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## Preliminary Assessment of Potential Disposal of Mercury-Saturated Natural and Modified Zeolite Clinoptilolite

*Antonija Jurić<sup>1</sup>, Marin Ugrina<sup>2</sup>, Ivona Nuić<sup>3</sup>, Marina Trgo<sup>3</sup>, Teja Čeru<sup>4</sup>, Martin Gaberšek<sup>4</sup>, Miloš Miler<sup>4</sup>, Mateja Gosar<sup>4</sup>*

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### Abstract

Utilization of natural raw materials, such as zeolites, for the remediation of mercury-contaminated environments is desirable. The main issue is the possibility of disposing mercury-saturated zeolite in nature without negative impact on the environment. In order to get an answer to this issue, the sorption properties of natural and modified zeolite were first examined. Two-stage modification of the natural zeolite was performed with aqueous solutions of  $\text{Fe}(\text{NO})_3 \cdot 9\text{H}_2\text{O}$  and  $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ , with detailed physicochemical characterization. At the defined optimal sorption conditions,  $\text{pH}_0 = 2$  and solid/liquid ratio of 10 g/L, the obtained sorption capacity of natural zeolite was 0.282 mmol Hg/g, and of modified zeolite 0.966 mmol Hg/g. Almost 3.5-fold higher sorption capacity of modified zeolite justifies modification method. Leaching experiments confirmed that the modified zeolite retains mercury better than the natural one. The Toxicity Characteristic Leaching Procedure indicated the presence of mercury in leachates above the prescribed value of 0.2 mg/L, pointing that saturated zeolites should be stabilized/solidified before disposal.

**Keywords:** natural zeolite, modified zeolite, sorption, leaching, mercury.

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### 1. INTRODUCTION

The removal of heavy metals from contaminated water or soil systems, particularly extremely toxic mercury, either by *in-situ* or *ex-situ* sorption remediation techniques primarily requires a high sorbent selectivity and capacity. For *in-situ* remediation such as soil sprinkling with sorbent, it is very important that the mercury is stabilized within the sorbent with minimal or no re-sorption, since the sorbent is permanently left in the environment. In case the sorbent is used for *ex-situ* remediation, the saturated sorbent must be adequately disposed of [1], [2]. The most common sorbent for mercury sorption is activated carbon, however, due to its high cost, it is replaced by different natural sorbents such as zeolites [3]. Natural zeolites belong to the group of aluminosilicate minerals. They have a negative charge due to the isomorphous substitution of  $\text{Si}^{4+}$  with

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<sup>1</sup> Regional Veterinary Institute Split, Croatian Veterinary Institute, Poljička Cesta 33, 21000 Split, Croatia

<sup>2</sup> Corresponding author: Faculty of Chemistry and Technology, University of Split, Ruđera Boškovića 35, 21000 Split, Croatia, [mugrin@ktf-split.hr](mailto:mugrin@ktf-split.hr)

<sup>3</sup> Faculty of Chemistry and Technology, University of Split, Ruđera Boškovića 35, 21000 Split, Croatia

<sup>4</sup> Geological Survey of Slovenia, Dimičeva ulica 14, 1000 Ljubljana, Slovenia

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$\text{Al}^{3+}$ , which is compensated by cations ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ). These cations participate in the ion exchange process with cations in sounding media [4]. Regarding the sorption of mercury, sorbents are usually modified with sulphur species such as  $\text{CS}_2$  [5],  $\text{Na}_2\text{S}$  [6],  $\text{K}_2\text{S}$  [7],  $\text{SO}_2$  [8], etc., due to the strong affinity of sulphur to mercury, forming sparingly soluble  $\text{HgS}$ . Since it is known that iron sulphides are the most common scavengers of mercury in nature, impregnation of the surface of some materials such as  $\text{Al}_2\text{O}_3$  [9] or carboxymethyl cellulose [10] with iron sulphide species can be found in literature. Investigations have shown increased sorption properties of stabilized iron sulphide species on the carriers compared to bare iron sulphide particles due to formation of iron sulphides nanoparticles. As far as we know, modification of the zeolite surface with iron sulphide particles has not been performed to date. Therefore, the first aim of this paper is to carry out the modification of natural zeolite with iron sulphides particles. Then, examine the possibilities of using natural zeolite and its modified form for remediation of mercury contaminated environment, with emphasizing the possibility of permanent disposal of saturated zeolites in the environment.

