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Adsorption of Cu²⁺, Zn²⁺, and Ni²⁺ Ions onto the Adsorbent Prepared from Fluid Catalytic Cracking Spent Catalyst Fines and Diatomite

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Abstract

An adsorbent prepared from fluid catalytic cracking (FCC) spent catalyst fines and diatomite, and its adsorption of Cu²⁺, Zn²⁺, and Ni²⁺ ions were investigated. The adsorbent was characterized by XRD, SEM, and N_2 adsorption-desorption techniques. The results showed that the specific surface area and pore volume of adsorbent increased with the increase in FCC spent catalyst fines. The influence factors on the adsorption of the adsorbents were studied. The suitable adsorption conditions were: pH value of 5.0, ratio of solid to liquid of 1 : 600 (g:ml), adsorption time of 4 h, room temperature. The adsorption of metal ions varied with the type of metal cations. The adsorption isotherms suggested that the sequence of the adsorption efficiency was $Cu^{2+} > Zn^{2+} > Ni^{2+}$. The amount of Cu²⁺, Zn²⁺, and Ni²⁺ metal ion adsorbed onto the adsorbent was 49.17 mg g⁻¹, 46.83 mg g⁻¹, and 35.72 mg g⁻¹, respectively. The adsorption data of Cu²⁺, Zn²⁺, and Ni²⁺ ions fitted well with the Freundlich adsorption isotherm model.

Keywords

Adsorption, heavy metal ion, FCC spent catalyst fines, diatomite

1 Introduction

Industrial wastewater, especially that containing heavy metals, is becoming a serious environmental problem. In general, wastewater contains acid, as well as copper, zinc, nickel, chromium, and other metal ions, which are often high in concentration and toxic. The removal of toxic heavy-metal contaminants from industrial wastewater is one of the most important environmental issues to be solved today. The search for new and innovative treatment technologies has focused attention on the effect of heavy metal toxicity.[1–6]

A fluid catalytic cracking (FCC) unit can produce about 20 thousand tons of catalyst fines (< 20 μm) annually in China.[7–8] The amount of the fines is expected to increase with the prospering development of FCC. The spent fine particles are mainly composed of silica, alumina, zeolite, and trace heavy metals like V, Fe, and Ni. Although these fines have lost the catalytic activity to meet application requirements, they are still an appropriate raw material of high-value silicon/aluminum products, either from crystal phase composition or main chemical elements, and have higher specific surface area and pore volume. *N. S. Japtiwale* has put forward that spent fluidized cracking catalyst can be utilized as an adsorbent for heavy metals from wastewater.[9]

Diatomite is a material of sedimentary origin consisting mainly of an accumulation of skeletons formed as a protective covering by diatoms. The skeletons are essentially amorphous hydrated or opaline silica, but occasionally are partly composed of alumina. Diatomite usually contains other sediments, such as clay and fine sand, but its deposits sometimes consist of diatom shells only. On complete calcination, diatomite produces porous structure.

There are many studies of the adsorption of heavy metal ions onto the adsorbent prepared by clay and modified clay.[10–13] For example, diatomite has been tested as a potential adsorbent for Pb(II) ions. The intrinsic exchange properties were further improved by modification with manganese oxides. Mn-diatomite adsorbent showed a higher tendency for adsorbing lead ions from solution at pH 4. *A. Mellah et al.* have studied the equilibrium isotherms of zinc adsorption onto natural bentonite. Their results show that the data correlate well with Freundlich and Langmuir models, and that the adsorption is physical in nature. *S.-H. Lin et al.* have studied the removal of Cu²⁺ and Zn²⁺ from aqueous solutions by sorption on montmorillonite modified with sodium dodecylsulfate (SDS).

