

## Removal of $\text{Co}^{2+}$ and $\text{Ni}^{2+}$ Ions from Aqueous Solutions by Low Cost Synthetics Na-4-mica

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### Authors' contributions

This work was carried out in collaboration between both authors. Author FSH designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Author ZAA did the experimental parts and managed the analyses of the study. Authors FSH and ZAA managed the literature searches. Both authors read and approved the final manuscript.

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### ABSTRACT

A high charged density sodium fluorophlogopite mica Na-4-mica, ideal chemical structure  $\text{Na}_4\text{Si}_4\text{Al}_4\text{Mg}_6\text{O}_{20}\text{F}_4 \cdot x\text{H}_2\text{O}$ , was prepared by a simple and economic method using kaolinite as a source for  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{MgO}$ . The prepared mica was characterized by using XRD, DSC and SEM. Na-4-mica was used for removal of  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions from aqueous solutions using a stepwise remediation method. The factors affecting the removal efficiency such as pH change during the exchange reaction, particle size of Na-4-mica, initial metal concentration were investigated. The results showed the removal efficiency increased by decreasing the particle size of the mica and initial concentration of the heavy metal in the solution. Na-4 mica showed removal efficiency for  $\text{Ni}^{2+}$  ions higher than that for  $\text{Co}^{2+}$  ions. This related to the difference in the hydrated ions radii between  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions.

**Keywords:** Na-4-mica; removal; heavy metals;  $\text{Co}^{2+}$ ;  $\text{Ni}^{2+}$  ions; water pollution; water treatment.

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## 1. INTRODUCTION

The contamination by heavy metals species such as, Cr, Co, Ni, Cu, Zn, Cd, Hg, and Pb has resulted from various sources such as landfill, agricultural activities, and industrial wastewater [1,2]. These heavy metal species are considered as toxicologically interesting elements. Among the above heavy metals, Cobalt and nickel have received considerable attention because of their geochemical behavior and industrial uses [3]. Cobalt has high affinity for iron and manganese oxides and phyllosilicate minerals [4]. On the other hand, nickel is often detected in soils because there are many nickel-bearing silicate [5,6]. Both nickel and cobalt have been widely used as industrial catalysts. Nickel has been used in mining, electroplating, pigment, or battery industry [7] and cobalt has been used in electroplating, pigment, or metal alloy industry [8]. Many inorganic ion exchange materials such as micas, zeolites, and synthesized inorganic materials can be used for treatment of heavy metals in industrial wastewater.

Micas are one of the most interesting groups of ion exchange materials because of their high layer charge and radiation stability. The two main categories of micas based on layer charge density are: (1) true micas with 1 negative charges per formula unit and (2) brittle micas with 2 negative charges per formula unit. Owing to their high charge density, naturally occurring micas do not normally swell in water and other polar solvents; as a result, exchange reactions with the interlayer cations do not readily occur. Synthetic micas, which swell in water, with cation exchange capacities of more than 200 mequiv (100 g)<sup>-1</sup>.

The synthetic clay mineral, "Na-4-mica" contains four Na<sup>+</sup> ions per unit cell and has a theoretical cation exchange capacity of 468 meq/100 g on an anhydrous basis [9,10]. This synthetic clay mineral, ideally Na<sub>4</sub>Si<sub>4</sub>A<sub>14</sub>Mg<sub>6</sub>O<sub>20</sub>F<sub>4</sub>.xH<sub>2</sub>O, has a high A1 content but exhibits unusual swelling behavior and selective cation exchange properties potentially useful in hazardous cation separations for waste disposal or metal recovery from dissolved ores [11,12]. The unusual swelling behavior was attributed to the high number of interlayer Na<sup>+</sup> ions per unit cell, which are accommodated in the ditrigonal holes by an offset layer stacking of *b*/3.

Many researchers developed low cost-effective methods to prepare Na-4-mica such as

hydrothermal synthesis [13], topotactic exchange [14] and solid-state methods. However, the raw materials used were natural materials such as talc, kaolinite [15,16], diatomite [17] and fly-ash [18]. In the present work we synthesis Na-4-mica from kaolinite as a low source for alumina and silica. The selective exchange properties of Co<sup>2+</sup> and Ni<sup>2+</sup> with Na-4-mica for potential treatment of groundwater contaminated with heavy metal was investigated.

