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NEW ADSORBENTS DEVELOPED FROM NATURAL CLAYS TO REMOVE NI (II) FROM WASTEWATER

Abstract: Treatment of wastewater by adsorption is one of the most environmentally friendly methods, along with high efficiency. The main criteria when choosing materials for wastewater treatment by adsorption are its adsorption properties, porous structure and costeffectiveness. Heavy metal ions are the main pollutants present in wastewater from machine-building, non-ferrous metallurgy and instrument-making enterprises, its removal being mandatory employing processes such as adsorption. This work deals with the development of materials based on natural clays and on pillared clays and further application in the the adsorption of Ni (II), used as model pollutant, from aqueous solution. The starting natural clay (montmorillonite) was obtained in a deposit of Kazakhstan (Asa), and two additional samples were prepared by treatment of the natural clay with acid and by pillarization with Fe^{2+} and Zn^{2+}. Batch adsorption experiments were carried out to explore the effect of pH, adsorbent load, contact time and the initial ion concentrations of Ni (II) in the adsorption of the heavy metal from aqueous solution with the three adsorbent samples. A maximum removal of Ni (II) of 98.9% was achieved at pH 10 with a dose of adsorbent of 2 g/L, a contact time of 120 min and an initial concentration of metal ions of 50 mg/L, when considering the Asa natural clay.

Key words*:* clay, adsorption, nickel, heavy metal, wastewater, pillared clay.

1. Introduction

At present, humanity is facing a serious global water crisis - one billion people around the world do not have access to clean drinking water, and more than 2 billion people do not have enough water purification systems, which is the main cause of water-borne diseases. Heavy metal ions are the main pollutants contained in the wastewater of machine-building enterprises, non-ferrous metallurgy and instrument-making enterprises. Since heavy metals are priority pollutants, its observation is mandatory in all environments. The release of those metals in waters have a high impact in nature, due to their use in significant volumes in the production activity. As consequence, as a result of accumulation in the environment, they provide serious openness in terms of their biological activity and toxic properties. The presented requirements are fully met by natural clays with a high specific surface [1].These metals include nickel (II), chrome (II) and (VI), zinc (II), copper (II), cadmium (II), among others [2]. In particular, nickel is one of the toxic heavy metals that are released into the environment at an exceedingly high level through the discharge of wastewater from electroplating, mining and battery industries [3]. Nickel exists in the form of Ni (II) in aqueous solution and causes cancer of lung, nose and bone when at high concentrations [4].

However, these methods are not suitable for the removal of metals at low concentrations, as typically found in contaminated water streams. Adsorption appears in this context as a suitable process for removing inorganic heavy metals, as well as organic pollutants, from wastewater, due to significant advantages such as low cost, availability, ease of operation, efficiency and effectiveness, when compared to other methods. The process involves the separation of a substance from one phase (liquid) and its accumulation on a solid surface. This method is easy to use and equally effective in removing toxic pollutants even at low concentrations [5]. Efficiency of removal of non-ferrous, heavy and rare metal ions from wastewater by adsorption depends on their concentration in water, pH and total water salinity [6].

Various low cost adsorbents have been employed for removal of heavy metals from wastewater, including bentonite, coir pith, expanded pert lite, fly ash, magnate, montmorillonite, peat moss and smectite [7-11]. Clays are known as lowcost materials with higher surface area, making them good adsorbents. In order to increase its specific surface area, clays can be activated by treatment with acid [12]. In recent works, our group developed methods to produce successfully materials considering environmentally friendly and sustainable routes, such as Zn-based photocatalyst. Following the recognized advantages of developing environmentally and economically sustainable materials, in this work, clay-based materials were prepared from a lowcost natural clay extracted from a Kazakh deposit and applied in the adsorption of Ni (II), used as representative toxic heavy metal. Three samples were considered, the natural clay washed, activated by hydrochloric acid and pillared with Zn and Fe. The main operating conditions involved in the Ni (II) adsorption process (adsorbent load, pH, contact time and initial concentration of Ni (II)) were assessed with all prepared clays.