In the present work, an adsorbent was prepared from FCC spent catalyst fines and diatomite. An XRD, SEM, and N_2 adsorption-desorption analysis of the FCC spent catalyst fines and diatomite was also performed in order to explore the properties of the adsorbent. The main interest in the properties of the adsorbent is related to their high selectivity toward metals, such as Cu²⁺, Zn²⁺, and Ni²⁺ ions. The aim of our study was to determine the effect of different operating parameters, such as pH, initial concentration, ionic strength, and temperature on these adsorption pro-

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Table 1 – Chemical composition of SCF and diatomite

Sample	Chemical composition/%									
	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	Re_2O_3	Ni	V	Cu	CaO	MgO
SCF	43.6	49.3	0.43	0.40	4.5	0.38	0.27	0.16	0.25	–
D	91.4	6.1	0.61	0.09	–	–	–	–	0.27	0.54

cesses, as well as study the determination of adsorption isotherms.

2 Experimental

2.1 Materials

Diatomite (marked as D) was obtained from Jilin Changbai diatomite Co., Ltd. FCC spent catalyst fines (marked as SCF) were obtained from Hunan Zhonghe Chemical Co., Ltd. The chemical composition of the SCF and D is listed in Table 1. Specific surface area and total pore volume of SCF and D were 102 m² g⁻¹, 48 m² g⁻¹, and 0.15 ml g⁻¹, 0.42 ml g⁻¹, respectively, and the particle median diameter was 18 μm and 7.2 μm, respectively. Copper nitrate, zinc nitrate, and nickel nitrate were obtained from Tianjin Fuchen Petrochemical Company. Hydrochloric acid was supplied from the Beijing Chemical Industry Company. Solutions of all cations were prepared from reagent-grade nitrate salts.

2.2 Preparation of adsorbent

SCF and D were dispersed in 100 cm³ of HCl (c = 1 mol l⁻¹) solution, and the suspension was stirred for 2 h at 65 °C. After the reaction, the reactant slurry was separated from the mother liquor by vacuum filtration on Buchner funnels, washed with warm distilled water until Cl⁻ ion free, oven-dried at 120 °C, powdered, and sieved. A fraction less than 60 μm was used in all the experiments. The addition of SCF and D was 9 : 1, 7 : 3, 5 : 5, and 2 : 8 (weight ratio), respectively. The samples were marked as A1–A4.

Reference sample: With the same method, the reference samples were prepared from the FCC spent catalyst fines and diatomite alone, respectively (marked as RA1, RA2).

2.3 Adsorption

The amount of Cu²⁺, Zn²⁺ and Ni²⁺ ions adsorbed on the adsorbents was determined: 0.050-g samples were respectively suspended in 25-ml aqueous solutions, containing each cation of Cu²⁺, Zn²⁺, and Ni²⁺ ions at several different concentrations, varying from 10 to 100 mg l⁻¹. At room temperature, initial concentration, pH, and adsorption time were investigated. The pH of the solutions was adjusted with NaOH or HNO₃ (c = 1.0 mol l⁻¹) solution. The solutions were mechanically stirred in an orbital shaker incubator. At the end of this process, the solid was separated by centrifugation for 5 min at 5000 rpm. The adsorbed

metal concentration was determined by the difference between the initial concentration in the aqueous solution and that found in the supernatant, using an atomic absorption spectroscopy (AAS) apparatus.

2.4 Analysis and characterization

Contents of SiO_2, Al_2O_3, Fe_2O_3, Re_2O_3, Na_2O, CaO, and MgO in SCF, diatomite and adsorbents were determined by chemical analysis. Contents of Ni, V, Cu in SCF, and adsorbents were determined by plasma optical emission spectrometry.

XRD: Phase of samples was recorded on a Rigaku Ultimi IV diffractometer using Cu-kα radiation (λ = 1.54056 Å) operating at a tube voltage of 40 kV and a tube current of 30 mA. The samples were scanned in the 2θ angle range from 3° to 50°, at a speed of 0.2 ° min⁻¹. N_2 *adsorption-desorption methods:* The specific surface areas and pore volumes were measured on an ASAP 2020 sorptometer using adsorption and desorption isotherm plots at −196 °C, and the samples were out-gassed at 90 °C for 1 h, and at 350 °C for 4 h prior to testing. *SEM:* The morphology and size of the samples were determined using scanning electron microscopy (SEM) (JEOL JSM-6360) after coating with an Au evaporated film. *Particle size distribution:* A Malvern Micro-P particle size distribution analyser was used for determining the size distribution of the samples. *Atomic absorption spectrometer:* Concentrations of Cu²⁺, Zn²⁺, and Ni²⁺ in the aqueous solutions were measured on an AA-6880 atomic absorption spectrometer. The amount of metal ion adsorption was obtained by the following equation:

$$q = (C_0 - C) \cdot V/m \qquad (1)$$

where, q is the amount of metal ion adsorbed on samples (mg g⁻¹), C_0 and C are the initial and equilibrium liquid-phase concentrations of metal ion solution (mg l⁻¹), respectively, V is the volume of metal ion solution (ml), m is the mass of the adsorbent sample (g).

