

Modelling and Optimisation of Copper Adsorption in Solution by the Response Surface Method

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Abstract

Copper is considered a heavy metal that can be toxic at certain concentrations and its presence in water is a potential threat to public health. These heavy metals also contribute to a remarkable degradation of the environment, hence the need for effective treatment methods to remove them. In this study, a mixture of titaniferous sand and calcium silicate was used as adsorbent material to eliminate copper in solution. The calcium silicate was synthesised from fluosilicic acid, which is a by-product of phosphoric acid manufacture. The titaniferous sand is a residue from a mining industry. Both adsorbents were characterised by infrared spectroscopy and X-ray fluorescence to determine their compositions and physicochemical properties. The response surfaces, through the Box-Behnken model, were used to model and optimise various adsorption parameters, namely initial copper concentration (A: 60 - 200 mg/L), adsorbent dose (B: 0.1 - 0.6 g) and pH (C: 4 - 10). The copper removal efficiency (98.92%), after statistical analysis, was obtained under the following optimal conditions: an adsorbent dose of 0.55 g, an initial copper concentration of 197.25 mg/L and a pH of 9.85. The study of the effects of the operating parameters showed that they had a positive effect on the copper removal efficiency.

Keywords

Adsorption, Calcium Silicate, Copper, Response Surface, Titaniferous Sand

1. Introduction

The world is nowadays increasingly impacted by environmental pollutions such as chemical, physical or biological, thus affecting the physiology and behaviour

of exposed organisms [1] [2]. These pollutions generally come from energy use and production, industrial activities and agriculture. However, the pollution from industrial activities is the most dangerous and best known [3] [4].

Indeed, industrial effluents usually contain significant amounts of chemical pollutants which can be organic such as petroleum hydrocarbons or inorganic such as heavy metals [5] [6]. Moreover, when the concentration of heavy metals in certain effluents is not high, these metals have the particularity of being able to accumulate in soils or water reserves until they reach toxic thresholds [7]. Their release into the environment would cause serious health problems for humans, alter the living conditions of aquatic and terrestrial animals and disturb the ecosystem [8] [9].

Copper is an essential trace element for micro-organisms, animals and plants. However, it is one of those metals that when accumulated in the human body can lead to serious health problems such as cancers, liver damage and kidney disease [10] [11].

It is then necessary to proceed with its elimination and, failing that, its reduction to limit values before discharging any effluent containing it into the environment [12] [13]. Several methods of eliminating these metals are used, including precipitation, adsorption, ion exchange and membrane filtration [14] [15] [16] [17]. Compared to other treatment systems, adsorption is an inexpensive, accessible and feasible method for the decontamination of water containing copper at very high concentrations. The effectiveness and simplicity of this method for copper removal has been proven by some researchers such as Maruf Mortula *et al.* in the study of Copper Removal via Adsorption [18], Muhammad Shahrain Shuhaimen *et al.* in the adsorption study of removal of copper ions from aqueous solution using modified sodium hydroxide: carica papaya peels [19], Victor Uzoma Nwagbara *et al.* in evaluating the potential effectiveness of *Moringa oleifera* seed biomass as an adsorbent for copper removal from water [20] and P. Hanzlík *et al.* in the adsorption of copper, cadmium and silver from aqueous solutions on natural carbon materials [21].

In the context of waste recovery, titaniferous sand combined with hydrated calcium silicate formulated from an industrial effluent, fluosilicic acid, will be used for copper removal.

The study of the effects of different parameters such as the adsorbent dose, the concentration of copper and the pH as well as their interactions will be carried out through a Box-Behnken design.

2. Materials and Methods

2.1. Adsorbat

Copper occurs naturally in the earth's crust, rivers, oceans and lakes in various concentrations and forms. A copper stock solution with a concentration of 200 ppm is prepared from copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and the other daughter solutions are obtained by making successive dilutions from the

stock solution.