## 2. EXPERIMENTAL

### 2.1 Materials

Naturally occurring kaolinite, MgO, CaF<sub>2</sub> and NaF were the starting materials for the synthesis of Na-4-mica. Co (NO<sub>3</sub>) and Ni(NO<sub>3</sub>) were used as a source of Co<sup>2+</sup> and Ni<sup>2+</sup> ions. The chemical oxides composition of kaolinite were 49.5%, 35.22%, 1.05%, 0.2%, 0.36%, 0.6% and 0.19% for SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, SO<sub>3</sub> and K<sub>2</sub>O respectively. All chemical used were SIGMA\_ACS grade and were used as received without any further purification procedure.

### 2.2 Preparation of Na-4-mica

Na-4 mica was prepared as follows [14]: kaolinite was first calcinated at 700°C for 3 hours to transform it to amorphous product (on heating to around 600°C, kaolinite is completely dehydrated and loses its crystalline character) which is the so-called metakaolin (Al<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>). Metakaoline was used as a source for Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with molar ratio of 1:2. Metakoline then mixes with MgO to obtain a stoichiometric composition of the mixture, which when reduced to pure oxides was exactly 3MgO–Al<sub>2</sub>O<sub>3</sub>–2SiO<sub>2</sub>. The mixture was mixed with an equal mass of NaF and heated for 12 hours at 890°C in an air-atmosphere furnace. The resulting solid was ground up and washed in deionized water several times to remove the remaining NaF. The remaining of impurity phases that are not water soluble, which may include insoluble fluoride salts, was then removed with repeated washings with a saturated boric acid solution. Then, the solid was washed with NaCl (1 molar) solution three times to completely saturate all the exchange sites with Na<sup>+</sup>. The product was again washed with deionized water and dried at 60°C in an oven for 2-3 days.

### 2.3 Characterization of Na-4-mica

The characterization of resulting Na-4-mica was examined using X-ray diffraction (XRD),

Differential Scanning Calorimetry (DSC) and Scanning Electron Microscope (SEM). For X-ray examination; a stabilized X-ray generator was used fitted with copper target and nickel filter under working conditions of 40 kV and 25 mA. Differential scanning calorimetry runs were conducted using a Shimadzu DSC-50 thermal analyzer at a heating rate of 20°C/min. The sample chamber was purged with nitrogen at a flow of 30 ml/min. For SEM examination, a freshly fractured surface of the dried sample coated with a thin film of gold (about 300-400 x 10<sup>-6</sup> m thickness) using a vacuum evaporation technique. A JEOL-JSM-5400 high resolution scanning electron microscopy was used (Shimadzu Co., Japan).

## 2.4 Cation Exchange Process

### 2.4.1 Effect of particle size

The prepared Na-4 mica was divided into two groups of different particle sizes by sieving with a specific meshes. The first group of mica, which called M1, of particle size < 0.125 mm. The 2<sup>nd</sup> group of particle size > 0.125 mm which called M2. 1.5 g of mica samples, M1 and M2, were weighed to packed different 200 mm high, 10 mm diameter glass columns. Then 50 ml containing 1000 ppm of heavy metal of ions (Co<sup>2+</sup> and Ni<sup>2+</sup> ions), of initial pH 4 and at 25°C, were added and the column was kept close for 1 hour until complete matrix humidification. After that, the column was open to allow elution flow. From the elution, sample (2.0 ml) was obtained, its pH was measured with pH meter (Orion Research Inc.), then labeled and stored for analysis. The rest of the elution was reloaded into the column and the pervious step repeated to test the amount of metal removed and the pH change after more than one elution.

### 2.4.2 Effect of initial concentration of heavy metal solution

In this process, 1.5 g of M1 mica sample, particle size < 0.125 mm, was packed in different glass column with 50 ml containing 100, 500, 1000 and 3000 mg/L of heavy metal of ions (Co<sup>2+</sup> and Ni<sup>2+</sup> ions), for one hour. Then the column was open and the elution was allowed to flow. The elution was reloaded again into the column and the pervious step repeated to 2<sup>nd</sup> and 3<sup>rd</sup> solution loading cycle. The concentration remaining of each heavy metal in the elution in each solution loading cycle was measured using atomic absorption spectroscopy (AAS).

