lines, Bhattacharyya and Gupta [3] indicated that the processes for removal of the metals had become global importance to the minimization of the water contamination problem [3].

1.1. Review of the adsorption process for material removal

The adsorption process has been carried out in water and wastewater in order to eliminate toxic metals. In particular,

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Corapcioglu and Huang [4] investigated that the adsorption process could eliminate colors, odors, and organic pollutants in the wastewater and water from industries. Similarly, the process of liquid-phase adsorption is another effective technique. For example, El-Geundi [5] reported that granular and powdered activated carbon as widely applied adsorbents in the literature; however, these adsorbents could be often restricted due to high cost. El-Geundi [5] and Bailey et al. [6] looked for inexpensive adsorption substitutes, involving fly ash, lignite, natural materials, peat, and clays. In addition, Sharma and Forster [7,8], Gupta et al. [9], Park and Jung [10], and Selvi et al. [11] investigated natural inexpensive materials, such as activated carbon fibers, coconut waste, green algae, leaf mold, peat moss, rubber, and wood. Moreover, most studies have focused on searching for more effective and inexpensive adsorbents [12]. For example, Pehlivan and Arslan [13] reported high porosity, ion exchange capacity, and some adsorption abilities for many natural compounds. Additionally, Alslaibi et al. [14] investigated the efficiencies of Cu^{2+}, Cd^{2+}, Ni^{2+}, Pb^{2+}, Fe^{2+}, and Zn^{2+} from aqueous solution using olive stone activated carbon. Recent studies of Cu(II) heavy metal removal were conducted by Bohli and Ouedemi [15], Hu et al. [16], Mohammadifard and Amiri [17], and Kong et al. [18].

1.2. Review of Taguchi method

Taguchi method may provide an uncomplicated, effective, and systematic technique in order to analyze performance, quality, and cost for the experimental design [19]. Moreover, Bendell et al. [20] reported that the Taguchi technique had guided to a large number of industrial applications. In addition, the most significant phase in the design of an experiment is how to select control factors. Indeed, many control factors are involved in the design. Therefore, it is necessary to examine non-significant variables at the early stage. In the same vein, Ghani et al. [21] reported that an orthogonal array (OA) technique could adapt this requirement in the Taguchi method. For recent studies, Montgomery [22] provided comprehensive discussions of the design of experiments, including the Taguchi method.

1.3. Research motivation and scope

A Taguchi L_{18} design method was preferred as an effective and inexpensive technique because this design requires eighteen design runs for three coded levels of each parameter. Four adsorption parameters, pH, initial metal concentration (ppm), stirring rate (rpm), and contact time (min) were selected in order to conduct an experiment. On the contrary, other designs require more design runs comparing a Taguchi design. For example, a three-level full factorial design needs 80 design runs in order to conduct an experiment. The analysis of variance (ANOVA) was then investigated. Next, an optimum adsorption condition was found with the signal-to-noise ratio (SNR) concept. Furthermore, an optimization model was also proposed to find the best optimal setting of design parameters. Moreover, the confirmation tests with the optimum adsorption condition were presented to show the effectiveness of the Taguchi L_{18} design method for the experiment. To the best of our knowledge, this research paper is the first research study using natural stone as a potential adsorbent for heavy metal removal process.

2. Materials and methods

In this research study, the natural white Bayburt stone used as the adsorbent was supplied by Bayburt, Turkey. In addition, Table 1 shows the physical and chemical characteristics of the white Bayburt natural stone. X-ray diffraction (XRD) analysis of the sample was given in Fig. 1. Natural stone is a rock which has quartz, zeolitic mineral, such as clinoptilolite and heulandite, alkali feldspar, plajiyoklas, and smectite clay mineral. The sample was crushed and ground with a jaw crusher and ball mill, respectively. The distribution of the ground sample particle size was specified using the Malvern Mastersizer 3000, and its cumulative undersize values are given in Fig. 2. Notice that the d_{10}, d_{50} and d_{90} values size are 1.04, 4.88, and 14.42 μm, respectively.