## **2. MATERIALS AND METHODS**

### **2.1. Sample Preparation**

Natural zeolite, NZ originating from the Zlatokop deposit (Vranjska Banja, Serbia), was milled and sieved to a particle size fraction of 0.6–0.8 mm and used as a starting material to obtain its chemically modified form, MZ. Two-stage modification of the NZ was performed according to the procedure described by Schwartzman and Cornell [11] and Liu et. al [12]. The 1 g of NZ was refluxed for 2 h at 100 °C with 50 mL of 1 mol/L  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , and then for 4 h at 150 °C with 10 mL of 1 mol/L  $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ . Thereafter, the sample was washed with ultrapure water until a neutral pH was reached, dried at 40 °C and marked as MZ.

Detailed mineralogical and physicochemical characterization of both NZ and MZ (results are not presented here) showed that the amount of iron increased by 2.5 times, and sulphur by 12 times, indicating the successful modification of the NZ surface with iron sulphide species. The deposition of iron sulphide species on the entire surface of the NZ was confirmed by SEM-EDS analysis, which also revealed that the sodium exchangeable cation dominates on the surface of the MZ as a consequence of modification. XRPD analysis of the NZ revealed that clinoptilolite is the main zeolite mineral (minimum 80 %) as well as a slight decrease in the crystallinity of the MZ. Namely, the first stage of modification in an acidic medium is responsible for the fixation of iron, which also leads to an increase in the specific surface area due to pore opening. In the second stage, fixed iron on the NZ acts as a carrier for stabilization of sulphur species, which is accompanied by a decrease in specific surface area. In addition, the alkaline medium contributes to desilication of the zeolite structure, an increase in the negative charge and basicity of the MZ, which was confirmed by determining the total basicity and zeta potential. The formed negative lattice charge is compensated by sodium cations. Thus, the resulting material should pose improved ion-exchange properties as well as enhanced affinity towards mercury species due to the presence of sulphur in its structure.

### **2.2. Saturation Experiments**

The purpose of the saturation experiments was to find the optimal conditions of pH, solid/liquid (S/L) ratio and mercury concentration at which maximum saturation of the NZ and the MZ is achieved. The chemicals used,  $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ , 1 mol/L  $\text{HNO}_3$  and ultrapure water, were of analytical grade. All experiments were performed in the batch mode, during 24 h, at 230 rpm and at ambient temperature. The concentration of  $\text{Hg}(\text{II})$  was determined on a Flame Atomic Absorption Spectrophotometer PinAAcle 900F, AAS.

Based on the experience of heavy metals sorption on zeolite, a  $\text{Hg}(\text{II})$  solution with an initial concentration of 4.006 mmol/L was prepared to test the effect of  $\text{pH}_0$ . Then, 1 g of NZ or MZ was mixed with 100 mL of the prepared  $\text{Hg}(\text{II})$  solution with a pre-adjusted pH value at 2.09 and 2.20 by addition of 1 mol/L  $\text{HNO}_3$ .

To test the effect of the S/L ratio, a solution of 4.056 mmol  $\text{Hg}/\text{L}$  with pH adjusted to 1.99 was prepared. Different masses of the NZ in the range 0.2–3.6 g ( $\text{S}/\text{L} = 2\text{--}36 \text{ g}/\text{L}$ ) and the MZ in the range 0.2–1.4 g ( $\text{S}/\text{L} = 2\text{--}14 \text{ g}/\text{L}$ ) were mixed with 100 mL of  $\text{Hg}(\text{II})$  solution.