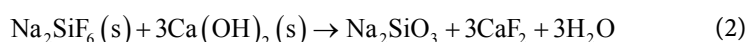
2.2. Preparation of Adsorbents

2.2.1. The Titaniferous Sand

The titaniferous sand is a residue from a mining industry in Senegal. The sand was treated by placing it in contact with a sulphuric acid solution of $4 \text{ mol}\cdot\text{L}^{-1}$. The mixture was stirred for 4 hours, and at the end of the operation, the sand was recovered and washed several times with distilled water until a pH close to neutrality was obtained. It is then dried in an oven for 24 hours and placed in flasks for later use.

2.2.2. Calcium Silicate

The synthesis of calcium silicate ($\text{CaSiO}_3(\text{s})$) involves several reaction steps. The first step consists of reacting fluosilicic acid with sodium chloride (**Figure 1**) for 15 minutes under standard temperature and pressure conditions to give sodium fluosilicate (Na_2SiF_6) and 18% hydrochloric acid (HCl), according to reaction 1. The sodium fluosilicate (Na_2SiF_6), recovered from reaction 1, after filtration, oven drying at 110°C and grinding, is causticized (**Figure 2**) with slaked lime for 150 minutes at 50°C according to reaction 2. The sodium silicate (Na_2SiO_3), which constitutes the aqueous phase of reaction 2, after filtration under vacuum, is subjected to further causticization (**Figure 3**) to obtain soda ash and calcium silicate, according to reaction 3, which lasts 150 minutes at 50°C .



2.3. Experimental Procedure

For each test, 50 mL of a copper solution of given concentration and initial pH was added to 250 mL reactors. The adsorbent mixture consisting of titanium sand and calcium silicate was added to the reaction medium and the reactors were stirred at 120 rpm and at room temperature ($25^\circ\text{C} \pm 2^\circ\text{C}$) using a "MULTISTIRRER" type multi-stage stirrer. After a certain period of time, the samples were taken, filtered through a $0.45 \mu\text{m}$ HA Millipore membrane and then subjected to atomic adsorption spectrometry analysis to determine the residual copper concentration. The percentage removal of copper was calculated using Equation (4).

$$\text{Yield}(\%) = \frac{(C_i - C_f) * 100}{C_0} \quad (4)$$

where:

C_i is the initial concentration (mg/L);

C_f is the final concentration (mg/L);

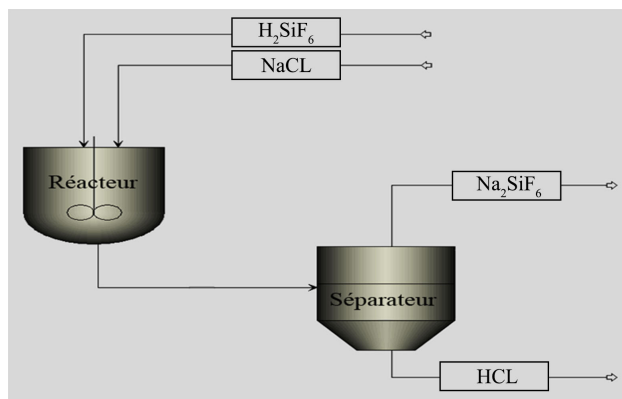


Figure 1. Simplified diagram of the process for obtaining sodium fluosilicate.

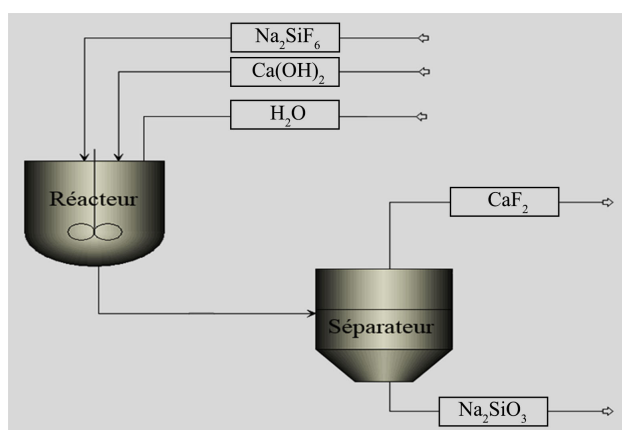


Figure 2. Simplified diagram of the process for obtaining sodium silicate.