2. Experimental

2.1. Materials and Preparation Procedures

The natural clay from the Kazakh deposits of Asa (Zhambyl region) was taken as raw material for the preparation of three samples of adsorbents. The first sample resulted from washing of the natural clay with distilled water (Asa). The second sample was obtained after activation of the natural clay with HCl (37%) at room temperature for 24 hours followed by drying at 384 K (Asa-activated). For the third sample, the natural clay was pillared using cations of iron and zinc (Fe/Zn-Asa). The pillarizing solution was prepared at room temperature by slow addition of a 0.2 M NaOH to a solution containing iron (II) sulphate (99,5%) and zinc (II) chloride (98,03%) until pH = 2.8. The resulting solution was aged at room temperature for 24 hours. The pillarization procedure considered a total metal content of 10 mmol per gram of clay. The samples obtained were dried at 350 K and then calcined during 2 hours at 823 K with a heating rate of 275 K/min.

2.2. Materials Characterization and Analytical Methods

To determine the physico-chemical characteristics of the natural clay, X-ray spectral analysis was used. An electron probe microprobe of the Superprobe 733 (Super Probe 733) brand from JEOL (Jael, Japan) was used for determination of the angular position and intensity of reflexes. Analyses of the elemental composition of samples and photography in various types of radiation were performed using the Inca Energy with dispersive spectrometer from Oxford Instruments, England. The concentration of Ni (II) was determined by AES. All measurements were carried out using the Agilent 4200 MP-AES fitted with the Agilent 4107 Nitrogen Generator. The sample introduction system consisted on a double pass cyclonic spray chamber, OneNeb nebulizer, Solvaflex pump tube (orange/green) and Easy-fit torch to introduce the sample. Multi-element calibration standards containing Ni (II) with a concentration of 50 mg/L were used. The standard was prepared in a 5% HNO_3/0.2% HF (v/v) medium (Made in USA).

2.3. Static adsorption runs

Preparation of solutions and pH adjustment

The corresponding amounts of $NiSO_4$•$6H_2O$ were dissolved in distilled water to obtain Ni (II) solutions with concentrations of 50, 40, 30, 20 and 10 mg/L. Adjustment of the solutions pH was carried out using 0.01 M HCl and 0.01 M NaOH solutions.

Effect of adsorbent load

Masses of adsorbent to result in different concentrations of 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0 and 10.0 g/L were placed separately in 250 mL Erlenmeyer flasks and 100 mL of a solution containing 50 mg/L Ni (II) was poured into each. The pH of each was adjusted to 6.0 and the resulting solutions were stirred for 2 hours. The concentration of unadsorbed Ni (II) ions in the filtrate was determined by AES.

Effect of contact time

The adsorbent at the level of 2 g/L was added to each 250 mL Erlenmeyer flask containing a solution of 50 mg/L Ni (II) and the pH was adjusted to 6.0. The mixture was shaken on a laboratory shaker for different time intervals of 0.25, 0.5, 1, 2, 4, 6 and 24 hours. The residual concentration of Ni (II) in the filtrate of each sample was determined by AES.

Effect of the initial concentration of Ni(II)

Solutions containing Ni (II) in different concentrations of 10, 20, 30, 40 and 50 mg/L were placed in separate Erlenmeyer flasks. Then, the adsorbent (2 g/L) was added and the pH was adjusted to 6.0. The resultant suspension was shaken during the contact time of 2 hours. The unabsorbed Ni (II) concentration in the supernatant solution was determined by AES.

3. Results and discussion

3.1 . Characteristics of natural clays and pillared clays

The results of elemental composition of the natural washed clay and of the pillared clay were obtained by EMP analysis. Table 1 presents the content of elements.