## 2.5 Analytical Tests

Metal ion concentrations in different elutions after 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> load were performed by atomic absorption spectroscopy (AAS) using a Perkin-Elmer, PE 703 instrument. The removal efficiency (Q) was calculated using equation 1:

$$Q \% = (C_0 - C_t) / C_0 \cdot 100 \dots\dots\dots (1)$$

Where: Q is the of the removal efficiency and C<sub>0</sub> and C<sub>t</sub> is the concentration of the heavy metal ion in solution before and after solution loading cycle process (mg/L).

## 3. RESULTS AND DISCUSSION

### 3.1 Characterization of the Na-4-mica

#### 3.1.1 X-ray diffraction

Fig. 1 shows the X-ray diffraction of Na-4-mica prepared from metakaoline as a source of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> and MgO. In the pattern, the strongest peak observed at 2θ = 7.35° which may be attributed to the symmetrical basal [001] reflection for the hydrated Na-n-(Mica) (n = 4). This peak being more broader and its intensity decreased when the mica is soaked with Co<sup>2+</sup> and Ni<sup>2+</sup> solution, Fig 1b,c. This can be related to the slight splitting and deformation in the layer structure of the mica as a results of exchange of Na<sup>+</sup> ions by Co<sup>2+</sup> or Ni<sup>2+</sup> ions.

#### 3.1.2 SEM

SEM micrographs of Na-4-micas synthesized from the meta-kaolinite precursor are given in Fig. 2. Hexagonal crystals of size ranges between 1-5 μm, can be detected in the micrographs. SEM micrographs also showed presence of a small amount of glassy phase on the edges of the particles. These glassy phases represented the hydrated phase of Na-4 mica.

#### 3.1.3 DSC

Fig. 3 shows DSC curve for the prepared Na-4 mica. Four endothermic peaks can be observed DSC curve. The first endothermic peak located between 85-110°C which related to the physically adsorbed water and interlayer water. This is followed by 2<sup>nd</sup> endothermic peaks located between 230-260°C which due to the removal of interlayer anion and dehydroxylation of the hydroxyl layer. The third peak which represents

the major weight loss was located at 400-420°C and was related to the combustion of the relatively less volatile, metal oxide. The fourth peak located at 950°C and represent the deformation of the structure of Na-4-mica.

### 3.2 Cation Exchange Process

#### 3.2.1 pH change

Table 1 shows initial and final pH values of  $\text{Co}^{2+}$  solutions packed with M1 and M2 sample after 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> solution loading cycle. Initial pH of  $\text{Co}^{2+}$  solutions were kept at 4 to prevent the precipitation of the heavy metal as hydroxides. From the results of (Table 1), the final pH increases after 1<sup>st</sup> to be 4.5-6.1 depending on the initial concentration of  $\text{Co}^{2+}$  ions. This increase in the final pH continues in the 2<sup>nd</sup> load. In the 3<sup>rd</sup> load there is a slight increase in the final pH value compared to these obtained in the 2<sup>nd</sup> load to be in the range 5.1-6.9. M1 mica showed higher increase in the final pH than M2 mica sample. Such results indicate the occurrence of  $2\text{Na}^+ \rightarrow \text{Co}^{2+}$  exchange reaction which increases with increasing the concentration of  $\text{Co}^{2+}$  ions in

the adding solution and decreases by increasing the particle size of the mica. Such results agree with those of Elsabawy k. et al. [16] which showed the power responsible for remediation and removal of trace elements not only cation exchanging of Na-4-mica but also molecular sieving phenomenon within silicate structure of mica.

This increase of pH of the solution during  $2\text{Na}^+ \rightarrow \text{Co}^{2+}$  exchange reaction may be due to buffering capacity of the Na-4-mica. During the  $2\text{Na}^+ \rightarrow \text{Co}^{2+}$  exchange process, more  $\text{Na}^+$  ions was released which consumes  $\text{H}^+$  ions in solution by the reaction ( $\text{R-Na} + \text{H}_2\text{O} \rightleftharpoons \text{RH} + \text{Na}^+ + \text{OH}^-$ ), which leads to higher pH value. According to the obtained values of the final pH during the three cycles of loading, most of the exchange reactions occurred during the 1<sup>st</sup> and 2<sup>nd</sup> cycles and the exchanged solutions still in the acidic range and no precipitation of cobalt hydroxide occurred even in Co solution with the highest concentration, 3000 mg/L.  $\text{Co}(\text{OH})_2$  precipitation is expected to start at  $\text{pH} = 8.36$ .