3. Results and discussion

3.1 Properties of the adsorbent

3.1.1 XRD characterization

Figs. 1 and 2 show XRD patterns of the samples and the adsorbents, respectively. In Fig. 1, the curve revealed that FCC spent catalyst fines were mainly composed of Al_2O_3 and small amounts of zeolite Y and zeolite ZSM-5. From
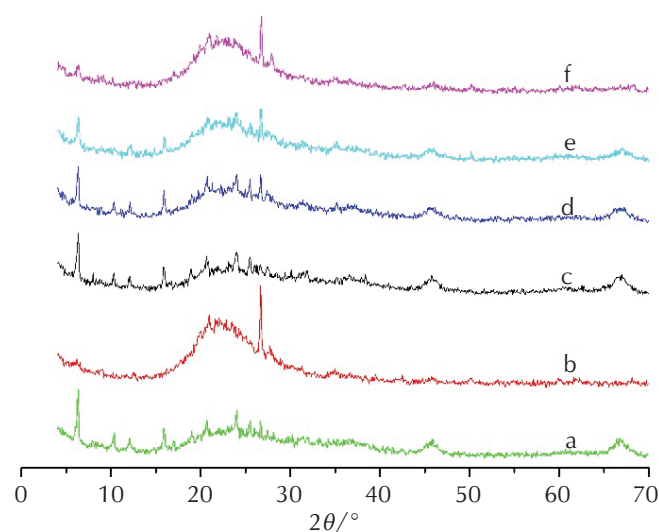
Fig. 1 – XRD patterns of the samples (a) SCF; (b) D; (c) SCF : D = 90 : 10; (d) SCF : D = 70 : 30; (e) SCF : D = 50 : 50; (f) SCF : D = 20 : 80

Fig. 2 – XRD patterns of the adsorbents (a) RA1; (b) RA2; (c) A2; (d) A3; (e) A4

the curve b, diatomite had a very wide asymmetric diffraction peak between 18 and 28 degrees of 2θ, indicating that the internal structure in the diatomite was highly disordered, which was basically consistent with the XRD pattern of opal-AG. Diatomite had a diffraction peak at d value of 4.255 and 3.343, indicating that diatomite mixed with a minority amount of quartz.[14] Curves c–f were the mixing samples of FCC spent catalyst fines and diatomite in different proportions. It was found that the diffraction peak intensity of SCF decreased with its amount decreasing. It was the same for diatomite.

Fig. 2 reveals that the patterns of RA1 and RA2 basically had not changed compared to SCF and diatomite. A2–A4 were basically the same as that of curves d–f in Fig. 1, indicating that these modification conditions had not caused change in the phase.