Table 1. The results of elemental analysis

Natural and Pillared clays	**Mass of the metals (%)**									
	Na	Mg	Al	Si	K	Ca	Ti	S	Fe	Zn
Asa	1,13	3,41	11,32	58,29	2,70	18,03	0,65	n.d.	2,66	n.d.
Fe/Zn-Asa	1,38	1,55	5,31	17,82	1,78	3,84	0,56	0,34	22,39	2,62

From the analysis of Table 1 it is observed that the pillaring treatment of the Asa clay with Fe and Zn it is observed that the Zn content of the pillared clay is 2.62% and that the amount of iron increased from 2.66% in the natural clay of the Asa deposit to 22.39% after the pillaring process. The chemical composition analysis also shows a high Si content when compared to the other elements.

X-ray diffraction (XRD) spectra were also determined and can be observed in Figure 1. Studies of the mineralogical composition of clay confirm that the clay of the Asa deposit is a representative of polymineral clay. The quantitative ratio of crystalline phases of the clay samples were determined by X-ray diffractometric analysis. The polymineral composition was confirmed by the appearance of the corresponding signals on the X-ray patterns: montmorillonite (d = 14.35 Å), muscovite (d = 9.98, 3.45 Å), quartz (d = 4.25-3.34 Å) with the formula Al_2O_3•$4SiO_2$•xH_2O. Table 2 shows the chemical composition of the natural clay Asa. As can be observed, Asa contain a high quantity of impurities in the form of quartz.

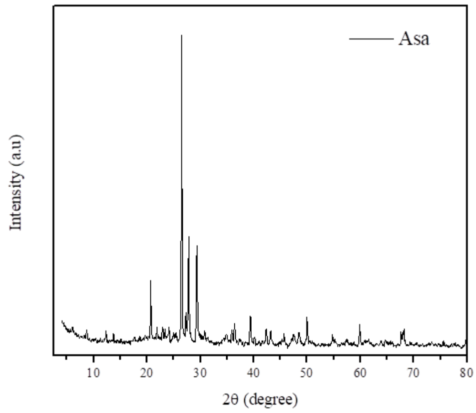
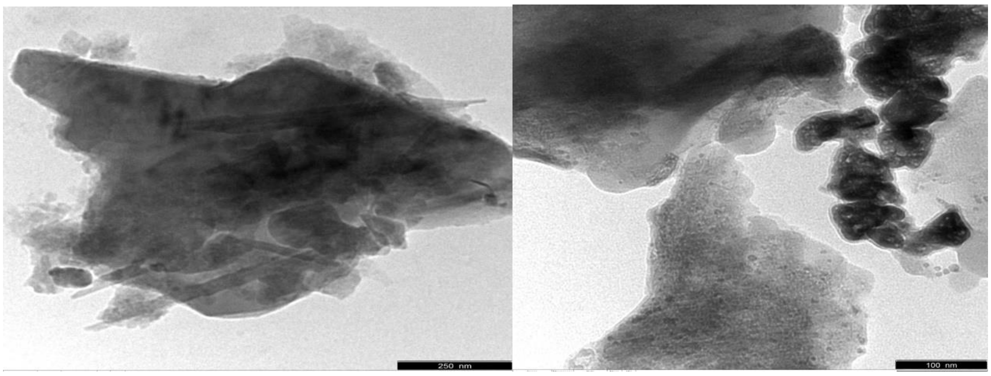


Figure 1. X-ray diffraction spectra of natural clay Asa by EMP

Table 2. Chemical composition of the natural clay Asa determined by semi quantitative analysis from XRD

Mineral	**Asa**
Quartz, SiO_2	32.9%
Calcite, $CaCO_3$	16.2%
Muscovite, $(K_{0.82}Na_{0.18})$ $(Fe_{0.03}Al_{1.97})$ $(AlSi_3)O_{10}(OH)_2$	9.1%
Albite endmember feldspar, $Na(AlSi_3O_8)$	13.7%
Hydrated aluminum silicate, Al_2O_3•$4SiO_2$•xH_2O	4.9%
Kaolinite, $Al_2Si_2O_5(OH)_4$	5.2%
Microcline feldspar, Ksi_3AlO_8	18.6%

