

Competitive Heavy Metal Removal from Binary Solution

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Abstract

The removal of copper and cobalt ions from binary metal solutions on zeolite NaX by ion exchange process was investigated. Experiments were conducted in unbaffled glass reactor with a Rushton turbine as a stirrer. The dependence of ion exchange kinetics and the amount exchanged were tested using different initial concentrations of metal ions in mixtures. The results obtained indicate that the removal efficiency depends on the initial heavy metal concentrations in binary solutions. Experimental kinetics data were analysed using Ritchie and Weber-Morris models. According to AARD values, the rate in this study was reaction-controlled.

Keywords

Copper, cobalt, binary mixture, kinetic models

1 Introduction

An alarming environmental problem today is pollution by heavy metal ions. Many industries, such as metal plating or mining, discharge their metal-containing effluents into the environment without adequate treatment even though this type of pollution is considered one of the most harmful for the environment.¹ Most heavy metals, such as copper and cobalt, do not biodegrade into non-toxic end products, but tend to accumulate in the environment or in living organisms, causing various diseases and disorders.¹ The intake of copper from the environment results in its accumulation in the human liver causing serious gastrointestinal and kidney problems, while higher cobalt intake has harmful effects on the lungs, skin, and heart.^{2,3} Conventional heavy metal ions removal treatment methods from wastewaters include chemical precipitation, membrane separation processes, adsorption, coagulation, activated carbon adsorption, evaporation, solvent extraction, filtration cementation, chemical reduction, etc. The adsorption process is found to be a highly effective and economical method for the removal of various heavy metal ions from wastewater.⁴ It is a separation process in which the ions exchanged or adsorbed are not chemically altered, and the adsorbent can be easily recovered and reused by regeneration operations.⁵ The used adsorbent needs to be either disposed of or regenerated.⁵ Regeneration is important because of adsorbent reusability. Ezzeddine *et al.*⁶ found that the capacity for zeolite NaX after three consecutive copper adsorption-desorption cycles at 25 °C remained 92 %. Zeolites are a group of microporous, polyhedron aluminosilicates. They may be represented by the general formu-

la: $\text{MaMb}_{0.5}[(\text{Al}_2\text{O}_3)_x(\text{SiO}_2)_y(\text{H}_2\text{O})_z]$, where Ma and Mb are elements of the first and second group of the periodic element system, x and y represent the number of oxide variables, and z the number of water molecules. Primary zeolite structure consists of aluminium (AlO_4^-) and silicon (SiO_4) tetrahedral linked by oxygen atoms in secondary structures which connect in tertiary structure. Aside from being used as ion exchangers, zeolites are also used as catalysts (heterogeneous catalysts with shape/size-selective character) and molecular sieves.^{7,8}

The main goal of this work was to determine the efficiency of zeolite NaX as ion exchanger in the batch reactor to remove two different heavy metals, copper and cobalt, from a mixture of their solutions. In addition, one of the goals was to test obtained experimental kinetics data in order to verify that the determined just suspended impeller speed, N_{js} , provided conditions in which the reaction was the slowest step.

2 Experimental

2.1 Materials and methods

Zeolite NaX (Alfa Aesar) was crushed in a ball mill and sieved to obtain particles in the range 0.050–0.063 mm. Solutions containing Cu or/and Co ions were prepared using the appropriate weight of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (Kemika) and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Kemika), and by dissolving it in ultrapure water. The initial concentrations of copper and cobalt in the solutions were determined by Atomic Absorption Spectrometer Perkin Elmer AAnalyst 600 (AAS). The initial concentrations used in the experiments are listed in Table 1.

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Table 1 – Initial concentrations used in the experiments
 Tablica 1 – Početne koncentracije u provedenim eksperimentima

Experiment	Copper/ mmol l^{-1}	Cobalt/ mmol l^{-1}
1	3.348	–
2	–	3.202
3	1.698	2.132
4	0.846	2.722
5	2.453	0.899

All experiments were carried out in a glass batch reactor (internal diameter $d_T = 0.14$ m (Fig. 1a)).