3.1.2 Specific surface area and pore volume of the adsorbent

Table 2 shows the specific surface area and pore volume of the adsorbents. The results showed that the specific surface area of the adsorbent increased with the increase in the amount of FCC spent catalyst fines in the adsorbent, while the pore volume of the adsorbent increased with the increase in the diatomite amount in the adsorbent. This was because the material itself had a larger surface area and pore volume. After modification, the specific surface area and pore volume of FCC spent catalyst fines and diatomite all increased. These results showed that acid activation of FCC spent catalyst fines and diatomite lead to increases in specific surface area, total pore volume, and mesopore volume.
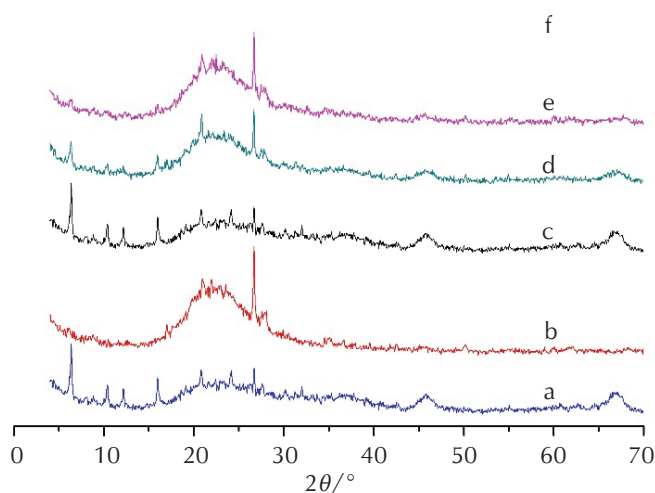
Table 2 – Specific surface area and pore volume of the samples

Sample	Pore volume / ml g⁻¹	Mesopore volume / ml g⁻¹	Special surface area / m² g⁻¹
A1	0.21	0.18	109
A2	0.28	0.22	103
A3	0.34	0.25	81
A4	0.42	0.31	66
SCF	0.15	0.14	102
D	0.42	0.28	48
RA1	0.17	0.16	128
RA2	0.48	0.33	53

3.1.3 Chemical composition of the adsorbent

Table 3 shows the chemical composition of the adsorbents. The results showed that the adsorbents mainly contained SiO_2, Al_2O_3, Re_2O_3 and trace metals of Fe, V, Ni, Cu, CaO, and Na_2O. The acid treatment reduced or eliminated all other oxides relative to SiO_2. With the increase in the content of diatomite in the adsorbents, the loss of alumina in the adsorbents reduced, indicating that the presence of diatomite effectively reduced the aluminum from the FCC waste catalyst fines in the acid system.

3.1.4 SEM characterization

Fig. 3 shows SEM of the SCF and diatomite. Fig. 3(a) displays that FCC spent catalyst fines possessed lamellar structure with rough surface. The particle size was over 10 μm and non-uniform. The diatomite morphology is shown in

Table 3 – Chemical composition of the adsorbents

Sample	Chemical composition/%									
	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	Re_2O_3	Ni	V	Cu	CaO	MgO
A1	52.1	41.6	0.25	0.16	3.8	0.14	0.17	0.05	0.13	0
A2	61.4	33.9	0.27	0.17	3.0	0.10	0.11	0.04	0.09	0.02
A3	70.6	25.8	0.17	0.10	2.1	0.07	0.09	0.03	0.06	0.05
A4	84.2	13.6	0.13	0.05	0.9	0.03	0.04	0.01	0.03	0.06
RA1	50.8	43.6	0.21	0.16	4.1	0.15	0.18	0.06	0.15	–
RA2	94.7	4.2	0.11	0.01	–	–	–	–	0.06	0.08

Fig. 3(b). The diatomite was mainly dominated by disc algae, and the micropores on the surface of algae were clearly visible and evenly distributed.
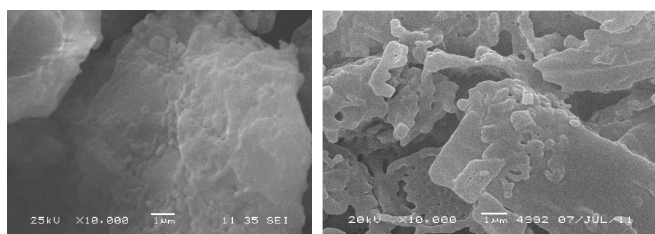

Fig. 3 – XRD patterns of the samples (a) SCF; (b) D

3.2 Metal ion adsorption

3.2.1 Adsorption properties of different adsorbents

0.050 g of samples were suspended in 25 ml aqueous solution, respectively. The concentration of Cu²⁺, Zn²⁺, and Ni²⁺ metal ions in this aqueous solution was 100 mg l⁻¹, respectively. The conditions were room temperature, pH = 5.0, ratio of solid-liquid 1 : 600 (g:ml), and adsorption time 6 h. The results are shown in Table 4. It was found that the adsorption of Ni²⁺ ions onto A1 was greater than that onto A2–A4. The adsorption of Cu²⁺ ions was just the opposite, and A3 had the best effect on the adsorption of Zn²⁺ ions. This demonstrated that the adsorption