The surface morphologies of the natural clays and of the PILCs prepared were observed by transmission electron microscopy (TEM). The micrographs obtained in bright field mode are shown in Figure 2. The TEM images of the pillared clays obtained in dark field mode put in evidence the defects of the structure, as well as fine particles present in the material, as dark-colored spots.



a) Asa b) Fe/Zn-Asa

Figure 2. TEM micrographs obtained for the natural clays and pillared clays

3.2 . Adsorption of Ni

Effect of adsorbent load

The effect of doses of the adsorbent on Ni (II) adsorption using the three clays prepared is shown in Figure 3. It is observed that the adsorption of Ni (II), irrespectively of the clay sample used, increases with an increase in the dose of adsorbent from 1.0 to 10.0 g/L, increasing with the natural clay Asa from 74.9 to 94.5%, with the activated clay from 69.4 to 92.4% and with the pillared clay from 82.0 to 95.6%. This is due to an increase of the available surface area, which in turn increases the availability of interchangeable sites on clay for Ni (II) adsorption. An increase in the dose of the adsorbent up to 10 g/L retains the static adsorption of Ni (II), which may be due to the achievement of equilibrium between the liquid and the solid phases. Based on the results obtained, it can be concluded that all three clay materials have good sorption properties with an increase in their dose up to 3 g/L. The adsorption of Ni (II) with the three samples does not increase significantly when increasing the concentration of adsorbent above 3 g/L.

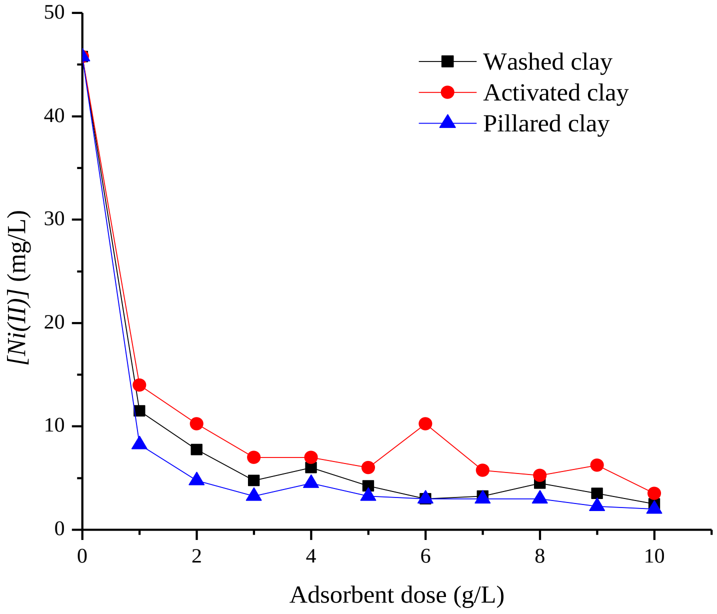


Figure 3. Adsorption of Ni (II) on adsorbents Asa, Asa-activated and Fe/Zn-Asa pillared clay at different adsorbent doses

Effect of contact time

Figure 5 shows the results of Ni (II) adsorption using the natural clay Asa, the activated clay and the Fe/Zn-Asa pillared clay at different contact times. Generically it is observed that adsorption increases slightly over time, the natural clay showing the maximum adsorption after 24 hours (92.9%). With the pillared clay, a rapid Ni (II) adsorption of 89.6% took place in the first 15 minutes, proceeding after more slowly until equilibrium was reached. Similar results can be observed for the clay activated with hydrochloric acid, the maximum adsorption being reached after 24 hours with a value of 83.6%. The fast adsorption of Ni (II) at the beginning of process is explained by the greater number of free adsorption sites at the surface of the clays, which in later stages are occupied by Ni (II) ions, decreasing the number of free adsorption sites.

