

MACHINING OF PARTS AND REDUCING THE CONTENT OF COPPER CATIONS FROM AN AQUEOUS SOLUTION BY SORPTION ON A SOLID LAYER USING NATURAL AND MODIFIED CLINOPTILOLITE

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Galvanic plating is one of the most widespread methods of surface treatment of metal parts. The process consists of immersion of modified parts in an electrolytic bath. These processes produce wastewater with a high metal content. One of the methods of reduction metal content in water is sorption on a solid layer. Natural zeolites in natural form, but also in modified form, can be used for these purposes. To be able to design organization of the cleaning process, it is necessary to know the kinetic course of sorption. The article contains the results of laboratory experiments aimed at determining the sorption parameters of the sorption of copper and nickel cations on natural zeolite - clinoptilolite, and on modified Fe-clinoptilolite.

KEYWORDS

metal ions, wastewater, clinoptilolite, sorption, kinetic

1 INTRODUCTION

Industrial wastewater containing heavy metals must undergo special treatment and only after the removal of these pollutants can they be discharged into the public sewer system. Wastewater containing heavy metals is produced in the metallurgy and engineering industry, among other things, also during surface treatment. Metal ions in water are not biodegradable and cause health problems. There are several physical and chemical methods such as chemical precipitation, filtration, coagulation, membrane processes, ion exchange, which are used to remove metal ions from water [Korkmaz 2012, Roca 2017]. These methods have various limitations, such as high financial costs and low removal efficiency, so other methods, such as sorption, are being researched [Bailey 1999, Inglezakis 2002, Lehman 2014]. Sorption is a highly efficient method that provides design flexibility and is easy to operate. Based on the latest literature, sorption can be classified as one of the methods that have recently gained a dominant position for water treatment processes, especially if locally available, economically inexpensive natural materials are used. Such materials include natural zeolites, which, due to their chemical composition, represent inorganic, aluminosilicate cations. Zeolite as a supporting matrix has the prerequisites to potentially meet the demanding criteria for the production of

new composite materials. A large number of experiments, as well as model studies of the adsorption of heavy metals on various materials, are described in publications [Monkova 2013, Barlokova 2014, Michalik 2014, Panda 2014, Baron 2016, Mrkvica 2016, Macala 2017, Balara 2018, Chaus 2018, Duplakova 2018, Pandova 2018 & 2020, Sukhodub 2018 & 2019, Bolisetz 2019, Flegner 2019 & 2020, Harnicarova 2019].

Various adsorbents, such as activated carbon [Pyrzynska 2010], carbon nanotubes [Li 2002, Lu 2006] and a large number of biosorbents were useful [Lloyd-Jones 2004]. However, there is still a need for new and effective adsorbents with local availability in addition to economic suitability.

2 WASTEWATER PURIFICATION

In water treatment technology, sorption is one of the newest methods. For a discontinuous arrangement of the process, it is necessary to know the kinetic course of sorption. Therefore, the purpose of our experiments was to obtain important characteristics of sorption mechanisms and to explore the possibility of technological use of natural zeolite-clinoptilolite in the process of water purification. Chemical modifications can be used to improve the sorption properties of zeolites. Iron oxides are used to improve the ability of zeolites to sorb heavy metals. They are either added to the reaction mixture during the zeolite synthesis or deposited on the zeolite surface. Several authors deal with the study of the removal of heavy metal ions by sorption, and in their works, they analyze equilibrium adsorption data using the Langmuir and Freundlich isotherm models [El-Sherif 2013, Khin 2012].