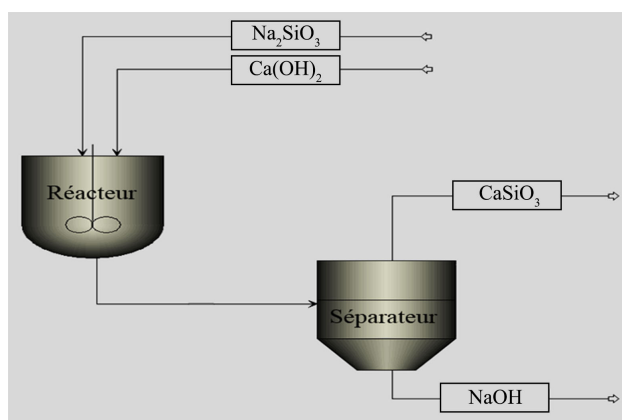


Figure 3. Simplified diagram of the process for obtaining calcium silicate.

2.4. Mathematical Procedure

The experiments were carried out according to the statistical Box-Bhnken Design (BBD) with five central points and involving 17 trials. The response surface methodology was used to study and optimise the effect of three parameters

namely the initial copper concentration (A: 60 - 200 mg/L), the adsorbent dose (B: 0.1 - 0.6 g), the pH (C: 4 - 10) and their interactions on the copper removal efficiency. The one-block design was selected and the order of the experiments was not randomised. The default model is quadratic. To express the observed response as a function of the experimental factors, the second order polynomial model was used and is written as Equation (5).

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=3=1}^k \beta_{ii} X_{ii}^2 + \sum_{i=1}^k \sum_{i \neq j=1}^k \beta_{ij} X_i X_j \quad (5)$$

where β_0 is the constant, β_i and β_{ij} are the linear and quadratic coefficients of the input variable X_i respectively and β_{ij} is the interaction coefficient of the input variables X_i and X_j . Furthermore, the experimental design, the regression coefficients of the models, the analyses of variances and the graphs were determined using the software Design Expert (version 13). Also, the results of the experimental design represent the means of analyses performed in triplicate. The procedure used is summarised in **Figure 4**.

3. Results and Discussion

3.1. Characterization of Adsorbents

3.1.1. Physico-Chemical Properties

The different physico-chemical properties of the adsorbents are given in **Table 1**. These values obtained show on the one hand that titaniferous sand treated with sulphuric acid is very dense, a little acidic and a relatively low specific surface, while calcium silicate is very basic, medium dense than titaniferous sand with a very high specific surface.

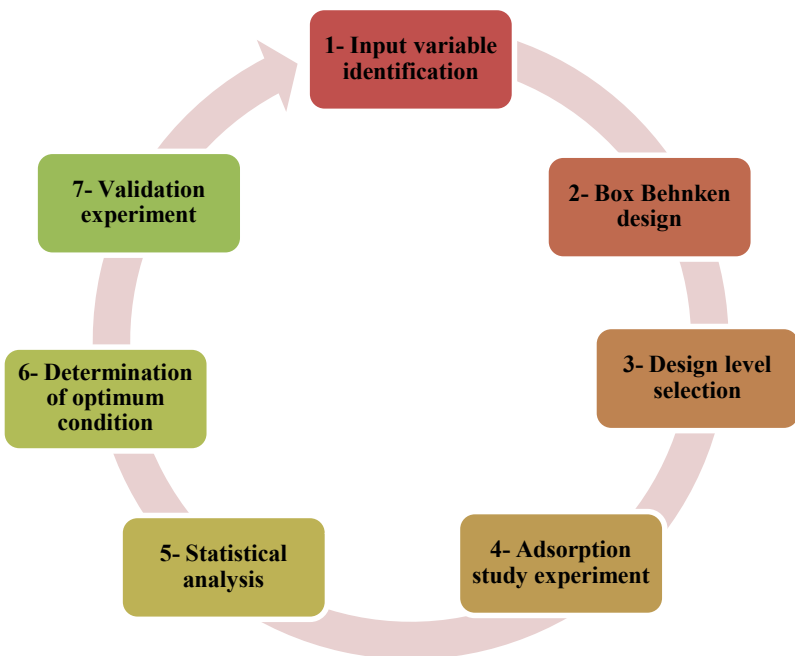


Figure 4. Experimental flow chart.