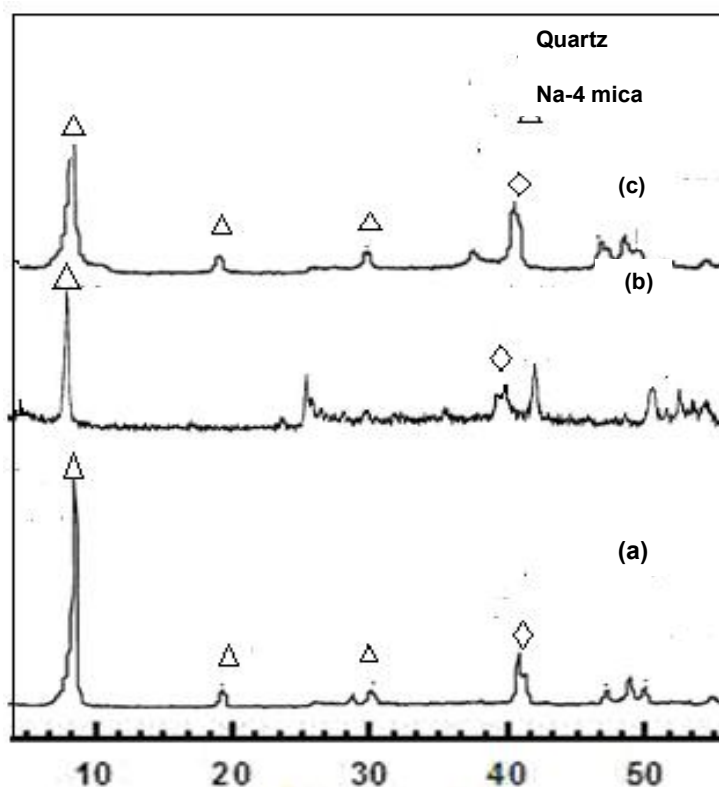


Fig. 1. X-ray diffraction of (a) Na-4 mica (b) mica equilibrated with  $\text{Ni}^{2+}$  solution (c) mica equilibrated with  $\text{Co}^{2+}$  solution

The values of final pH of Ni<sup>2+</sup> solution is represented in (Table 2). Similarly, the final pH increased in all equilibrium solutions after the 2Na<sup>+</sup> → Ni<sup>2+</sup> exchange reactions and has the same trend during the three cycles of solutions loading. However, with increasing Ni<sup>2+</sup> concentration, the difference between initial and final pH values was smaller than these observed for Co<sup>2+</sup> ions in the three loading steps. Also, according to the obtained pH values, Ni<sup>2+</sup> is the predominant species and there is no precipitation of Ni (II) during the exchange process.

### 3.2.2 Effect of particle size

Fig. 4 shows the change of percentage of removal efficiency, Q%, of M1 and M2 mica samples for Co<sup>2+</sup> ions after 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> load. Each data point was obtained in triplicate runs and represents the average of a mean variation of less than ± 5%. According to results in Fig. 4, the percentage of removal efficiency increases with the increasing the number of loading cycle to be in the 3<sup>rd</sup> load 64% and 45% for M1 and M2 respectively. There is a slight difference between values of Q% in the 2<sup>nd</sup> load and in the 3<sup>rd</sup> load. This indicates the equilibrium state nearly reached in the 2<sup>nd</sup> cycle of solutions loading in the two mica samples. M1 mica sample showed higher values of percentage of removal coefficient than M2 and this was more obvious in the 2<sup>nd</sup> and 3<sup>rd</sup> load than in the 1<sup>st</sup> load. These

can be related to the difference in particle size between the two mica samples. In the case of particles with small size, here M1 mica sample, there are more edge sites when compared to larger particles. In the initial stage of ion exchange process, the great number of edge sites of the small particle fraction may take up heavy metal ions rapidly. On the other hand, larger particles may have relatively small number of edge sites compared to small particles. As a consequence, the larger particles have the longer diffusion path from outside edges to center in each layer. These longer diffusion path decrease the 2Na<sup>+</sup> → M<sup>2+</sup> exchange reaction.