Height of the solution, H , was equal to the internal reactor diameter ($H = d_T$). The suspension, 2.1 l solution containing copper or cobalt or both metals and 10.5 g of zeolite, was stirred, at $T = 300$ °C, using Rushton turbine (Fig. 1b). Impeller diameter, D , was 0.080 m. The impeller off-bottom clearance (C/H) was 0.33. Mixing was performed by Lightnin LabMaster LB2 mixer, a mixer that allows impeller speed regulation and torque measurement during process time. Just suspended impeller speed, N_{JS} , and power consumption at just suspended impeller speed, P_{JS} , were determined. Just suspended impeller speed, was determined by applying visual Zwietering method – criterion 1 s.⁹ While measuring the critical impeller speed, the reactor base was illuminated, and the mirror was positioned below its bottom. Impeller speed was continuously increased until all particles of zeolite, settled at the bottom, were in motion and none remained on the reactor bottom for more than 1 second. The average value was calculated after measurements had been repeated ten times.

P_{JS} was calculated as:

$$P_{JS} = 2 \pi N_{JS} \tau \quad (1)$$

where τ represents torque (N cm), which was measured using Lightnin LabMaster LB2 mixer.

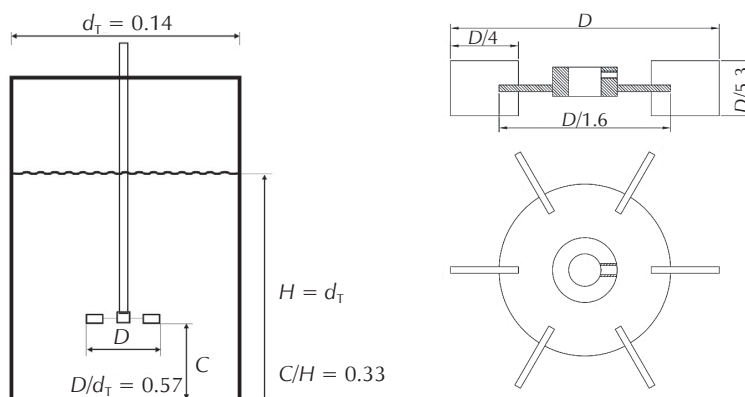


Fig. 1 – Geometric characteristics of un baffled batch reactor (a), and Rushton turbine (b)

Slika 1 – Geometrijske karakteristike kotlastog reaktora bez razbijala virova (a) i Rushtonove turbine (b)

The kinetics data were obtained in experiments lasting 30 min. For the purpose of the kinetic study, samples were taken from the batch reactor at defined times. Preceding the sample analysis with AAS, the samples were centrifuged and filtrated. The amount of metal retained on the zeolite, q_t , was calculated as:

$$q_t = \frac{(c_0 - c_t)V}{m} \quad (2)$$

where c_0 is the initial concentration of metal solution (mmol l^{-1}), c_t is the concentration of metal solution at time t (mmol l^{-1}), V is the volume of solution (l), and m is the mass of zeolite (g).

2.2 Kinetic models

Obtained kinetic experimental data were analysed using Ritchie reaction-based models, and Weber-Morris diffusion-based model.

Ritchie model is expressed as:¹⁰

$$q_t = q_e \left[1 - \frac{1}{1 + kt} \right] \quad (3)$$

where q_e is amount of copper ions exchanged at equilibrium (equilibrium capacity or maximum amount of copper exchanged) (mmol g^{-1}), and k is the reaction rate constant ($\text{mmol g}^{-1} \text{min}^{-1}$).

Weber-Morris model, diffusion-based model, is presented as:^{11,12}

$$q_t = k_d \sqrt{t} + I \quad (4)$$

where, k_d is the diffusion rate constant ($\text{mmol g}^{-1} \text{min}^{-1/2}$), t is time (min), and I is the intercept (mmol g^{-1}).

3 Results and discussion

When the complete suspension is attained, no particle stays on the bottom of the reactor for more than 1 to 2 s. This state is characterised by the complete motion of all particles, and the maximum surface area of the particles is exposed to the solution for transfer processes or the reaction in the reactor.¹³ The N_{JS} was measured 10 times and the value of 74 rpm was calculated as the arithmetic mean. A power consumption of 0.3119 W was calculated by Eq. (1). The kinetics of the copper and cobalt ion exchange, separately and in a mixture of the metal solutions, was studied at given N_{JS} , and the concentrations of metals measured by AAS are presented in Tables 2–5.