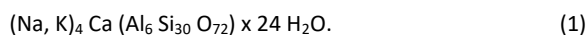
Based on various studies, it has been shown that zeolite with iron oxide on the surface has a higher sorption capacity for Cu, Pb, Zn, Mn and Cd. The reason for this phenomenon is the increase in Fe-OH binding sites contained in iron oxides. Some heavy metals have the ability to adsorb onto zeolite not only by ion exchange, but also by binding to specific sites containing Al-OH, Si-OH or Fe-OH [Dimirkou 2008, Doula 2009, Nguyen 2015]. Several models can be used to describe the adsorption of pollutants from aqueous solutions. One is the pseudo-second-order model. The advantage of using this model is that it is not necessary to know the equilibrium capacity from the experiments, as it can be calculated from the model [Weckhuysen 2015]. A very frequently used method of removing iron from water is by contact filtration, namely filtration on manganese filters [Zaborowski 2007, Adamcik 2014, Svetlik 2014, Rimar 2016, Olejarova 2017 & 2021, Sedlackova 2017, Catlos 2018, Labun 2018, Gamec 2019, Kuznetsov 2019, Murcinkova 2019, Pollak 2019 & 2020, Straka 2021 & 2022, Vagaska 2021].

The addition of potassium permanganate forms a coating on the surface of the filter cartridge, which serves as an oxidation catalyst. The oxidation state of the MnO_x filler coating plays an important role in the removal of dissolved iron [Barlokova 2014].

3 EXPERIMENTAL

The subject of the research was the use of natural and chemically modified zeolite clinoptilolite for the purpose of reducing the content of copper, nickel and iron cations from wastewater. The natural zeolite clinoptilolite was used for the experiments.

Clinoptilolite composition is expressed by formula [Zeocem 2013]:



Chemical and physical properties of the clinoptilolite are presented in Table 1.

Table 1. Clinoptilolite chemical composition [Zeocem]

SiO ₂	65 - 71.3 %	Fe ₂ O ₃	0.7 - 1.9 %
Al ₂ O ₃	11.5 - 13.1 %	MgO	0.6 - 1.2 %
CaO	2.7 - 5.2 %	Na ₂ O	0.2 - 1.3 %
K ₂ O	2.2 - 3.4 %	TiO ₂	0.1 - 1.3 %
P ₂ O ₅	0.02 %	Si/Al	4.5 - 5.4 %

To improve the sorption capacity for the sorption of copper cations, natural clinoptilolite was modified to Fe - modification by treatment with iron oxide (Fe₂O₃) with a molar concentration of 1mol·dm⁻³ for 24 hours. A sample of Fe-clinoptilolite was compared with a sample of natural zeolite. For the sorption of nickel cations, natural clinoptilolite was modified into ammonium form. A natural form of clinoptilolite was used also for the sorption of iron cations. All experiments were carried out at a temperature of 22 °C. The kinetic course of sorption of successively monitored cations was investigated. At regular time intervals, a sample of the solution was taken, and the concentration of cations was determined. The optimal time for reaching equilibrium in the sorption system is determined from the kinetic dependences.

4 RESULTS AND DISCUSSION

The change in the concentration of copper cations in the solution under the continuous action of the sorbent was recorded at precise time intervals until the equilibrium state of the system. Measurements were performed three times with both natural zeolite clinoptilolite and modified Fe clinoptilolite. Average values are shown in Tables 2 and 3. Sorption took place at pH = 4.03. For the experiments, we used 40g zeolite samples and model samples of aqueous solutions of copper cations.

Table 2. Decrease of copper cations in the solution using clinoptilolite

Time [min.]	c [g·dm ⁻³]
0	1.90
15	1.88
30	1.54
45	0.35
60	0.29
75	0.29
90	0.29

Table 3. Decrease of copper cations in the solution using modified Fe - clinoptilolite

Time [min.]	c [g·dm ⁻³]
0	1.90
15	1.48
30	0.56
45	0.46
60	0.11
75	0.07
90	0.07

The kinetic course of sorption is shown graphically in Figure 1 and 2.