The effect of the initial  $\text{Hg}(\text{II})$  concentration was tested at  $\text{pH} \approx 2$  and  $\text{S}/\text{L} = 10$  based on previously conducted experiments. A mass of 1 g of zeolite was mixed with 100 mL of  $\text{Hg}(\text{II})$  solution, for the NZ in the concentration range 0.461–12.258 mmol  $\text{Hg}/\text{L}$ , i.e. for the MZ in the range 0.461–14.099 mmol  $\text{Hg}/\text{L}$ .

### **2.3. Leaching Experiments**

Based on sorption experiments, the concentration at which the maximum saturation of zeolite with  $\text{Hg}(\text{II})$  is achieved, was selected for leaching experiments. Then the zeolites were saturated as follows: 20 g of zeolite with 2 L of 7.917 mmol  $\text{Hg}/\text{L}$  for NZ and 10.619 mmol  $\text{Hg}/\text{L}$  for MZ, under optimal sorption conditions,

pH=2 and S/L = 10, during 24 h, 25 rpm and ambient temperature. After that, the samples were washed in ultrapure water, dried at 40 °C and marked as NZHg and MZHg.

The leaching of Hg(II) from NZHg and MZHg was carried out according to the standard leaching method, DIN 38414 S4 [13]. A mass of 1 g of saturated zeolites was mixed with 10 mL of ultrapure water with different pH<sub>0</sub> in the range 2.04–12.13, during 24 h, at 25 rpm and ambient temperature. The concentration of leached Hg(II) was determined in the supernatants by means of AAS.

Toxicity Characteristic Leaching Procedure, TCLP was performed with two extraction solutions of pH values of 2.88 and 4.93 prepared according to the detailed procedure described, by mixing 1 mol/L NaOH and/or 0.1 mol/L glacial acetic acid [14]. A mass of 2.5 g of the NZHg or MZHg was leached with 50 mL of extraction solutions during 20 h, at 30 rpm and at ambient temperature. For both leaching experiments, the leached Hg(II) concentration was determined in the supernatants by means AAS.

### **Calculation of Sorption and Leaching Parameters**

The amount of mercury sorbed on zeolites in equilibrium,  $q_e$  (mmol/g), and sorption efficiency in equilibrium,  $\alpha_e$  (%) was calculated by equations (1) and (2):

$$q_e = (c_o - c_e) \cdot \frac{V}{m} \quad (1)$$

$$\alpha_e = \frac{(c_o - c_e)}{c_o} \cdot 100 \quad (2)$$

where  $c_o$  and  $c_e$  are the initial and equilibrium Hg(II) concentration (mmol/L),  $V$  is the volume of the solution (L) and  $m$  is the mass of zeolite (g).

The amount of Hg(II) leached from zeolite,  $q_l$  (mmol/g) and the amount of Hg(II) retained on zeolite,  $q_r$  (mmol/g) was calculated by equations (3) and (4):

$$q_l = c_l \cdot \frac{V}{m} \quad (3)$$

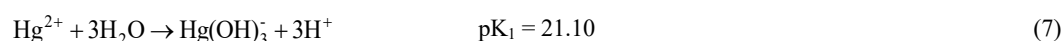
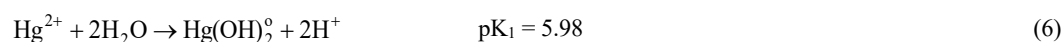
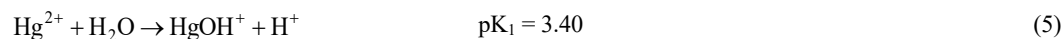
$$q_r = q_e - q_l \quad (4)$$

where  $c_l$  is the leached Hg(II) concentration from the zeolite (mmol/L).

## **3. RESULTS AND DISCUSSION**

### **3.1. Determination of Optimal Sorption Conditions for Saturation of Natural and Modified Zeolite**

The pH is a key factor in sorption processes since it directly affects the type of metal species as well as the sorbents surface charge. Based on the mercury hydrolysis constants represented by reactions (5)-(7) [15], fractions of mercury species depending on pH were constructed and shown in Figure 1.