Table 4 – Adsorption of Cu²⁺, Zn²⁺, and Ni²⁺ ions onto the adsorbents

Sample	Adsorption value/mg g⁻¹		
	Cu²⁺	Zn²⁺	Ni²⁺
A1	49.2	38.3	37.5
A2	52.9	38.6	37.2
A3	54.7	40.1	36.8
A4	54.9	39.4	36.1
SCF	18.8	19.3	10.7
D	11.5	10.1	8.9

of Ni²⁺ ions onto the FCC spent catalyst fines was greater than that of Cu²⁺ and Zn²⁺ ions, and the adsorption of Cu²⁺ ions onto the diatomite was greater than that of Zn²⁺ and Ni²⁺ ions. Overall, the adsorption value of A3 was the best. Therefore, the A3 adsorbent was used as the adsorbent in the following experiment. The adsorption capacity of Cu²⁺, Zn²⁺, and Ni²⁺ metal ions by FCC spent catalyst fines and diatomite alone is much lower than that of other adsorbents. To adsorption performance, diatomite has synergistic effect with FCC spent catalyst fines.

3.2.2 Effect of solid-liquid ratio on adsorption

0.050 g of A3 was suspended in 25 ml aqueous solution. The concentration of Cu²⁺, Zn²⁺, and Ni²⁺ metal ions in this aqueous solution was 100 mg l⁻¹, respectively. At room temperature, pH = 4.0, effect of solid-liquid ratio (g ml⁻¹) on adsorption was tested. The results are shown in Fig. 4. It was found that the amount of Cu²⁺, Zn²⁺, and Ni²⁺ ions adsorbed onto A3 increased with the increase in solid-liquid ratio . When solid-liquid ratio was 1 : 700, the amount of Cu²⁺, Zn²⁺, and Ni²⁺ metal ions adsorbed onto the adsorbent became more balanced. Based on the results, the optimum ratio of solid-liquid was 1 : 600 (g:ml).
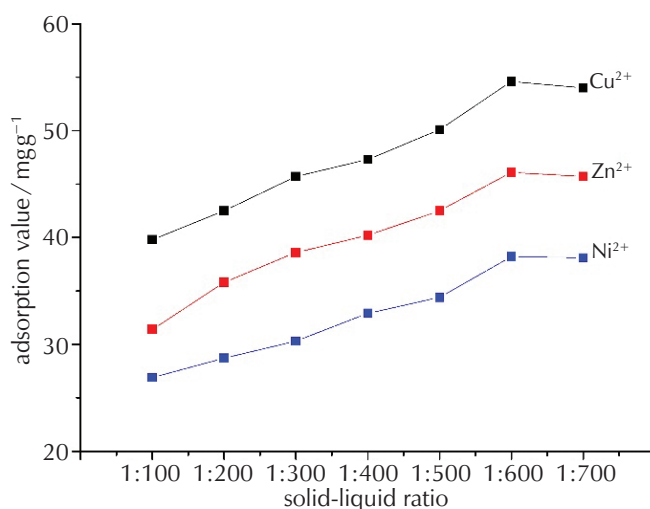
Fig. 4 – Effects of solid-liquid ratio on adsorption of Cu²⁺, Zn²⁺, and Ni²⁺