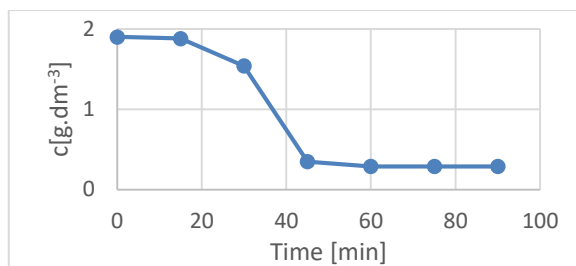


Figure 1. Decrease of copper cations in the solution using clinoptilolite

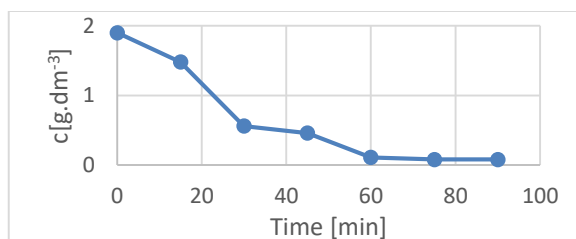


Figure 2. Decrease of copper cations in the solution using modified Fe - clinoptilolite

The equilibrium state of the system was reached in the first case after 60 minutes and in the second case after 75 minutes.

The specific adsorption of a certain component is expressed by relation (2) [Nguyen 2015, Ghobadi Nejad 2018].

$$a = \frac{c_0 - c_{eq}}{m} \times V \quad (2)$$

The quantitative distribution parameter of the substance between the solid and the liquid phase is the K_D partition coefficient [Jordy 2005]. Partition coefficient is defined as the ratio of the sorbed quantity of monitored cations in the solid matrix (sorbent) to their concentration in the aqueous solution c_{eq} during the equilibrium state. The importance of determining partition coefficients lies in the fact that they belong to the basic input data in mathematical models of the transport of monitored substances in water. We calculated the distribution coefficient according to the formula (3).

$$K_d = \frac{c_0 - c_{eq}}{c_{eq}} \times \frac{V}{m} \quad (3)$$

The distribution coefficients for natural clinoptilolite had the value K_d = 0.034; 0.041; 0.035 and for the modified coefficient it had the value K_d = 0.156; 0.062 and 0.159.

Table 4. Measured and calculated parameters

c ₀ [g · dm ⁻³]	c _{eq} [g · dm ⁻³]	a [g · g ⁻¹]	K _d
1.9	0.29	0.010	0.036
1.9	0.07	0.012	0.125

The measurements were performed three times under the same conditions. The Grubbs test excluded extreme values from the set of measured values. The lowest and highest values were tested by substituting them into equations (4), (5), and then compared to the Grubbs test table value. The standard deviations were evaluated according to equation (6), their numerical values were determined as 0.004506; 0.6153; 0.00308 and 0.01732.

$$H_{min} = \frac{|x_{avg} - x_{min}|}{s} \quad (4)$$

$$H_{max} = \frac{|x_{avg} - x_{max}|}{s} \quad (5)$$

$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - x_{avg})^2}{n-1}} \quad (6)$$

the efficiency of the sorbents was calculated according to (7)

$$\eta = \left(\frac{c_0 - c_{eq}}{c_0} \right) \times 100 \quad (7)$$

The effect of modifying the natural zeolite clinoptilolite into the Fe form was confirmed experimentally. The absorbed amount of copper cations increased slightly, the efficiency of 84% increased to 95%.

The study of the course of the adsorption isotherm of copper cations was carried out on natural zeolite clinoptilolite with a grain size of 2,5 mm - 5 mm originating from Nizny Hrabovec. For the sorption of copper cations, we used model solutions with an initial mass concentration of 2.6 g·dm⁻³; 5.0 g·dm⁻³; 7.0 g·dm⁻³; 8.6 g·dm⁻³ and 9.0 g·dm⁻³. To define the time required to reach equilibrium in the system, we monitored the dependence of the sorbed amount on the contact time of the sorbent with the adsorbate at a temperature of 25 °C. We processed the results graphically and mathematically evaluated them using the Freundlich adsorption isotherm. The course of sorption in all model samples was monitored in precise one-hour time intervals. With increasing contact time of the sorbent with the solution, the concentration of copper cations in the solution asymptotically approached the equilibrium concentration c_{eq} . The amount of adsorbed copper cations was calculated according to equation (2) as the difference between the initial concentration c_0 and the concentration in the solution in the equilibrium state c_{eq} , where a is the sorption capacity [mg g⁻¹], V is the volume of the solution and m is the mass of the sorbent.