3.1.2. X-Ray Fluorescence Characterisation

Analysis of the titaniferous sand treated with sulphuric acid (Figure 5) shows that titanium, iron and aluminium are the predominant elements present at concentrations of 300,000 ppm, 63423.27 ppm and 18676.65 ppm respectively (Table 2). This confirms the presence of oxides such as titanium dioxide, iron oxide and alumina, which are essential elements in the adsorption of refractory organic and inorganic compounds. However, for calcium silicate (Figure 6), the major elements are calcium and silicon with concentrations of 349186.13 ppm and 136833.75 ppm respectively (Table 3). The literature has revealed a great importance given to these elements in the context of surface adsorption of organic compounds [22] and heavy metals [23].

Table 1. Physico-chemical characteristics of adsorbents.

physico-chemical properties	titaniferous sand	calcium silicate
pH	6.4	12.3
pH _{pzc}	8.75	9.2
Absolute density (kg/m ³)	4054	1980
Bulk density (kg/m ³)	2500	400
Porosity (%)	38	79
Specific surface (m ² /g)	13.4	314.2

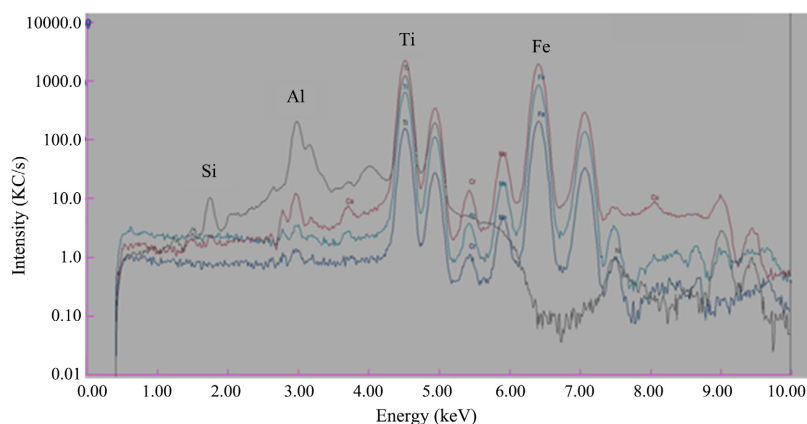


Figure 5. X-ray spectrum of titaniferous sand.

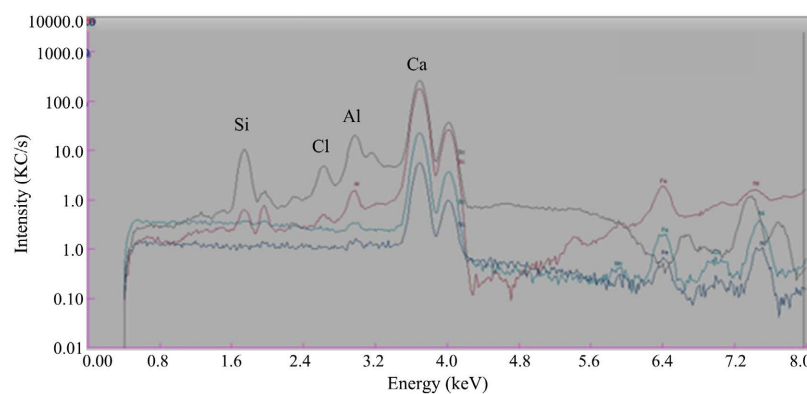


Figure 6. X-ray calcium silicate.

Table 2. Constituents of titaniferous sand.