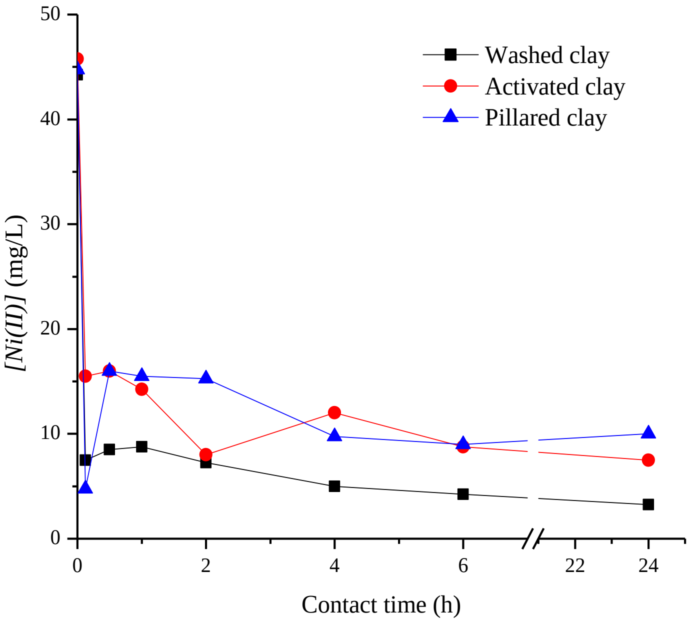


Figure 4. Adsorption of Ni (II) on adsorbents Asa, Asa-activated and Fe/Zn-Asa with varying contact time

Effect of the initial concentration of Ni (II)

Ni (II) adsorption decreases from 88.9 to 83.1% with an increase in the initial concentration of Ni (II) from 10 to 50 mg/L with the natural clay Asa. With the activated clay, the adsorption reached equilibrium with the results at 10 mg/L - 77.8% and at 50 mg/L - 77.6%. This is due to the fact that an increase in the concentration of metal ions causes saturation of the adsorption sites on the clay, blocking the adsorption of Ni (II) on the sites of adsorption, and as a result, the efficiency of adsorption decreases. With the Fe/Zn-Asa pillared clay, the adsorption results change from 75.0 to 89.6%.

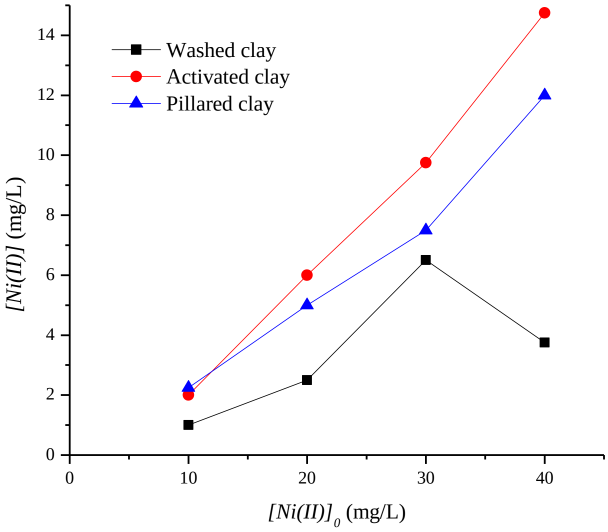


Figure 5. Adsorption of Ni (II) on adsorbents Asa, Asa-activated and Fe/Zn-Asa for varying initial metal ion concentration

4. Conclusions

The use of natural clays as adsorbents for the adsorption of Ni(II) from aqueous solutions shows encouraging results. The adsorption process is influenced by several parameters, such as pH, dose of adsorbent, contact time and initial concentration of metal ions. With the natural clay Asa, the adsorption equilibrium was reached after 120 min and the adsorption removal reached a maximum of 98.9% with an optimum pH of 10.0, a dose of adsorbent of 2 g/L and an initial concentration of metal ions of 50 mg/L. The results of the study convincingly proved the effectiveness of natural clay as adsorbents for the removal of Ni (II) from wastewater.