Table 2 – Removal of copper or cobalt from single component solution
Tablica 2 – Uklanjanje iona bakra ili kobalta iz otopine jednog iona

t/min	$\text{Cu} - c_t/\text{mmol l}^{-1}$	$\text{Cu} - q_t/\text{mmol g}^{-1}$	$\text{Co} - c_t/\text{mmol l}^{-1}$	$\text{Co} - q_t/\text{mmol g}^{-1}$
0	3.348	0.000	3.202	0.000
0.75	0.012	0.667	0.055	0.629
2	0.002	0.669	0.022	0.636
4	0.004	0.669	0.019	0.637
7	0.003	0.669	0.020	0.636
11	0.003	0.669	0.016	0.637
15	0.004	0.669	0.013	0.638
22	0.004	0.669	0.013	0.638
30	0.004	0.669	0.011	0.638

Table 3 – Removal of copper and cobalt from binary component solution (56 % Co + 44 % Cu)
Tablica 3 – Uklanjanje bakra i kobalta iz binarne smjese (56 % Co + 44 % Cu)

t/min	$c_t(\text{Cu+Co})/\text{mmol l}^{-1}$	$q_t(\text{Cu+Co})/\text{mmol g}^{-1}$	$c_t(\text{Cu})/\text{mmol l}^{-1}$	$q_t(\text{Cu})/\text{mmol g}^{-1}$	$c_t(\text{Co})/\text{mmol l}^{-1}$	$q_t(\text{Co})/\text{mmol g}^{-1}$
0	3.831	0.000	1.698	0.000	2.132	0.000
0.75	0.021	0.762	0.003	0.339	0.018	0.423
2	0.013	0.764	0.002	0.339	0.011	0.424
4	0.011	0.764	0.001	0.339	0.010	0.425
7	0.009	0.764	0.001	0.339	0.008	0.425
11	0.008	0.765	0.001	0.339	0.007	0.425
15	0.008	0.765	0.001	0.339	0.007	0.425
22	0.009	0.764	0.002	0.339	0.007	0.425
30	0.007	0.764	0.001	0.339	0.006	0.425

Table 4 – Removal of copper and cobalt from binary component solution (76 % Co + 24 % Cu)
Tablica 4 – Uklanjanje bakra i kobalta iz binarne smjese (76 % Co + 24 % Cu)

t/min	$c_t(\text{Cu+Co})/\text{mmol l}^{-1}$	$q_t(\text{Cu+Co})/\text{mmol g}^{-1}$	$c_t(\text{Cu})/\text{mmol l}^{-1}$	$q_t(\text{Cu})/\text{mmol g}^{-1}$	$c_t(\text{Co})/\text{mmol l}^{-1}$	$q_t(\text{Co})/\text{mmol g}^{-1}$
0	3.568	0.000	0.846	0	2.722	0.000
0.75	0.154	0.683	0.002	0.169	0.152	0.514
2	0.043	0.705	0.001	0.169	0.042	0.536
4	0.032	0.707	0.001	0.169	0.032	0.540
7	0.023	0.709	0.001	0.169	0.022	0.541
11	0.018	0.710	0.001	0.169	0.017	0.542
15	0.012	0.711	0.001	0.169	0.012	0.542
22	0.013	0.711	0.002	0.169	0.011	0.542
30	0.011	0.711	0.001	0.169	0.010	0.542

Table 5 – Removal of copper and cobalt from binary component solution (27 % Co + 73 % Cu)

Tablica 5 – Uklanjanje bakra i kobalta iz binarne smjese (27 % Co + 73 % Cu)

t/min	$c_t(\text{Cu+Co})/\text{mmol l}^{-1}$	$q_t(\text{Cu+Co})/\text{mmol g}^{-1}$	$c_t(\text{Cu})/\text{mmol l}^{-1}$	$q_t(\text{Cu})/\text{mmol g}^{-1}$	$c_t(\text{Co})/\text{mmol l}^{-1}$	$q_t(\text{Co})/\text{mmol g}^{-1}$
0	3.352	0.000	2.453	0.000	0.899	0.000
0.75	0.042	0.662	0.021	0.486	0.021	0.176
2	0.023	0.666	0.009	0.489	0.014	0.177
4	0.021	0.668	0.011	0.488	0.010	0.178
7	0.014	0.668	0.006	0.489	0.008	0.178
11	0.013	0.668	0.006	0.489	0.007	0.178
15	0.009	0.669	0.004	0.490	0.005	0.179
22	0.004	0.670	0.002	0.490	0.002	0.179
30	0.005	0.669	0.001	0.490	0.004	0.179

The rate of metal removal, separately and in a mixture, tended to decrease over time. The results showed that, in general, the exchange process proceeded in two steps. During the first, rapid step, a major part of ions was exchanged; this step took less than a minute. In the following second step, the ion exchange remained essentially unchanged. The initial faster rate of exchange may be explained by the large and accessible surface area of the small zeolite particles, and achievement of complete suspension.