The metal adsorption on the natural stone was investigated with the batch method. One, each solution was made up in the deionized water. Then, a metal stock solution with 1,000 ppm Cu(II) was prepared by dissolving $CuSO_4$. Each solution for pH level was set using HNO_3 and NaOH. For every instance, 1.0 g adsorbent (dry wt.) was included in 100 mL Cu solution at room temperature. Then, each solution was mechanically stirred. Next, the phase of aqueous was divided from the sample by filtration after the contact time. The Cu finding was performed with atomic absorption spectrophotometry at the beginning and after completion of adsorption. Microwave plasma–atomic emission spectrometer (MP–AES) model 4200 was used for determining the Cu^{2+} concentration. The system offers unique features to users: safety, higher sensitivity, lower detection limits and cost efficiency, and no combustible gases when compared with flame atomic absorption spectrometry [23]. Table 2 shows the operating conditions and performance of the MP–AES device. Before measuring the concentrations, the device was calibrated by the standard solutions of the study metals, and then the linear graphs were plotted.

Table 1
Chemical properties of natural stone

Oxides	(%)
SiO_2	71.50
Al_2O_3	11.70
Fe_2O_3	0.80
CaO	3.70
MgO	0.50
Na_2O	0.60
K_2O	2.70
P_2O_5	<0.10
TiO_2	0.10
MnO	0.10
LOI	8.25

LOI: loss of ignition.

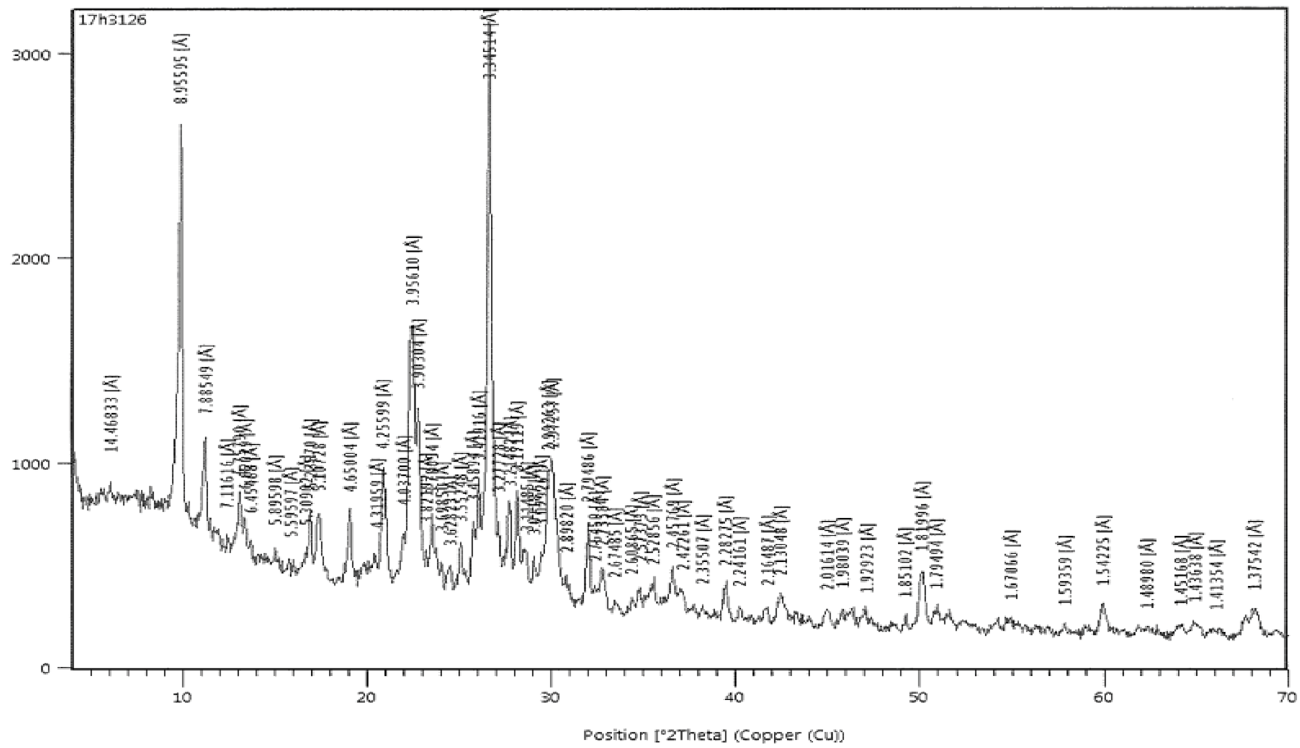

Fig. 1. XRD analysis of natural stone.