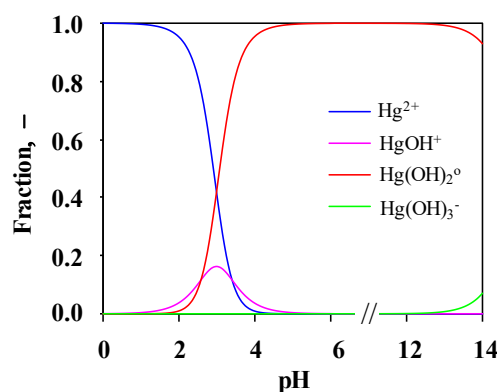


Figure 1. Mercury speciation diagram.

According to the speciation diagram, up to  $\text{pH} = 2.9$ , the dominant species is  $\text{Hg}^{2+}$ , after that the fraction of the  $\text{Hg}(\text{OH})_2^\circ$  species increases, which becomes predominant with further increase in pH. The  $\text{HgOH}^+$  species reaches a maximum of 20 % at  $\text{pH} = 3$ , while the  $\text{Hg}(\text{OH})_3^-$  species appears only above  $\text{pH} = 13.2$  with a maximum fraction of 7% at  $\text{pH} = 14$ . Since the precipitation of mercury starts at  $\text{pH} = 2.45$ , this suggests that the sorption of  $\text{Hg}(\text{II})$  onto the zeolite must take place in a very narrow range of  $\text{pH } 2.00 < \text{pH} < 2.45$  in order to completely exclude the precipitation as well as the decomposition of the zeolite structure ( $\text{pH} < 2$ ).

The comparison of equilibrium and initial pH values ( $\text{pH}_e$  vs.  $\text{pH}_o$ ) after sorption of  $\text{Hg}(\text{II})$  on the NZ and MZ is shown in Figure 2a, as well as sorption efficiency in Figure 2b.

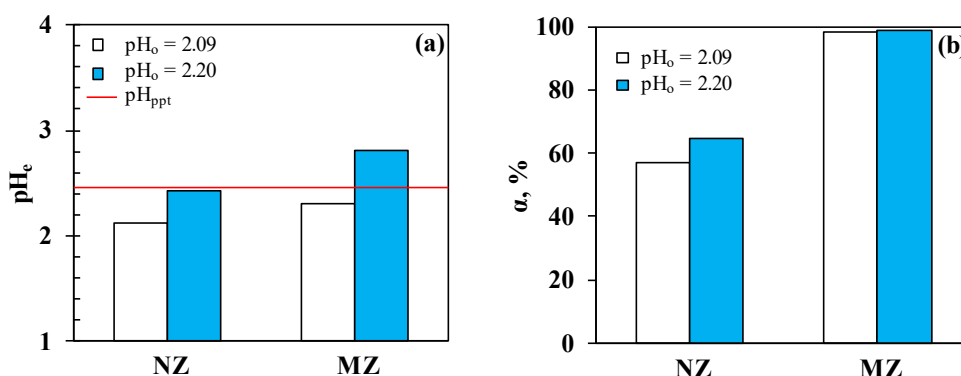


Figure 2. (a)  $\text{pH}_e$  for NZ and MZ at  $\text{pH}_o = 2.09$  and 2.20 (red line represents the precipitation pH,  $\text{pH}_{\text{ppt}}$ ). (b) Sorption efficiency of  $\text{Hg}(\text{II})$  at  $\text{pH}_o = 2.09$  and 2.20 for NZ and MZ.

Figure 2a shows a significant increase in  $\text{pH}_e$  for MZ compared to NZ, which is probably a consequence of the competitive effect of  $\text{Hg}(\text{II})$  and  $\text{H}^+$  ions in an acidic medium, which is more pronounced for MZ due to a higher negative surface charge. It is also noted that at  $\text{pH}_o = 2.09$ , the  $\text{pH}_e$  values for both, NZ and MZ are lower than the critical pH ( $\text{pH}_{\text{pt}}$ ) at which precipitation occurs. At  $\text{pH}_o = 2.20$ ,  $\text{pH}_e$  values for NZ are very close to the  $\text{pH}_{\text{ppt}}$ , while for MZ it is above  $\text{pH}_{\text{ppt}}$  which confirms precipitation. Therefore, these results are not taken into consideration since in this case, sorption efficiency is the contribution of both  $\text{Hg}(\text{II})$  precipitation and sorption. Taking all into account,  $\text{pH} = 2.09$  is chosen as optimal, at which the sorption efficiency of 56.94 % for NZ and 99.06 % for MZ was achieved (Figure 2b). Almost 2-fold sorption efficiency of  $\text{Hg}(\text{II})$  on the MZ than that NZ justifies the zeolite modification.