Sample	Units	Ni	Al	Fe	Mn	Ca	
Titaniferous sand + H ₂ SO ₄ (4M)	ppm	92.77	18676.65	229129.56	6573.29	2233.36	
Si	Zn	V	Zr	Ti	P	Cr	As
63423.27	182	750.3	2421.75	3E+05	1130.31	1116.39	149.35
Se	S	Cl	Sn	Nb	Sr	Au	
15.94	544.35	167.69	28.71	430.57	6.04	105.8	

Table 3. Constituents of the synthesised calcium silicate.

Sample	Units	Al	Fe	Mn	Ca	Si	Zn			
Calcium silicate	Ppm	44685.42	266.44	209.63	349186.13	136833.75	10.4			
Zr	P	S	Cl	Sn	Mo	Nb	Sr	Rb	Co	Mg
3.1	3380.74	1079.6	461.24	6.52	7.35	5.72	110.58	1.16	44.31	1983.88

3.1.3. Characterisation with IR Spectroscopy

The different adsorbents such as titaniferous sand and calcium silicate were also characterised by the infrared spectroscopy method and the different bands obtained are given in **Figure 7** and **Figure 8**. For the silicate compounds of calcium silicate, the major absorption range of the (Si_pO_q)_n radicals is around 950 - 900 cm⁻¹ for the isolated silicates, which explains the strong absorption band visualised around 950 cm⁻¹. However, the other associated silicates or in sheets are detected at 1100 cm⁻¹. This characteristic absorption of silicates shifts towards higher frequencies. Similar results were found by Maglione *et al.* [24]. However, for titaniferous sand, no absorption band was detected but the pattern of the curve shows that the constituents in its structure are linked together by ionic bonds (crystal structure).

3.2. Statistical Analysis

In order to study the combined effect of the three factors, the experiments were conducted using the Box-Behnken design. A quadratic polynomial model was selected to establish the mathematical relationship between the removal efficiency and the independent variables. The levels of the factors and the experimental results of the different combinations between the operating variables are presented in **Table 4** and **Table 5** respectively.

The mathematical model relating the copper removal efficiency to the different experimental variables is given by equation 6. The significance of the model and the different terms associated with it is given by an analysis of the corresponding probabilities from **Table 6**. Thus the analysis of variance showed that the probabilities of the model ($p < 0.0001$), the effect of the initial concentration ($p = 0.0033$), the effect of the adsorbent dose ($p = 0.0155$) and the effect of the

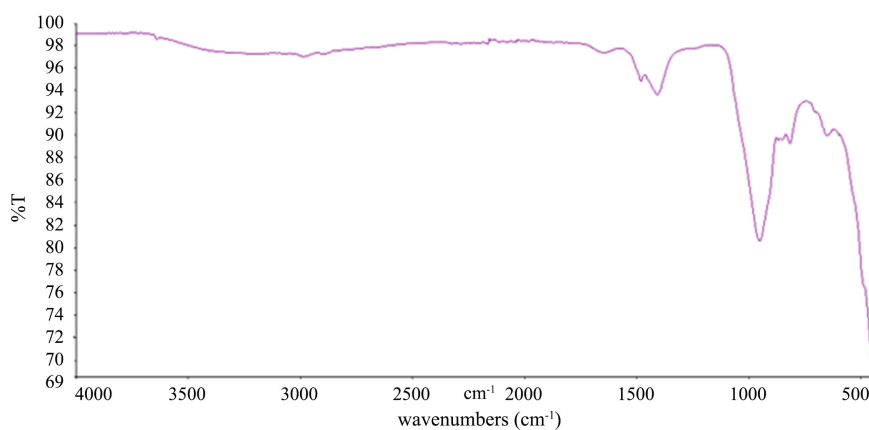


Figure 7. IR spectrum of calcium silicate.