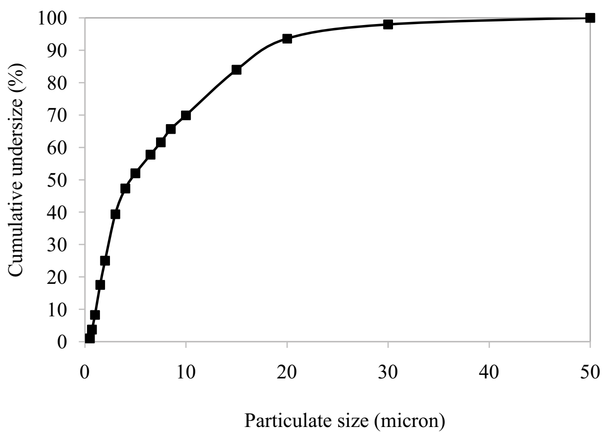

Fig. 2. Cumulative undersize values of the ground sample.

Table 2
Operating conditions of the MP–AES device and the system analytical characteristics

	Cu
Wavelength, nm	324.754
Nebulizer pressure, kPa	240
Sampling and stability time, s	10
Peristaltic pump speed, rpm	15
LOD, µg/L	1.0
LOQ, µg/L	3.3
RSD, %	0.65
Working range, mg/L	0.1–10.0

LOD: limit of detection; LOQ: limit of quantification; RSD: relative standard deviation.

The number of metal ions adsorbed (MIA), which is metal ions/sample (mg/g), was calculated as follows:

$$\text{MIA} = \frac{\left(C_0 - C\right)V}{1{,}000\ m} \quad (1)$$

where C_0 and C represent the concentration of the metal ions before and after the incubation period in the aqueous phase, respectively (ppm). In addition, V and m denote the aqueous phase volume (mL) and natural stone amount (g), respectively.

2.1. Implementation of Taguchi method for this research study

In this paper, the optimization of design parameters is an important phase to obtain the highest metal ions. In addition, Montgomery [22] reported that the classical parameter designs were not easy to use. For example, one of the most used methods is a full factorial experimental design. This design has 2^f combinations that should be checked where f represents the number of design parameters at two coded levels. Furthermore, it may be costly and time-consuming when considering a large number of design factors. Finally, the Taguchi method is the most appropriate experimental technique chosen in this study.

In this paper, the following major phases, which were systematically modified from Chen et al. [24], were used for the optimization step:

- Define main factors
- Define the coded levels of each design parameter
- Choose a suitable OA technique
- Run an adsorption experiment
- Examine the data and obtain optimum adsorption levels
- Perform a confirmation test

2.2. Implementation of SNR for this research study

Taguchi [19] proposed the SNR concept to analyze an outcome of experimental design for the specified quality characteristic. Along the same lines, Phadke [25] and Park [26] reported that the SNR concept could be an effective technique to analyze the results of experimental studies. For the SNR concept, Taguchi empirically proposed the two stages for the optimization procedure involving SNRs. The idea is that the process variance is minimum when the process mean is at the target value. In the literature, the SNR concept is useful to enhance the process quality and reduce the process variance for many real-life applications. In this research study, the Taguchi experimental design method was selected to obtain an optimum adsorption condition for the maximum MIA value. The larger the better (*L*-type) SNR quality characteristic was used for achieving the highest MIA value. The *L*-type SNR was denoted as follows:

$$L\text{-type SNR} = -10\log\frac{1}{n}\left(\sum\frac{1}{y^2}\right) \tag{2}$$

where y and n represent the collected data and the number of runs for the experiment.

Four adsorption parameters, which are (1) pH, (2) initial metal concentration, (3) stirring rate, and (4) contact time, were selected as controllable design parameters. Table 3 shows all design parameters with their coded levels. In addition, Table 4 shows an L_{18} OA with 18 experimental runs.