The two mica samples showed higher removal efficiency for Ni<sup>2+</sup> ions than Co<sup>2+</sup> ions, see Fig. 5, and these noticed in the three loading cycles. This indicates 2Na<sup>+</sup> → Ni<sup>2+</sup> exchange reaction occurred more than 2Na<sup>+</sup> → Co<sup>2+</sup> reaction which is unexpected results since cobalt solutions results in higher increase in the final pH than nickel solutions. Such results can be related to the difference in the hydrated radii of the cobalt (4.23 Å) and nickel (4.04 Å) ions and the exchanged ion (Na<sup>+</sup>). The ion with smaller hydrated radii exchanged faster than that of higher radii and its enter regularly in the crystal lattice of the mica results in less number of released Na<sup>+</sup> ions and in sequence less final pH values.

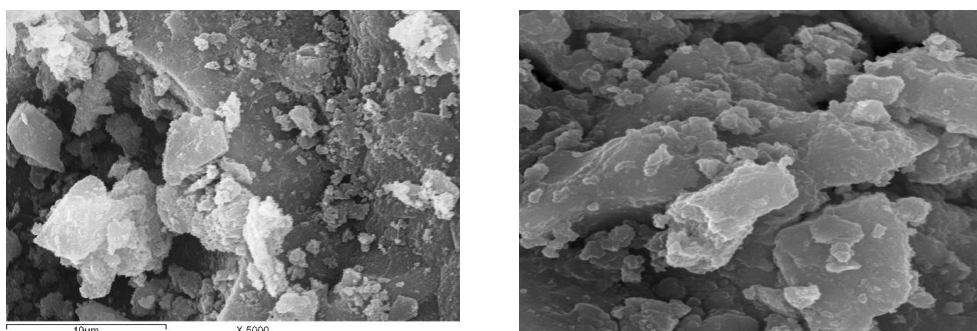


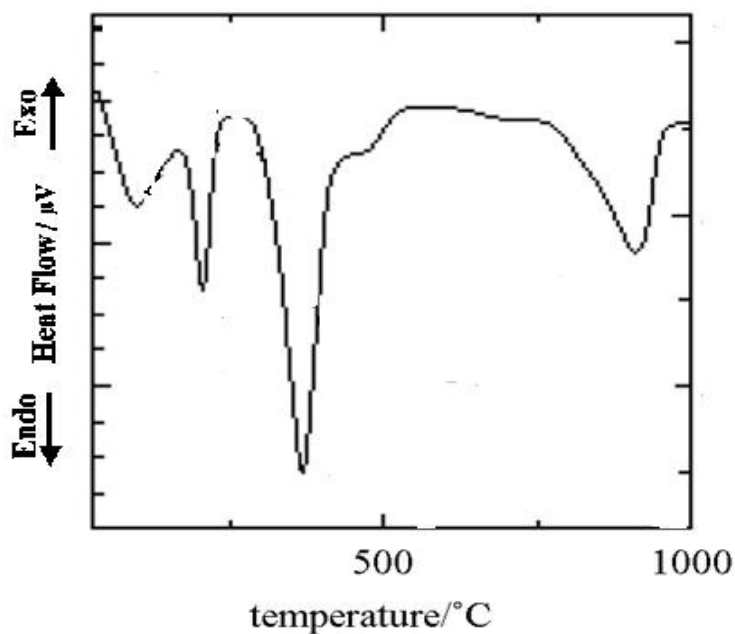
Fig. 2. SEM of Na-4 mica

Table 1. pH change of Na-4- mica equilibrated at different concentration of Co<sup>2+</sup> ion solutions