Sorption efficiency,  $\alpha$  and  $\text{pH}_e$  as a function of S/L ratio, after  $\text{Hg}(\text{II})$  sorption on the NZ and MZ are shown in Figure 3.

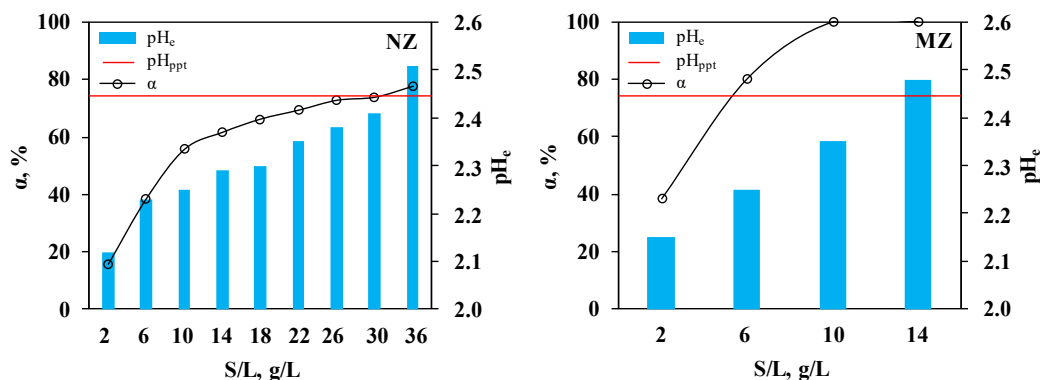


Figure 3. Comparison of  $pH_e$  and  $\alpha$  with S/L ratio after Hg(II) sorption on NZ and MZ (red line represents the precipitation pH,  $pH_{ppt}$ ).

Figure 3 shows a gradual increase in sorption efficiency for NZ and a sudden increase for MZ with an increase in the S/L ratio as a result of a higher zeolite mass, i.e. a higher number of active sorption sites. The twice higher sorption efficiency for MZ than for NZ at each S/L ratio is attributed to a higher negative surface charge and a higher number of active sorption sites. An increase in sorption efficiency is accompanied by an increase in  $pH_e$ , which is more pronounced for MZ. The increase in  $pH_e$  is the result of Hg(II) removal and competition of Hg(II) with  $H^+$  ions, i.e. ion exchange of  $H^+$  ions with zeolite exchangeable cations ( $Na^+$ ,  $K^+$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ). At the S/L ratio of 36 g/L for NZ and 14 g/L for MZ, the  $pH_e$  exceeds the  $pH_{ppt}$  value indicating the Hg(II) precipitation in the suspension. Therefore, S/L of 10 g/L for MZ was chosen as optimal where maximum sorption efficiency was obtained without precipitation. Although the optimal S/L ratio for NZ is 30 g/L, the optimal S/L of 10 g/L was chosen due to the negligible increase in sorption efficiency for S/L from 10 to 30 g/L, as well as the comparison of results at the same ratio of S/L to investigate the effect of initial concentration.

As stated, under pre-defined optimal conditions, pH = 2 and S/L = 10 for both samples, the effect of the initial Hg(II) concentration was tested and shown in Figure 4.