2.3. Proposed optimization method for obtaining optimal operating conditions

A linear regression model for a Taguchi method is given as follows:

$$E(y) = \beta_0 + \sum_{j=1}^{n}\sum_{i=1}^{m} x_{ij} f_x(\beta) \tag{3}$$

Table 4
Experimental design applying L_{18} orthogonal array

	Process parameter level			
Experiment number	A pH	B Initial metal concentration	C Stirring rate	D Contact time
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	1	3
5	2	2	2	1
6	2	3	3	2
7	3	1	2	3
8	3	2	3	1
9	3	3	1	2
10	1	1	3	1
11	1	2	1	2
12	1	3	2	3
13	2	1	2	2
14	2	2	3	3
15	2	3	1	1
16	3	1	3	2
17	3	2	1	3
18	3	3	2	1

where x_{ij} represents the ith design factor for the jth level, β_0 is an intercept, $f_x(\beta) = \beta_{ij}$ and $x_{ij} \in \{0,1\}$. In addition, β denotes a vector of regression coefficients. A proposed model is based on Eq. (3) and it is denoted as follows:

$$\begin{aligned} &\text{maximize } E(y) \\ &\text{s.t. } \sum_{i=1}^{m} x_{ij} = 1 \ (j = 1,\ 2,\ \ldots,\ n) \\ &x_{ij} \in \{0,1\} \end{aligned} \tag{4}$$

The objective of the proposed model is to maximize the regression function in Eq. (4) due to the *L*-type quality characteristic. To prevent meaningless results, constraints, $\sum_{i=1}^{m} x_{ij} = 1 \ (j = 1,\ 2,\ \ldots,\ n)$, are also included. This model in Eq. (4) is capable of obtaining optimal operating conditions for an adsorption process. In addition, this proposed model was compared with the results of the traditional

Table 3
Factors and their levels conducted in the experiment

Factor	Code	Level 1	Level 2	Level 3
pH	A	4	7	9
Initial metal concentration, ppm	B	150	300	450
Stirring rate, rpm	C	500	1,000	1,500
Contact time, min	D	30	60	90

SNR concept. It is believed that the proposed model may be useful for some cases and it is one of the contributions of this paper.

3. Results and discussion

The *L*-type quality characteristic for the MIA (mg/g) value was adopted for obtaining the optimum operating condition of the adsorption parameters. The aim of the adsorption experiment was to obtain optimum adsorption parameters and to find the highest MIA (mg/g). Table 5 shows the experimental results of the MIA (mg/g) values and the related SNR using Eq. (2). Because of the orthogonal experimental design, it is to divide the effect of each adsorption parameter at different coded levels. Particularly, the mean SNR for the MIA (mg/g) values at coded levels 1, 2, and 3 may be computed with averaging of the SNRs, such as for pH = 5, 1–3 and 10–12, respectively. The mean SNR for every coded level of each adsorption parameter is calculated in a similar way. Table 6 presents the mean SNR for every factor level of the adsorption parameters and the calculated total mean SNR for the 18 trials. Fig. 3 shows the plotted mean SNR for every coded level of the adsorption parameters and the SNR graph for the coded levels of each adsorption parameter. In addition, Fig. 3 suggests that the pH (factor A) and initial metal concentration (factor B) are more significant, succeeded by the interaction between the parameters (interaction A × B) on average SNR response for the metal ion adsorption. The stirring rate and contact time (factors C, D) are not as important as the slope is very small. From the SNR analysis, the optimal adsorption conditions are 9 pH (level 3), 450 ppm initial metal concentration (level 3), 1,500 rpm stirring rate (level 3), and 30 min contact time.

Table 5
Experimental results for MIA (mg/g) and SNR (dB)

Run	A	B	C	D	Actual value MIA (mg/g)	SNR (dB)	Residual for MIA (mg/g)	Residual for SNR
1	5	150	500	30	6.95	16.84	3.33	–0.06
2	5	300	1,000	60	8.60	18.69	–1.07	–0.96
3	5	450	1,500	90	17.15	24.69	–1.30	0.81
4	7	150	500	90	14.95	23.49	0.27	–0.11
5	7	300	1,000	30	29.98	29.54	1.43	1.00
6	7	450	1,500	60	44.55	32.98	5.59	0.83
7	9	150	1,000	90	14.99	23.52	0.12	0.14
8	9	300	1,500	30	29.99	29.54	–4.95	–1.55
9	9	450	500	60	44.99	33.06	4.85	1.41
10	5	150	1,500	30	9.70	19.74	4.67	1.73
11	5	300	500	60	11.10	20.91	–0.97	0.41
12	5	450	1,000	90	10.00	20.00	–4.65	–1.93
13	7	150	1,000	60	14.96	23.50	–1.01	0.42
14	7	300	1,500	90	29.97	29.53	3.38	0.30
15	7	450	500	30	29.96	29.53	–9.66	–2.44
16	9	150	1,500	60	14.99	23.52	–7.38	–2.12
17	9	300	500	90	29.96	29.53	2.18	0.79
18	9	450	1,000	30	44.99	33.06	5.18	1.32