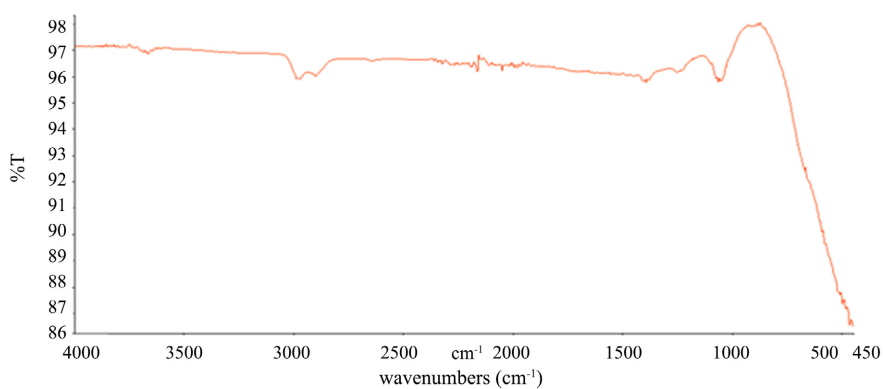


Figure 8. IR spectrum of titaniferous sand.

Table 4. Experimental variables and their levels.

Factors	Level	
	(+1)	(-1)
A: Concentration (mg/L)	60	200
B: Adsorbent dose (g)	0.1	0.6
C: pH	4	10

Table 5. Experience matrix.

Run	Factor 1	Factor 2	Factor 3	Response 1
	A: Concentration mg/L	B: Adsorbent dose g	C: pH	Yield %
1	200	0.1	7	99.89
2	60	0.35	4	59.2
3	130	0.35	7	99.92
4	130	0.1	10	99.87
5	200	0.6	7	99.91
6	130	0.35	7	99.89

Continued

7	60	0.1	7	92.44
8	130	0.35	7	99.91
9	130	0.35	7	99.9
10	60	0.35	10	99.74
11	130	0.6	10	99.9
12	130	0.35	7	99.92
13	200	0.35	10	99.91
14	60	0.6	7	99.67
15	130	0.6	4	62.54
16	200	0.35	4	70.17
17	130	0.1	4	56.11

Table 6. Analysis of variance in copper removal efficiency.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	4398.38	9	488.71	210.46	<0.0001	significant
A-Concentration	44.32	1	44.32	19.09	0.0033	
B-Adsorbent dose	23.50	1	23.50	10.12	0.0155	
C-pH	2865.24	1	2865.24	1233.90	<0.0001	
AB	13.00	1	13.00	5.60	0.0499	
AC	29.16	1	29.16	12.56	0.0094	
BC	10.24	1	10.24	4.41	0.0739	
A²	0.5449	1	0.5449	0.2347	0.6429	
B²	22.09	1	22.09	9.51	0.0177	
C²	1366.14	1	1366.14	588.32	<0.0001	
Residual	16.25	7	2.32			
Lack of Fit	16.25	3	5.42	31870.64	<0.0001	significant
Pure Error	0.0007	4	0.0002			
Cor Total	4414.63	16				

pH (<0.0001) were lower than the threshold value ($\alpha = 5\%$), therefore these last ones turned out to be significant. However, the significant interactions observed are those between the initial concentration and the dose of adsorbent (AB interaction) on the one hand and the initial concentration and the pH on the other hand (AC interaction). **Figures 9(a)-(c)** show the interaction between the different operating parameters and the analysis of the latter has shown that the yield reaches its maximum value when the initial concentration is at its maximum value and the mass of adsorbent is equal to 0.5 g. In other cases, a maximum yield is observed under the conditions where the initial concentration is in the defined range and the pH is equal to about 9.8 or the mass of adsorbent is in

the defined range and the pH is equal to about 9.8. Similar results were reported by L. YOUCEF, S. ACHOUR in the study of copper removal by chemical precipitation and adsorption processes [25]. Junaid Khan *et al.* also showed in the study of copper removal on modified hematite ($\alpha\text{-Fe}_2\text{O}_3$) iron oxide coated sand that the initial copper concentration, adsorbent dose and pH significantly influenced the copper removal efficiency [26].