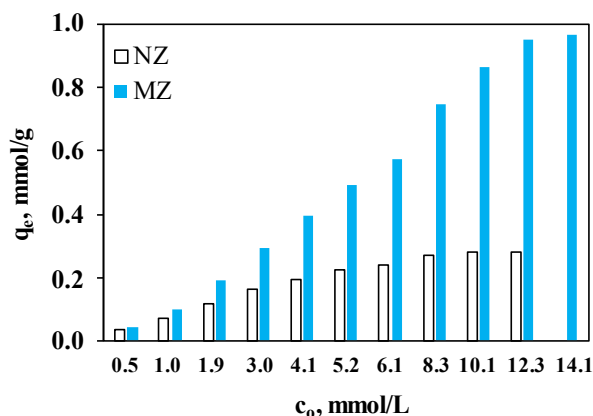


Figure 4. The influence of the initial Hg(II) concentration on the quantity of sorbed Hg(II) on NZ and MZ.

With the increase in the initial concentration of Hg(II), a drastic difference in the quantity of sorbed Hg(II) is observed on MZ compared to NZ. Namely, according to the hard and soft acid and base theory, it is well known that mercury as a soft Lewis acid has an exceptional affinity for sulphur, which is a soft Lewis base, therefore  $Na_2S$  has been selected as a modifying reagent with  $Fe(NO)_3 \cdot 9H_2O$  which stabilizes sulphur species. According to the physicochemical characterization, the increase of newly formed active sites containing sulphur species as well as negative charge contributes to the increase of sorption capacity. The 3.5 times higher amount of sorbed Hg(II) on the MZ compared to NZ (0.996 mmol/g vs. 0.288 mmol/g) justifies the implementation of the modification.

### 3.2. Assessment of Disposal of Mercury-saturated Natural and Modified Zeolite

In order to assess the possibility of hazardous mercury-saturated zeolite disposal in the environment after remediation, it is necessary to examine its leaching properties. The results of the leaching test of NZHg and MZHg in ultrapure water of different  $pH_0$  conducted according to the standard leaching method DIN 38414 S4 [13] are shown in Figure 5.

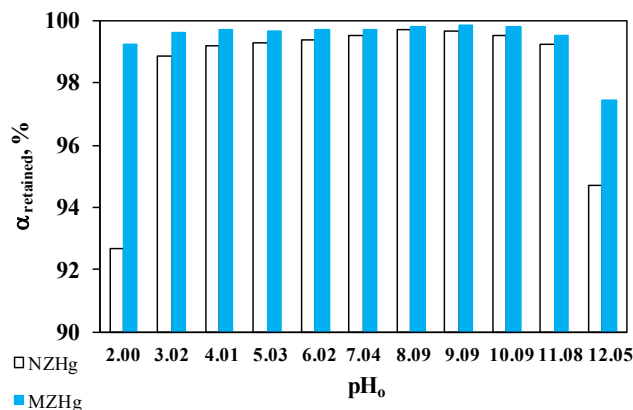


Figure 5. The percentage of retained Hg(II) after the leaching tests of NZHg and MZHg vs.  $pH_0$ .

At all tested  $pH_0$ , the percentage of retained mercury is higher on MZ compared to NZ, which is probably a consequence of the stronger affinity of MZ to Hg(II), i.e. stronger binding of Hg(II) to sulphur species. This is particularly pronounced under extremely acidic and alkaline conditions,  $pH = 2.00$  and  $pH = 12.05$ . Namely, under acidic conditions, dealumination of the zeolite structure occurs, and under alkaline conditions desilication, i.e. degradation of the zeolite lattice. Since the two-stage modification was carried out in an acidic medium with  $Fe(NO)_3$  and then in an alkaline medium with  $Na_2S$ , re-exposure of the zeolite to a strongly acidic or alkaline medium has a smaller effect on MZ compared to NZ. This is probably one of the reasons for the lower percentage of Hg(II) retention in NZ compared to MZ beside the Hg(II) binding mechanism. At  $3.02 \leq pH_0 \leq 11.08$ , the percentage of retained Hg(II) in NZ is in the range 98.88 %–99.71 %, while for MZ in the range 99.53 %–99.87 %, respectively. The results indicate that in a wide pH range, which can be expected in the environment, both zeolites show a significant ability to retain Hg(II).