MIA: metal ions adsorbed; SNR: signal-to-noise ratio.

Table 6
Average SNR for adsorption factors

Symbol	Factor	SNR (dB)		
		Level 1	Level 2	Level 3
A	pH	20.15	28.10	28.71[a]
B	Initial metal concentration (ppm)	21.77	26.29	28.89[a]
C	Stirring rate (rpm)	25.56	24.72	26.67[a]
D	Contact time (min)	26.38[a]	25.44	25.13

[a]Optimum level.
Total mean SNR (signal-to-noise ratio): 25.65 (dB).

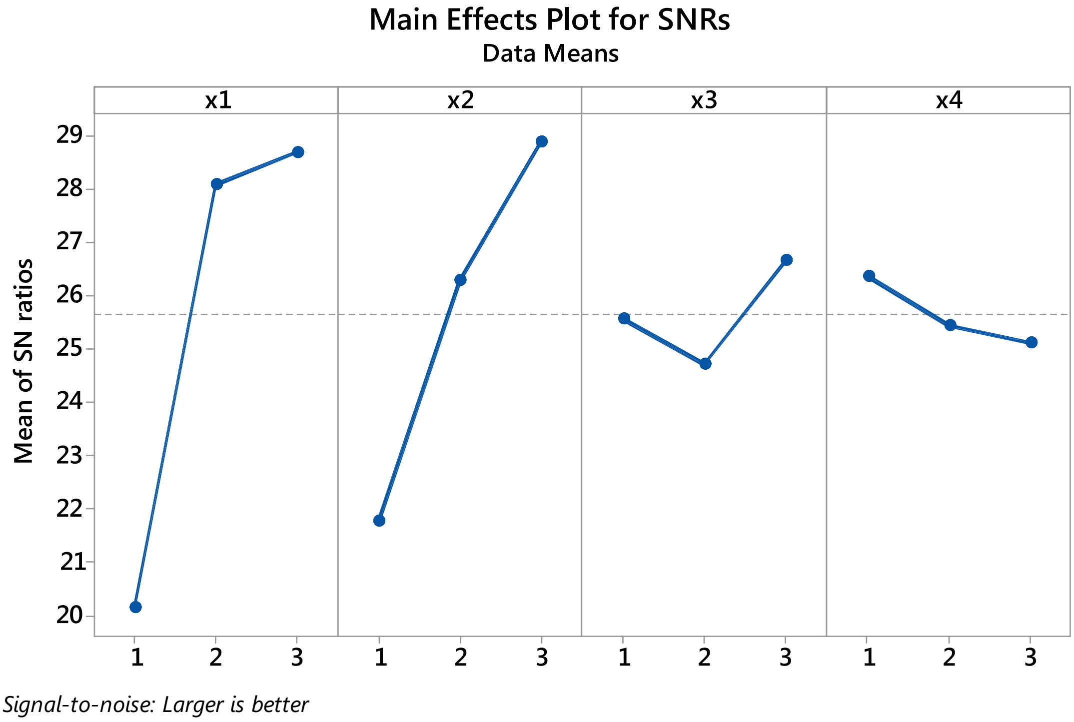

Fig. 3. Higher-the-better SNR graph for MIA.