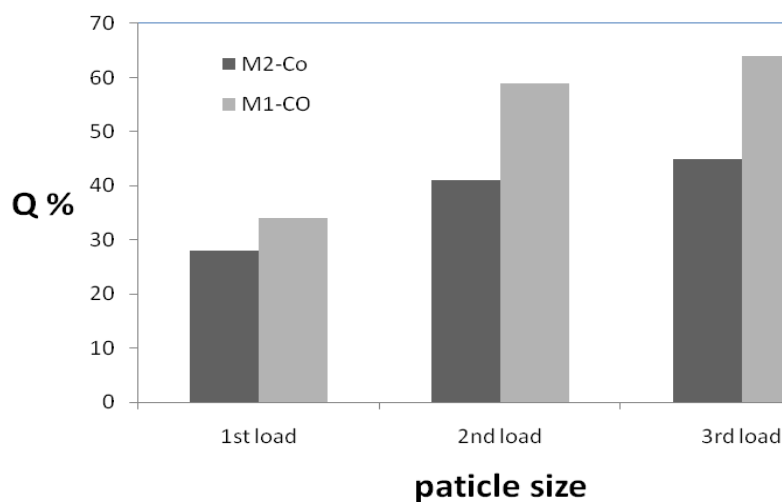
Concentration of M <sup>2+</sup> (ppm)	Initial pH	Final pH					
		Co <sup>2+</sup>					
		M 1			M2		
		1 <sup>st</sup> load	2 <sup>nd</sup> load	3 <sup>th</sup> load	1 <sup>st</sup> load	2 <sup>nd</sup> load	3 <sup>th</sup> load
100		4.5	4.9	5.1	4.3	4.5	4.7
500		5.1	5.6	5.7	4.6	4.8	5.0
1000	4	5.7	6.3	6.5	5	5.3	5.5
3000		6.1	6.6	6.9	5.3	5.5	5.8

**Table 2. pH change of Na-4- mica equilibrated at different concentration of Ni<sup>2+</sup> ion solutions**

Concentration of M <sup>+2</sup> (ppm)	Initial pH	Final pH					
		Ni <sup>2+</sup>					
		M 1			M2		
		1 <sup>st</sup> load	2 <sup>nd</sup> load	3 <sup>th</sup> load	1 <sup>st</sup> load	2 <sup>nd</sup> load	3 <sup>th</sup> load
100	4	4.3	4.7	4.9	4.2	4.4	4.6
500		4.8	5.2	5.3	4.4	4.7	5.0
1000		5.2	5.6	5.9	4.7	5.1	5.3
3000		5.6	5.9	6.2	5.1	5.4	5.7



**Fig. 3. DSC curve of Na-4mica**



**Fig. 4. Change of percentage of removal coefficient, Q, of M1 and M2 mica samples for Co<sup>2+</sup> ions after 1st, 2<sup>nd</sup> and 3rd load**

### 3.2.3 Effect of heavy metal concentration

The effect of the concentrations of  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions on the percentage of the removal efficiency of M1 mica are shown in (Figs. 6 and 7). Both the two heavy metal ions showed percentage of removal efficiency which decreased continually with increasing the concentration of the ions in the solution. It is clear that about 98% and 99% of  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions were removed in the solution of initial concentration 100 mg/L after 3<sup>rd</sup> load while 61% and 68% were recorded for  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$  ions of initial concentrations 3000 mg/L respectively, see Figs 6 and 7. These can be related to decrease in the diffusion velocity of the

metal ion by increase its concentration [19]. In addition, by increasing the heavy metal concentration in the solution the driving force for the exchange (concentration of the heavy metal ion / concentration of the exchangeable sites) decreased [20]. This decrease in the removal efficiency with metal concentrations was more obvious in the elutions of 2<sup>nd</sup> load than those of the 1<sup>st</sup> load. This reveals the equilibrium state reached in the stage of 2<sup>nd</sup> load rather than in the 1<sup>st</sup> load. This behavior has been related to the difficulty of the cations to diffuse into the narrow interlayer space at the beginning of the exchange process or to the fact that edges could collapse at the initial exchange process.

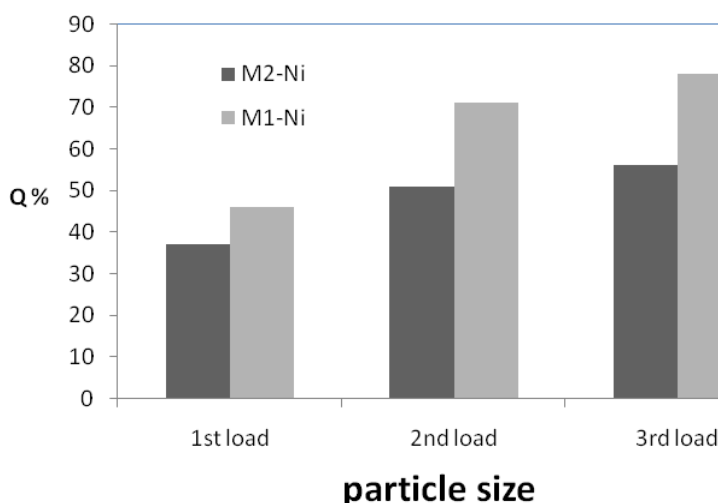


Fig. 5. Change of percentage of removal coefficient, Q, of M1 and M2 mica samples for Ni<sup>2+</sup> ions after 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> load