Table 5. Measured and calculated parameters

c_0 [g·dm ⁻³]	c_{eq} [mg·dm ⁻³]	a [mg·g ⁻¹]	Log a	Log c_r
2.6	190	15.06	1.178	2.279
5.0	280	29.5	1.470	2.45
7.0	500	40.6	1.608	2.69
8.6	800	48.8	1.688	2.90
9.0	820	51.1	1.708	2.914

The least-squares method was used to calculate the constants of the Freundlich isotherm, where the slope k and the coefficient q were evaluated according to the equations (8), (9).

We determined the values of the isotherm constants using the method of least squares. The least-squares method was used to calculate the constants of the Freundlich isotherm, where the slope k and the coefficient q were evaluated according to the equations (7), (8).

$$k = - \frac{n \cdot \sum_{i=1}^n x_i \cdot y_i - \sum_{i=1}^n y_i \cdot \sum_{i=1}^n x_i}{n \cdot \sum_{i=1}^n x_i^2 - \left(\sum_{i=1}^n x_i \right)^2} \quad (8)$$

$$q = \frac{1}{n} \cdot \left(\sum_{i=1}^n x_i - k \cdot \sum_{i=1}^n y_i \right) \quad (9)$$

$$x = \log c_e, y = \log a$$

The measured sorption data are consistent with the Freundlich sorption isotherm

$$a = 0.088 c_e^{0.869} \quad (10)$$

If $n = 1/k$ and $K_F = 10^q$, then K_F reaches the value of 0.088 and for coefficient n , the value of 1.15. Isotherm can be expressed by equation (10).

The reduction of the nickel cations content was investigated on both natural clinoptilolite and modified clinoptilolite to NH₄⁺ form. The changes of the concentration of Ni²⁺ ions are in the Table 6.

Table 6. Change in the concentration of Ni²⁺ ions in the solution in time intervals

Sorbente	Clinoptilolite	NH ₄ ⁺ clinoptilolite
Time [minutes]	c [g · dm ⁻³]	c [g · dm ⁻³]
0	2.00	2.00
60	1.50	1.56
120	1.38	1.19
180	1.12	1.02
240	1.12	0.94
300		0.94

By calculation according to (6), we found that the treatment of zeolite into ammonium form increased the efficiency of the sorbent against nickel ions from 44% to 53%. The change in the concentration of nickel ions in the model solutions using both sorbents is shown graphically in Figures 3 and 4. The kinetic course of sorption is shown graphically in Figures 5 and 6.

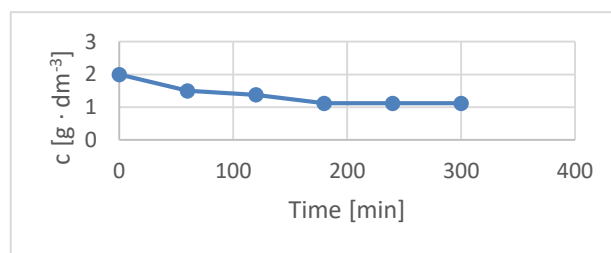


Figure 3. Change in the concentration of Ni²⁺ ions in the solution using clinoptilolite