The regression function was obtained using Minitab software as follows:

$$E(y) = 22.65 - 12.07x_{11} + 4.74x_{12} + 7.33x_{13} - 9.90x_{21} + 0.61x_{22} + 9.29x_{23} + 0.33x_{31} - 2.07x_{32} + 1.74x_{33} + 2.61x_{41} + 0.54x_{42} - 3.15x_{43} \quad (5)$$

In addition, the proposed model was denoted below.

$$\begin{aligned} \text{maximize } & 22.65 - 12.07x_{11} + 4.74x_{12} + 7.33x_{13} - 9.90x_{21} + 0.61x_{22} \\ & + 9.29x_{23} + 0.33x_{31} - 2.07x_{32} + 1.74x_{33} + 2.61x_{41} + 0.54x_{42} \\ & - 3.15x_{43} \\ \text{subject to } & x_{11} + x_{12} + x_{13} = 1 \\ & x_{21} + x_{22} + x_{23} = 1 \\ & x_{31} + x_{32} + x_{33} = 1 \\ & x_{41} + x_{42} + x_{43} = 1 \\ & x_{11}, x_{12}, x_{13}, x_{21}, x_{22}, x_{23}, x_{31}, x_{32}, x_{33}, x_{41}, x_{42}, x_{43} \in \{0,1\} \end{aligned} \quad (6)$$

The results of the model in Eq. (6) are $x_{13} = x_{23} = x_{33} = x_{41} = 1$ and $x_{11} = x_{12} = x_{21} = x_{22} = x_{31} = x_{32} = x_{42} = x_{43} = 0$. So, the optimal adsorption conditions are found at 9 pH (level 3), 450 ppm initial metal concentration (level 3), 1,500 rpm stirring rate (level 3), and 30 min contact time. In addition, the objective function is also found at 43.62 mg/g. The results of the model in Eq. (6) are consistent with the SNR concept.

3.1. ANOVA for the adsorption experiment

The ANOVA method is an effective statistical tool to analyze which design parameters importantly impact the quality characteristic. For this particular purpose, the total sum of the squared deviations (SS_T) is expressed as follows:

$$SS_T = \sum_{j=1}^{p} \left(\eta_j - \eta_m \right)^2 \quad (7)$$

where p, η_j and η_m represent the number of experiments, the mean SNR for the jth experiment and the total mean of the multi-response SNR, respectively [27].

In addition, the F-test investigates significant effects for design parameters. The mean of the squared deviations (SS_m) is calculated in performing the F-test due to each parameter. SS_m is found as the sum of the squared deviations (SS_d) separated by the number of degrees of freedom related to the design parameter [28]. Next, the F-value is:

$$\text{F - value} = \frac{SS_m}{SS_e} \quad (8)$$

Because of the design parameter, the big F-value is a great impact on the quality characteristic [29]. It is also concluded that the replacement of the design parameter has important effects on the desired quality characteristic when F-value > 4 [30].

Table 7 shows the ANOVA results. The F-ratios were found for a 95% confidence level. The table also shows that the change in the value of two parameters, which are the pH and initial metal concentration (ppm), within the range investigated in this research study, affect the MIA (mg/g) and thereby the MIA value significantly since the F-ratios are higher than 4. In addition, the F-ratios of other adsorption parameters (the stirring rate and contact time) are less than 4. Therefore, the two parameters are not effective in the adsorption process. Table 7 also shows the contribution of parameters to the quality characteristic. The contribution of

Table 7
ANOVA result for SNR

Parameters	Degree of freedom	Sum of square	Variance	F	Contribution (%)
A	2	273.68	136.84	9.91	57.78
B	2	155.77	77.89	5.64	32.89
C	2	11.48	5.74	0.42	2.42
D	2	5.08	2.54	0.21	1.07
Error	9	27.62	13.81	–	5.84
Total	17	473.63	–	–	100

Table 8
Confirmation experiment results

	Optimal process parameters		
	Prediction[a]	Prediction[b]	Experiment
Level	A3B3C1D1	A3B3C3D1	A3B3C1D1
MIA (mg/g)	43.48	43.62	43.66
SNR (dB)	32.77	32.79	32.80

[a]SNR (signal-to-noise ratio) concept.
[b]Proposed optimization model.
MIA: Metal ions adsorbed.

other parameters in descending order is as follows: stirring rate (2.42%) and the contact time (1.07%). Thus, the optimal design parameters and their levels for the maximum SNR is the pH at level 3 (pH = 9), the initial metal concentration at level 3 (450 ppm), the stirred rate at level 1 (500 rpm), and the contact time at level 1 (30 min) based on the main effects and the results of the ANOVA analyses.