The amount of copper or cobalt removed from the mixture of metal solutions is lower than the amount of these metals removed from their own solutions. However, the total amount of metals removed from the mixture of their solutions is greater than the amount of single metal removed from monometallic solutions, meaning that the exchanged amount in binary system was higher than that of monometallic system. Further study of data in Tables 2–5 shows that the total initial concentration of metals also increases. So, no reduction or increase in the uptake of copper ions were found compared to single metal system. For example, for single metal system, the copper concentration was $3.348 \text{ mmol l}^{-1}$ and q_e was $0.669 \text{ mmol g}^{-1}$, and for one of the mixtures (Table 5), the copper initial concentration was 73 % of initial concentration of single metal solution, and q_e for copper ions was also 73 % of q_e in single metal solutions. The same could be found for mixtures in Tables 3

and 4, as well as for cobalt. For these reasons, this behaviour could be associated with the non-competitive effect. Non-competitive effect and synergy are rarely obtained in multicomponent mixtures. Usually, the antagonistic competitive effect is found and explained by greater suppression that leads to electrostatic repulsion.¹⁴ The exceptions from this behaviour were found for the removal of copper, chromium, and cadmium on lava ash where the non-competitive effect was found, and for the removal of copper and mercury on chitosan membranes.¹⁵

In order to find the kinetic model with the highest accuracy that fitted the experimental data, the kinetic analysis of copper and cobalt ion exchange on the zeolite NaX, separately and in a mixture was performed. The parameters of the Ritchie and Weber-Morris model were determined (Tables 6 and 7). The AARD was calculated and used to evaluate the best fitting model with the experimental data (Tables 6 and 7). AARD is acceptable up to 5 %, and above 10 % indicates a poor approximation of model predicted values:¹⁶

$$\text{AARD} = \frac{1}{n} \sum_{i=1}^n \left[\left| \frac{q_{ti} - qt_model_i}{qt_i} \right| 100 \right] \quad (5)$$

Where, q_t is experimental value of q_t , and qt_model is calculated value of q_t , n is number of experimental data.

Table 6 – Kinetic parameters for Cu

Tablica 6 – Kinetički parametri za Cu

Model	Parameter	100 % Cu	73 % Cu	44 % Cu	24 % Cu
experimental data	$q_{e\text{exp}}/\text{mmol g}^{-1}$	0.669	0.490	0.339	0.169
Ritchie model	$k/\text{g mmol}^{-1} \text{ min}^{-1}$	557.209	179.054	226.175	1088.244
	$q_e/\text{mmol g}^{-1}$	0.669	0.490	0.340	0.169
	AARD/%	0.030	0.059	0.094	0.050
Weber-Morris model	$k_d/\text{mmol g}^{-1} \text{ min}^{-1/2}$	0.069	0.051	0.035	0.035
	$I/\text{mmol g}^{-1}$	0.409	0.298	0.207	0.207
	AARD/%	12.988	12.951	12.983	12.983

Table 7 – Kinetic parameters for Co
 Tablica 7 – Kinetički parametri za Co

Model	Parameter	100 % Co	76 % Co	56 % Co	27 % Co
experimental data	$q_{e,exp}/\text{mmol g}^{-1}$	0.638	0.542	0.425	0.179
Ritchie model	$k/\text{g mmol}^{-1} \text{min}^{-1}$	99.792	24.157	241.396	65.016
	$q_e/\text{mmol g}^{-1}$	0.638	0.544	0.425	0.179
	AARD/%	0.053	0.141	0.016	0.154
Weber-Morris model	$k_d/\text{mmol g}^{-1} \text{min}^{-1/2}$	0.066	0.058	0.044	0.019
	$l/\text{mmol g}^{-1}$	0.386	0.320	0.259	0.108
	AARD/%	12.962	12.970	12.974	12.908

The ion exchange rate of copper ions from solution was faster than the exchange of cobalt ions, both in monometallic metal solutions and the mixture. Thus, in binary, as in single systems, the kinetic adsorption order followed as copper > cobalt. These data are in accordance with the data obtained by other authors, and it can be concluded that higher molecular weight metals are removed much more than are lower molecular weight metals.^{17,18}

Ritchie's model assumes that the total reaction rate is controlled by the rate of ion exchange, and that the ion exchange is a second order reaction, i.e., in this case, the exchange itself took place by exchanging two ions of sodium from the zeolite with one ion of divalent metal from the solution. The Weber-Morris model was used to test whether the diffusion process defines the overall rate of reaction. This model can also show which diffusion (film or intraparticle) controlled the total rate. According to AARD values, the rate in this study was reaction-controlled.