3.2.3 Effect of pH on adsorption

The pH is one of the most important factors in controlling the adsorption of these cations at the surface. 0.050 g of A3 was suspended in 25 ml aqueous solution. The concentration of Cu^{2+}, Zn^{2+}, and Ni^{2+} metal ions in this aqueous solution was 100 mg l⁻¹, respectively. At room temperature, ratio of solid-liquid 1 : 600 (g:ml), and adsorption time 2 h. The effect of pH on the adsorption of metal ions onto the adsorbent is given in Fig. 5. It was found that the effect of pH on the adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent had two aspects: (1) the competitive adsorption between Cu^{2+}, Zn^{2+}, and Ni^{2+} ions and H^+, and (2) changes in surface hydroxyl groups indicated that the numbers of adsorbed metal ions increased with the increase in pH. With increased pH, H^+ concentration in the solution decreased, the competitive adsorption of H^+ ions decreased, the heavy metal ions had more opportunities to close to active site in surface.
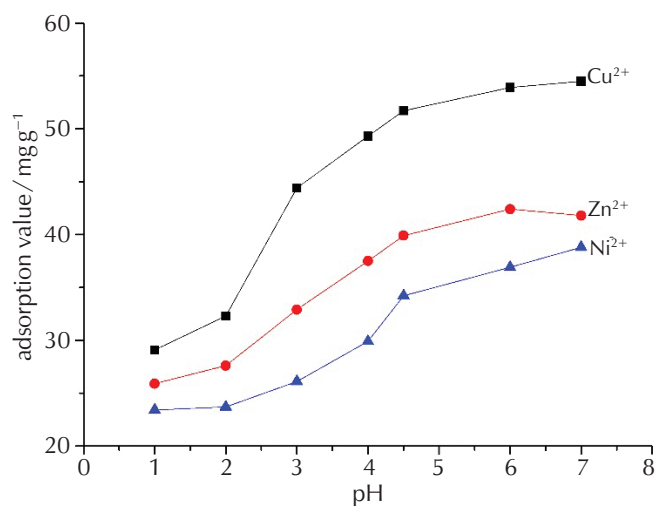

Fig. 5 – Effects of pH value on the adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+}

From Fig. 5, pH significantly affected the adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions. When pH was less than 4.0, the adsorption content of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent was low. With pH above 4.0, the adsorption content of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent continuously increased. When pH value above 6.0, the precipitation began to appear. Therefore, the moderate pH value was 5.0.

3.2.4 Effect of conditioning time on adsorption

0.050 g of A3 was suspended in 25 ml aqueous solution. The concentration of Cu^{2+}, Zn^{2+}, and Ni^{2+} metal ions in this aqueous solution was 100 mg l⁻¹, respectively. Conditions were: room temperature, pH = 5.0, and ratio of solid-liquid 1 : 600 (g:ml). The effect of adsorption time on the adsorption of metal ions onto the adsorbent is given in Fig. 6. From Fig. 6, it may be seen that, with the increase in adsorption time, the adsorption content of Cu^{2+}, Zn^{2+}, and

Ni^{2+} ions onto the adsorbent also increased. With adsorption time longer than 6 h, the rate of adsorption changed slowly, and the adsorption content of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent slightly increased as adsorption time increased further. When adsorption time was longer than 8 h, the change in the adsorption content of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent was not obvious. Therefore, the moderate adsorption time was 6 h.
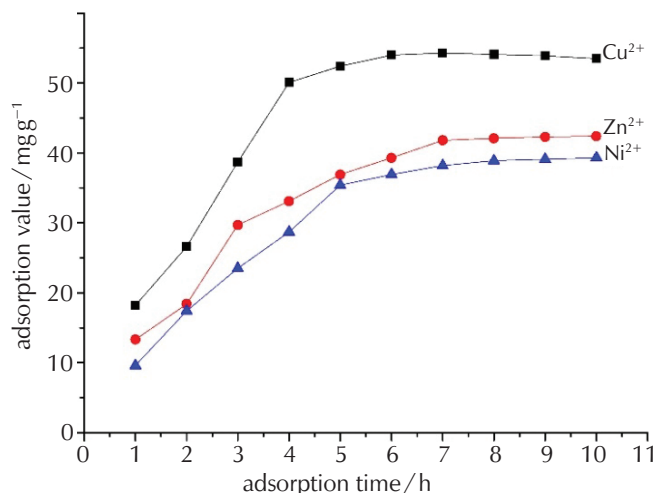

Fig. 6 – Effects of adsorption time on adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+}