$$\begin{aligned} \text{Yield} = & -73.951 + 0.140 * \text{Concentration} + 60.829 * \text{Adsorbent dose} \\ & + 36.746 * \text{pH} - 0.103 * \text{Concentration} * \text{Adsorbent dose} \\ & - 0.0128 * \text{Concentration} * \text{pH} - 2.133 * \text{Adsorbent dose} * \text{pH} \quad (6) \\ & + 7.341 \text{e-}05 * \text{Concentration}^2 - 36.644 * \text{Adsorbent dose}^2 \\ & - 2.001 * \text{pH}^2 \end{aligned}$$

Factor Coding: Actual

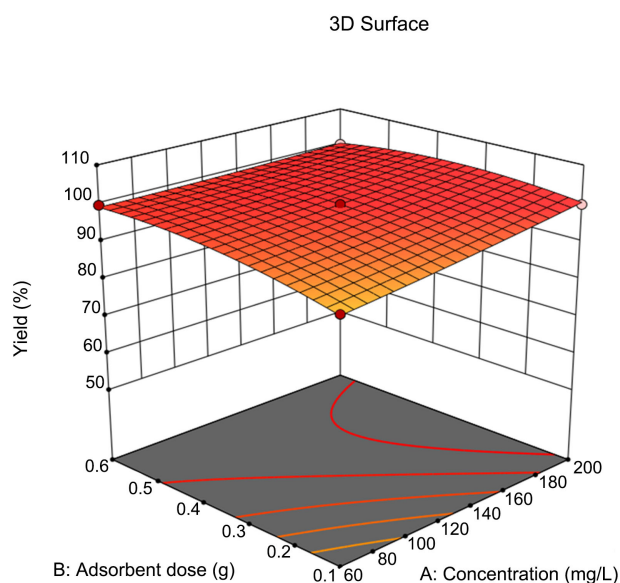
Yield (%)

Design Points:

● Above Surface
○ Below Surface
56.11  99.92

X1= A
X2= B

Actual Factor
C = 7



(a)

Factor Coding: Actual

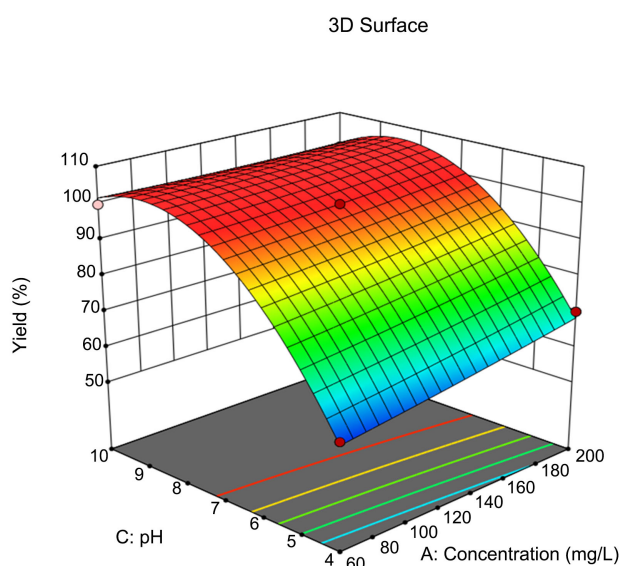
Yield (%)

Design Points:

● Above Surface
○ Below Surface
56.11  99.92

X1= A
X2= C

Actual Factor
B = 0.35



(b)

Factor Coding: Actual

3D Surface

Yield (%)

Design Points:

● Above Surface

○ Below Surface

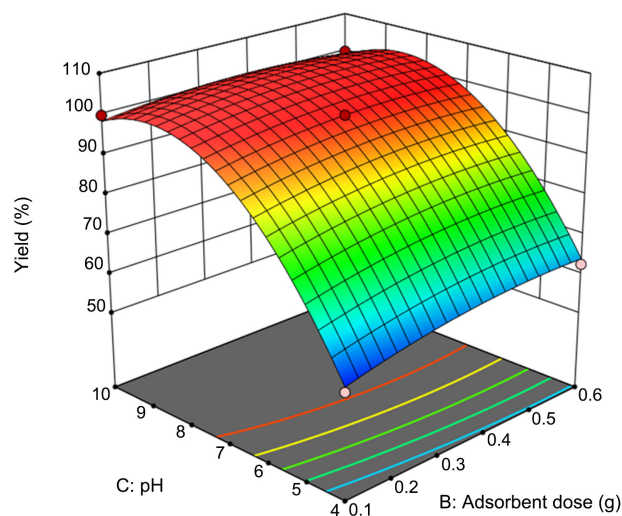
56.11  99.92

X1= B

X2= C

Actual Factor

A = 130



(c)

Figure 9. Three-dimensional plots of the interactions between adsorbent dose and concentration (a), pH and concentration (b), adsorbent dose and pH (c) on yield.

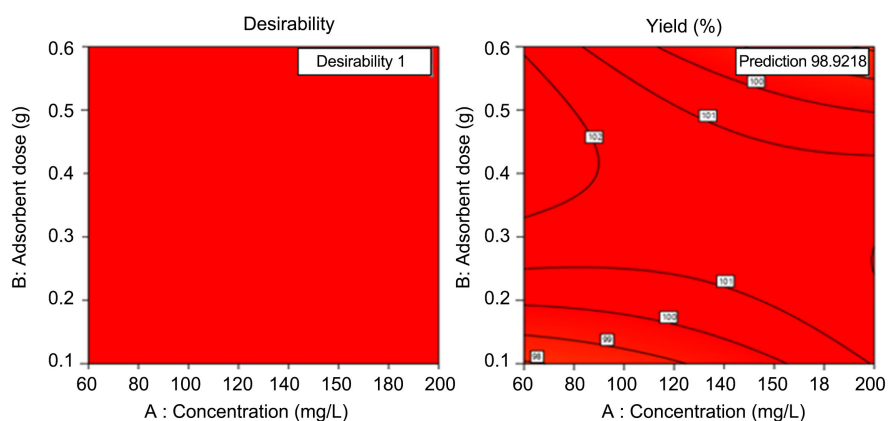


Figure 10. Optimisation diagram of the operating parameters for copper adsorption.

3.3. Optimisation by the Desirability Function

The desirability function (Figure 10) allows to simultaneously determine the optimal parameters of the input variables that can determine the optimal performance levels for one or more responses. It consists of converting each response (y_i) into a desirability function (d_i) varying between $0 \leq d_i \leq 1$. The value 0 is assigned when the factors lead to an unacceptable response. The value 1 is assigned when the response represents the maximum performance desired by the factors [27].

$$D = (d_1 * d_2 * \dots * d_n)^{1/n} \quad (7)$$

With n : the number of responses in the measure.

The optimal yield predicted by the desirability function is 98.92% under the

following optimal conditions: a dose adsorbent of 0.55 g, a concentration of 197.25 mg/L and a pH of 9.85. The confirmatory test showed a removal efficiency of 98.64% under the same conditions.

4. Conclusion and Prospects

The objective of this study was to use the response surface methodology through the Box-Behnken model to evaluate the performance of the adsorbent mixture in the process of copper removal in aqueous solution. The effects and interactions of different operating parameters such as initial copper concentration, adsorbent dose and pH were studied and the results confirmed that the Box-Behnken model was adequate to model and optimise the process of copper ion retention in solution by the adsorbent mixture. The analysis of variance (ANOVA) showed that the copper removal efficiency is strongly influenced by the concentration ($p = 0.0033$), the adsorbent dose ($p = 0.0155$) and the pH ($p < 0.0001$), their increases will strongly influence the magnitude of interest. Therefore, this adsorbent mixture can be used as a stable, environmentally friendly and effective adsorbent for the treatment of heavy metal contaminated waters. However, a dimensioning study of a fixed bed column has to be carried out in order to be able to treat effluents contaminated with refractory organic molecules and heavy metals in continuous mode on the one hand, and to carry out a simulation of the adsorption process in a fixed bed on the other hand.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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