4 Conclusion

The results obtained in this study lead to the conclusion that zeolite NaX can be used for effective removal of selected heavy metals from effluents containing copper and cobalt ions. The exchanged amount of copper was maintained more or less constant since the first contact time. The values of the exchanged rates of metal ions conducted in the mixture of metal ion solutions showed higher values than the rates of ion exchange conducted in the solutions of only one type of ion. The increase in the amount of metals removed from their binary mixture resulted from the higher total initial concentration of the metals in the binary mixture, and not due to synergistic behaviour of copper and cobalt ions in a binary metal solution. The behaviour in mixtures could be associated with the non-competitive effect. The experimental kinetics data can be fitted by different reaction- or diffusion-based models. Results obtained showed that the exchange of copper and cobalt on zeolite NaX, both from monometallic and bimetallic solutions, was better described by the reaction-based model. The process followed the second order kinetic model, i.e., in this case, the exchange itself took place by exchanging one ion of divalent metal from the solution with two ions of

sodium from the zeolite. The ion exchange rate of copper ions from solution was higher, both in single and in a binary metal solution, than the ion exchange rate of cobalt ions.

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List of abbreviations and symbols Popis kratica i simbola

- AARD – average absolute relative deviation, %
 – srednja vrijednost relativnog odstupanja podataka, %
- C – impeller bottom clearance, m
 – udaljenost miješala od dna posude, m
- c_0 – initial concentration of metal solution, mmol l^{-1}
 – početna koncentracija otopine metala, mmol l^{-1}
- c_t – concentration of metal solution at time t , mmol l^{-1}
 – koncentracija otopine metala u vremenu t , mmol l^{-1}
- D – impeller diameter, m
 – promjer miješala, m
- d_T – internal reactor diameter, m
 – promjer reaktora, m
- H – height of the solution, m
 – visina suspenzije, m
- l – intercept, mmol g^{-1}
 – odsječak na osi y , mmol g^{-1}
- k – reaction rate constant, $\text{mmol g}^{-1} \text{min}^{-1}$
 – konstanta brzine reakcije, $\text{mmol g}^{-1} \text{min}^{-1}$
- k_d – diffusion rate constant, $\text{mmol g}^{-1} \text{min}^{-1/2}$
 – konstanta brzine difuzije, $\text{mmol g}^{-1} \text{min}^{-1/2}$
- m – mass of zeolite, g
 – masa zeolita, g
- n – number of experimental data
 – broj eksperimentalnih podataka
- N_{JS} – just suspended impeller speed, rpm
 – kritična brzina suspendiranja, o min^{-1}

P_{js}	– power consumption at just suspended impeller speed, W – snaga pri kritičnoj brzini suspendiranja, W
q_e	– amount of metal ions exchanged at equilibrium, mmol g^{-1} – količina iona metala vezana na zeolit u ravnoteži, mmol g^{-1}
q_t	– amount of metal ions exchanged at time t , mmol g^{-1} – količina iona metala vezana na zeolit u trenutku t , mmol g^{-1}
q_{t_model}	– calculated value of q_t , mmol g^{-1} – izračunata vrijednost q_t , mmol g^{-1}
T	– temperature, $^{\circ}\text{C}$ – temperatura, $^{\circ}\text{C}$
t	– time, min – vrijeme, min
V	– volume of solution, l – volumen otopine, l
τ	– torque, N cm – zakretni moment, N cm

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SAŽETAK

Konkurentno uklanjanje teških metala iz binarne otopine

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Ispitano je uklanjanje iona bakra i kobalta iz binarnih otopina metala ionskom izmjenom na zeolitu NaX. Eksperimenti su provedeni u staklenom kotlastom reaktoru bez razbijala virova. Kao miješalo upotrijebljena je Rushtonova turbina. Ovisnost kinetike izmjene iona i izmijenjene količine iona metala ispitana je pri različitim početnim koncentracijama iona metala u smjesama. Prema dobivenim rezultatima, učinkovitost uklanjanja iona metala iz otopina ovisi o početnim koncentracijama metala u binarnim otopinama. Kinetički podatci dobiveni eksperimentom analizirani su Ritchievim i Weber-Morrisovim modelom. Prema vrijednostima AARD-a, ukupna brzina procesa u ovoj studiji kontrolirana je reakcijom.

Ključne riječi

Bakar, kobalt, binarna smjesa, kinetički modeli

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