3.2. Confirmation tests for the experiment

The final phase is the confirmation experiment, which validates the drawn conclusions during the analysis step. In addition, the confirmation experiment is also carried out by performing a test with an optimum operating condition of each design parameter. The last phase is to estimate and verify the results of the optimization phase. For this particular purpose, the predicted η with the optimum coded levels of the design parameters could be computed as follows:

$$\eta = \eta_m + \sum_{i=1}^{k}\left(\eta_i - \eta_m\right) \quad (9)$$

where η_m and η_i are the total mean of SNR and the mean of SNR at the optimum level, respectively. In addition, k is the number of main adsorption parameters that importantly influence the process performance [31].

As shown in Fig. 3, the pH 9, 450 ppm initial metal concentration, 1,500 rpm stirring rate, and 30 min stirring time gave the best results for SNR. The same optimal operating condition was found using the proposed model. It was determined that C (the stirring rate) and D (the contact time) parameters were not effective after the F-test. The test results supported this case because the A3B3C1D2 (pH = 9, initial metal concentration = 450 ppm, stirring time = 500 rpm and contact time = 60 min) and A3B3C2D1 (pH = 9, initial metal concentration = 450 ppm, stirring time = 1,000 rpm and contact time = 30 min) gave the same result for SNR as 32.80 dB. This result clearly showed that the C and D parameters were not effective in the adsorption parameters for the adsorption study. This situation confirmed the F-test results. This experimental study showed that the optimum adsorption parameters were: A3B3C1D1, that is, pH = 9, initial metal concentration = 450 ppm, stirring rate = 500 rpm, and stirring time = 30 min. The confirmation test was needed for this particular case because the optimal operating condition of the design parameters and their levels (i.e., A3B3C1D1) did not account for any experiment of the OA. After finding the optimal operating conditions and predicting the response for this condition, a new design of the experiment was conducted using the optimal coded levels of the adsorption parameters.

The estimated SNR with the optimum adsorption parameters (A3B3C1D1) for MIA (mg/g) could be found and the related SNR (dB) was also computed by using Eq. (2). In addition, Table 8 shows the confirmation experiment results with the optimum adsorption parameters. The prediction value was obtained by using Eq. (9) for MIA and SNR as 43.48 mg/g and 32.77 (dB) based on the confirmation test results. The results showed that the MIA values were smaller than the experiment result.

Notice that the optimal adsorption conditions showed the improved adsorption efficiency because the negative charge density on the surface of natural stone as an adsorbent increased the pH level. This claim was verified from the current literature and it was also observed that the pH level increased because of deprotonation of metal binding sites [32].

The results of the study by Kong et al. [18] showed that alkali-leaching residual wire sludge calcinated at 700°C exhibited the maximum Cu^{2+} removal capacity, which was 36.48 mg/g. For this study, the removal of Cu(II) heavy metal ions from an aqueous solution was found to be 43.66 mg/g. Therefore, the natural stone showed promising applications in the removal of Cu(II) heavy metal ions from an aqueous solution given good removal performance.

4. Conclusion

In this study, the adsorption parameters were analyzed using the Taguchi method for the MIA (mg/g) that occurred in the turning of white Bayburt natural stone material. For this purpose, four different adsorption parameters, (1) pH, (2) initial metal concentration (ppm), (3) stirring rate (rpm), and (4) contact time (min), were selected. The Taguchi method generated an uncomplicated, systematic, and effective technique for obtaining optimum adsorption parameters. In addition, the design parameters of the turning operations, such as pH and initial metal concentration were enhanced together with the proposed method. The A3B3C1D1 coded experiment, which is pH = 9, 450 ppm initial metal concentration, 500 rpm stirred rate and 30 min contact time, was determined the optimal adsorption conditions. The results that were obtained for the optimal operating conditions indicated that 1 g of the sample adsorbed 43.66 mg of the

Cu(II) ions. Similarly, the optimal operating conditions are obtained as 9 pH (level 3), 450 ppm initial metal concentration (level 3), 1,500 rpm stirring rate (level 3), and 30 min contact time. Additionally, the optimal result of the proposed model showed 43.62 mg/g. Finally, the results of the proposed model are consistent with the verification study.

Conflict of interest

The authors declare that they have no conflict of interest.

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