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АҒЫНДЫ СУЛАРДАН NI (II) ЖОЮ ҮШІН ҚОЛДАНЫЛҒАН ТАБИҒИ САЗБАЛШЫҚТАН ЖАСАЛҒАН ЖАҢА АДОСРБЕНТТЕР

Аннотация. Ағынды суларды адсорбциялық тазарту әдісі, жоғары тиімділікпен бірге, экологиялық қауіпсіз әдістердің бірі болып табылады. Ағынды суларды тазарту үшін материалдарды таңдаған кезде басты өлшемі оның адсорбциялық қасиеттері, кеуекті құрылымы және экономикалық тиімділігі болып табылады. Машина жасау кәсіпорындарының, түсті металлургия кәсіпорындарының, аспап жасау кәсіпорындарының ағынды суларында негізгі ластаушылар ауыр металл иондары болып табылады. Ауыр металдар басым ластаушылар болып табылады, оларды бақылау барлық орталарда міндетті болып табылады. Бұл зерттеу жұмысы Қазақстанын елді мекенінен алынған табиғи сазбалшықтын (монтмориллонит) негізінде, модельдік ластаушы ретінде қолданылған Ni (II) адсорбцисын сулы ерітіндіде жүргізуге арналған. Екі түрлі табиғи сазбалшықтың үлгілері қышқыл және Fe^{2+}, Zn^{2+} тұздарының ерітіндісімен өңделді. Үш түрлі адсорбент үлгілерімен сулы ерітіндіде Ni (II) адсорбциясының pH мәні, адсорбент мөлшері, байланыс уақыты және алғашқы концентрация иондарының әсері зерттелді. Аса табиғи сазбалшығын қодану арқылы Ni (II) ионың 98,9 % максималды жойылуы pH-10, адсорбент мөлшері 2г/л, байланысу уақыты 120 мин және метал ионың бастапқы коцентрациясы 50 мг/л болған кезде алынды.

Түйін сөздер: сазбалшықтар, адсорбция, никель, ауыр метал, ағынды су, бағаналы сазбалшық.

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НОВЫЕ АДСОРБЕНТЫ, ПОЛУЧЕННЫЕ ИЗ ПРИРОДНЫХ ГЛИН, ИСПОЛЬЗУЕМЫХ ДЛЯ УДАЛЕНИЯ NI (II) ИЗ СТОЧНЫХ ВОД

Аннотация. Адсорбционная очистка сточных вод – один из самых экологически чистых методов наряду с высокой эффективностью. Основными критериями при выборе материалов для очистки сточных вод являются его адсорбционные свойства, пористая структура и экономичность. Ионы тяжелых металлов являются основными загрязнителями сточных вод предприятий машиностроения, цветной металлургии, приборостроения. Тяжелые металлы являются преобладающими загрязнителями, контроль над которыми является обязательным во всех средах. Это исследование посвящено использованию природной глины (монтмориллонита), полученной на месторождении Казахстана, для адсорбции Ni (II), используемого в качестве модельного загрязнителя, из водного раствора. Два различных образца из природных глин были подвергнуты обработке кислотой и растворами солей Fe^{2+} и Zn^{2+}. Было изучено влияния pH, дозы адсорбента, времени контакта и начальных концентраций ионов на адсорбцию Ni (II) из водного раствора на трех образцах адсорбента. Максимальное удаление Ni (II) 98,9% было достигнуто при pH 10 и дозе адсорбента 2 г / л при времени контакта 120 мин и начальной концентраций ионов металлов 50 мг / л на природной глине Aca.

Ключевые слова: глина, адсорбция, никель, тяжелый металл, сточные воды, столбчатая глина.

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