Since the standard DIN method does not provide a value for the percentage of Hg retained or leached to evaluate the disposal of saturated zeolites, an additional test was performed to provide an evaluation, the toxicity characteristic leaching procedure, TCLP. The percentage of retained Hg(II) after leaching NZHg and MZHg at  $pH = 2.88$  and  $pH = 4.93$  is shown in Figure 6.

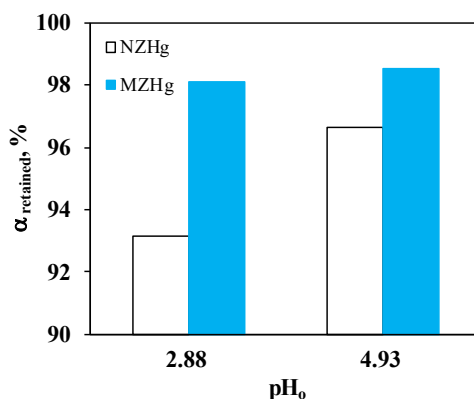


Figure 6. Percentage of retained Hg(II) after TCLP test on NZHg and MZHg at  $pH = 2.88$  and  $pH = 4.93$ .

At both pHs, the percentage of retained Hg(II) on MZ is higher than that on NZ, which is in accordance with the results obtained by the standard DIN method. It should be noted that slightly less Hg(II) was retained after leaching according to the TCLP procedure, which is probably a consequence of the stronger ionic strength of the solutions. A total of 93.13 % of the mercury was retained on NZ and 98.10 % on MZ at  $\text{pH}_0 = 2.88$ , while at  $\text{pH}_0 = 4.93$  these values were 96.63 % for NZ and 98.10 % for MZ, indicating fairly good retention properties. Since the TCLP test prescribes that the concentration of leached Hg must not exceed a value of 0.2 mg/L, the results are expressed as follows: 190.8 mg/L of the mercury was leached from the NZHg and 162.5 mg/L from the MZHg at  $\text{pH}_0 = 2.88$ , while at  $\text{pH}_0 = 4.93$  these values were 93.5 mg/L for NZ and 126.9 mg/L for MZ.

Considering the results of both tests, MZ has a higher Hg(II) retention ability compared to NZ, higher than 98%. This means that it could eventually be used for *in-situ* remediation, since a greater benefit would be achieved than the negative impact on the environment due to partial leaching of 2 %, which is greater than 0.2 mg/L. On the other hand, according to the TCLP procedure, the concentrations of leached Hg(II) at both tested pH values significantly exceed the prescribed value of 0.2 mg/L, indicating that both zeolites can be used for *ex-situ* remediation. Saturated zeolites should not be disposed of in the environment without prior solidification/stabilization. The possible solution could be found by adding saturated zeolites to the building materials, which requires upgrading for future investigations.

#### 4. CONCLUSION

The modification contributed to a 3.5-fold increase in the quantity of sorbed Hg(II) on MZ compared to the starting material, justifying the zeolite modification. Since the modified zeolite has a significantly higher ability to bind and retain Hg(II) compared to the natural one, this makes it a potential sorbent for the remediation of mercury-contaminated environments. It is preferable to use both zeolites for *ex-situ* remediation with appropriate stabilization/solidification before disposal in the environment.

#### ACKNOWLEDGMENT

This work was supported by the bilateral Croatian-Slovenian project Natural Modified Sorbents as Materials for Remediation of Mercury Contaminated Environment (2020–2023), founded by the Croatian Ministry of Science and Education and Slovenian Research Agency (ARRS) and by the Slovenian Research Agency (ARRS) in the framework of the research program Groundwater and Geochemistry (P1-0020).

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## BIOGRAPHY



**Antonija JURIĆ** works as a professional associate at the Croatian Veterinary Institute (Laboratory for analytical chemistry and residues). She graduated at the Faculty of Chemistry and Technology, University of Split, Croatia.

She can be contacted at [aantonija.juric@gmail.com](mailto:aantonija.juric@gmail.com)