3.2.5 Effect of concentration of metal ion on adsorption

Twenty-five ml of the solution containing Cu^{2+}, Zn^{2+}, and Ni^{2+} ions at 10, 20, 30, 40, 50, 60, 80, and 100 mg l⁻¹ was prepared, respectively. 0.050 g of A3 was added, respectively, at room temperature, pH = 5.0, adsorption time 6 h, and ratio of solid-liquid 1 : 600 (g:ml). The effect of concentration of Cu^{2+}, Zn^{2+}, and Ni^{2+} ion on adsorption was studied. The results are shown in Fig. 7.
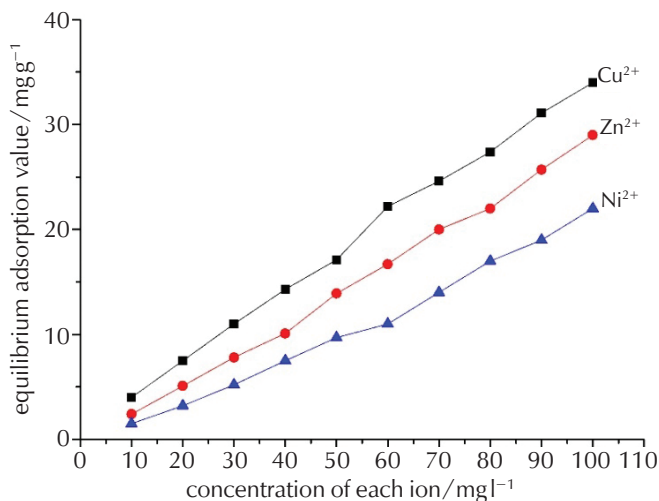

Fig. 7 – Relationship between the concentration of heavy-metal ions and adsorption value

From Fig. 7, the adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent surface can be interpreted with the Freundlich equation curve:

$$\lg q = (1/n) \lg C + \lg K_F \qquad (2)$$

where, C is equilibrium concentration ($mg\,l^{-1}$), q is the equilibrium adsorption capacity ($mg\,g^{-1}$), n and K_F are constants.

By mapping of $\lg q$ to $\lg C$, after regression analysis, three linear equations were obtained:

$$\lg q = 0.9423 \lg C - 0.3537 \qquad (3)$$
$$\lg q = 1.0856 \lg C - 0.7125 \qquad (4)$$
$$\lg q = 1.1749 \lg C - 1.0245 \qquad (5)$$

Computer simulation was used to fit the Freundlich equation to the adsorption data. The accuracy of these fits was estimated in terms of the computed correlation coefficient. Equations (3)–(5) are linear equations of the adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent surface, respectively. The correlation coefficients were 0.9967, 0.9959, 0.9957, respectively (see Fig. 8(1)–(3)). The experiment data of adsorption onto the adsorbent were in accordance with Freundlich adsorption model.

3.2.6 Comparison of the adsorption capability

The adsorption ability of the different adsorbents was tested. Under the optimized conditions mentioned previously, 1.0 g adsorbent and reference samples were suspended in aqueous solution, respectively. The concentration of Cu^{2+}, Zn^{2+}, and Ni^{2+} metal ions in this aqueous solution was 1000 mg l⁻¹, respectively. The solution was mechanically stirred for 6 h. The concentration of Cu^{2+}, Zn^{2+}, and Ni^{2+} in solution was determined, and the results are shown in Table 5. From Table 5, it may be seen that the capability of A3 adsorbent surface to adsorb cations from aqueous solution was significantly better than the reference samples prepared from the FCC spent catalyst fines and diatomite alone. The adsorption capability of A3 adsorbent onto the cations gave an order of $Cu^{2+} > Zn^{2+} > Ni^{2+}$.