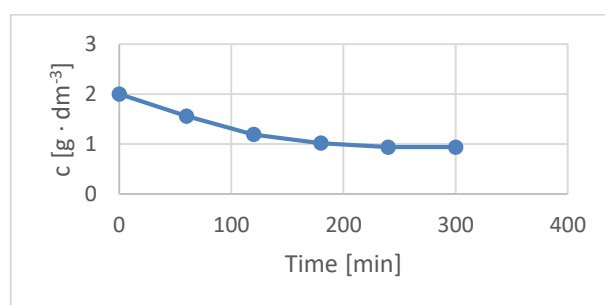


Figure 4. Change in the concentration of Ni²⁺ ions in the solution using NH₄⁺ clinoptilolite

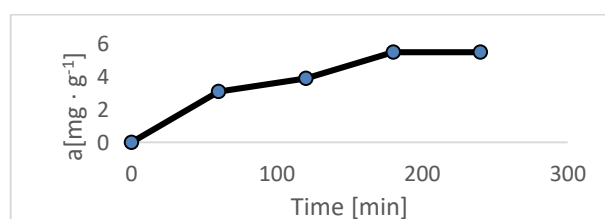


Figure 5. Kinetic course of Ni²⁺ sorption on clinoptilolite

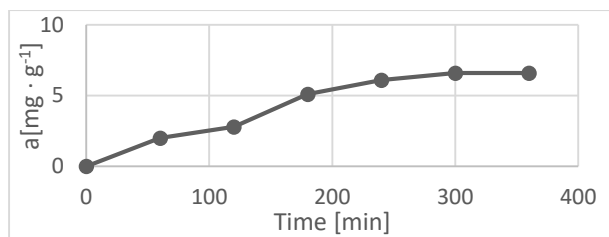


Figure 6. Kinetic course of Ni²⁺ sorption on NH₄⁺ clinoptilolite

The natural zeolite clinoptilolite was also used to experimentally reduce the content of iron cations in an aqueous solution. For this purpose, model samples with a content of ferric cations of 0.48; 0.6; 1.19; 1.4 and 11.8 g·dm⁻³ were used. The process of reducing the number of ferric cations on natural clinoptilolite at different initial concentrations is shown in Figure 7. The measured and calculated parameters are listed in Table 7.

Table 7. Measured and calculated parameters

c_0 [g·dm ⁻³]	C_{eq} [g·dm ⁻³]	a [mg·g ⁻¹]
0.48	0.2	1.75
0.60	0.3	1.88
1.19	0.6	3.69
1.40	0.8	3.75

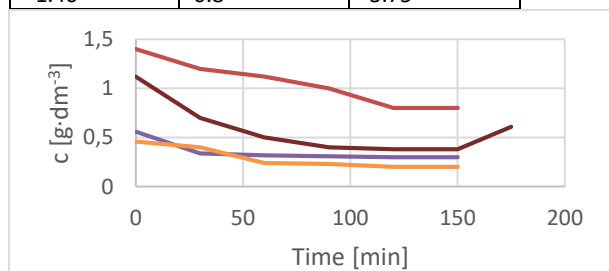


Figure 7. Sorption of ferric cations on clinoptilolite in samples with different initial concentration

5 CONCLUSIONS

The effect of modifying the natural zeolite clinoptilolite into the Fe form was confirmed experimentally. The absorbed amount of copper cations increased slightly, the efficiency of 84% increased to 95%.

Due to the influence of modifying of clinoptilolite into the ammonium form with respect to the sorption of nickel cations, the efficiency increased from 44% to 53%. The kinetic dependences showed that the optimal time for equilibration in the adsorption system is 60 to 70 minutes for the sorption of copper cations, 180 to 240 minutes for the sorption of nickel cations and 60 to 120 minutes for ferrous cations. In most cases, the steady-state concentration was reached after 90 minutes and in one case after 120 minutes. The use of sorption on natural materials is a potential alternative to conventional methods of removing copper, nickel and iron ions, also due to low costs. The search for suitable modifications will be the subject of longer research.

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