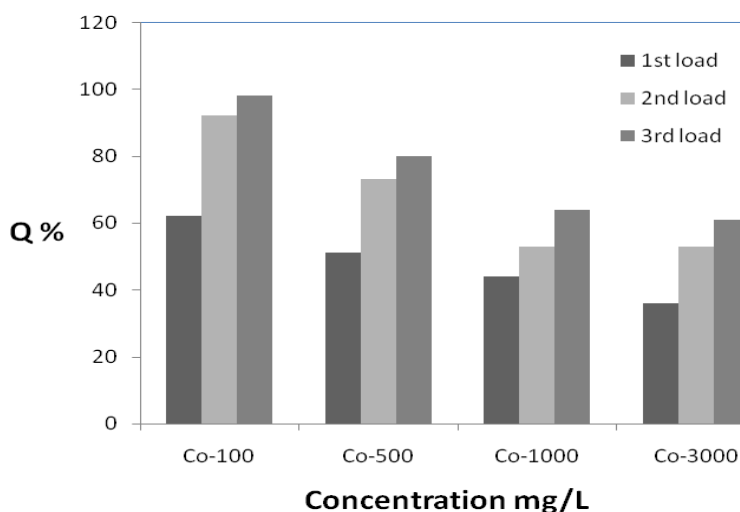


Fig. 6. Change of percentage of removal coefficient, Q, of M1 mica sample for Co<sup>2+</sup> ions with concentration

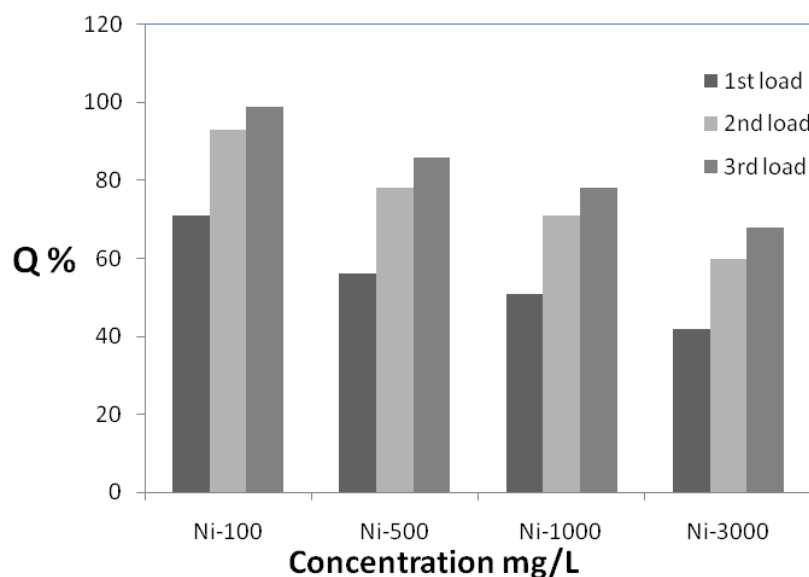


Fig. 7. Change of percentage of removal coefficient, Q, of M1 mica sample for Ni<sup>2+</sup> ions with concentration

#### 4. CONCLUSION

Na-4 mica used for treatment of Co<sup>2+</sup> and Ni<sup>2+</sup> ions from aqueous solutions using loading cycles on separation columns. Final pH of the treatment solutions increased slightly after each successive load but still in the acidic range reveals no precipitation of the metal ions occurred as hydroxides. The removal efficiency decreased by increasing both the particle size of the Na-4 mica and the concentration of the heavy metal ions in the solutions and increased by increasing the number of the loading cycle. According to the percentage of the removal efficiency, the equilibrium state reached during the 2<sup>nd</sup> loading cycle. Na-4 mica showed higher removal efficiency for Ni<sup>2+</sup> ions than Co<sup>2+</sup> ions which related to the difference in the hydrated radii of the removed ions.

#### COMPETING INTERESTS

The authors have declared that no competing interests exist.

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