Table 5 – Adsorption ability of the adsorbent

Sample	Adsorption value / mg g⁻¹		
	Cu^{2+}	Zn^{2+}	Ni^{2+}
A3	166.8	150.7	134.3
RA1	105.4	89.6	65.1
RA2	78.1	64.5	45.2
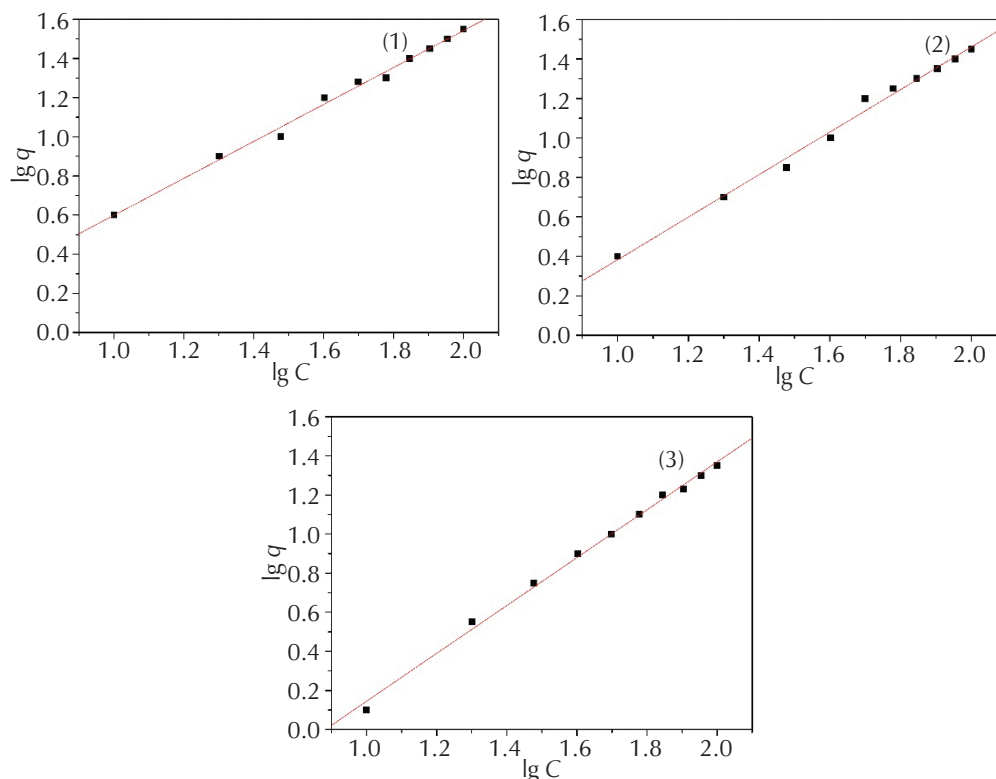
Fig. 8 – Linear fitting of the adsorption *vs.* initial concentration of heavy-metal ions: (1) Cu^{2+}; (2) Zn^{2+}; (3) Ni^{2+}

4. Conclusions

In this paper, the properties of the adsorbent prepared from FCC spent catalyst fines and diatomite, and its adsorption were investigated. We studied the effect of different operating parameters on these adsorption processes, such as initial concentration, pH, ionic strength, and adsorption time. The results showed that the adsorbent possessed larger specific surface area, pore volume, and mesopore volume. This confirmed that the adsorbents had strong ability to adsorb the metal cations. When the adsorbent contained more FCC spent catalyst fines, it showed higher adsorption capacity of Ni^{2+} ions than other metal ions. When the adsorbent contained more diatomite, the adsorbent had greater adsorption capacity of Cu^{2+} ions than other metal ions. The amount of Cu^{2+}, Zn^{2+}, and Ni^{2+} metal ion adsorbed onto the adsorbent was 49.17 mg g⁻¹, 46.83 mg g⁻¹, and 35.72 mg g⁻¹, respectively. The adsorption of Cu^{2+}, Zn^{2+}, and Ni^{2+} ions onto the adsorbent surface can be interpreted with the Freundlich adsorption model.

List of abbreviations

AAS   – atomic absorption spectroscopy

FCC   – fluid catalytic cracking

SDS   – sodium dodecylsulphate

SEM   – scanning electron microscopy

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SAŽETAK

Adsorpcija Cu²⁺, Zn²⁺ i Ni²⁺ iona na adsorbens
pripremljen iz potrošenog FCC katalizatora i kremenih diatomita

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Ispitivan je adsorbens pripremljen iz katalizatora za katalitičko krekiranje u vrtložnom sloju (FCC) i diatomita, te njegova adsorpcija iona Cu²⁺, Zn²⁺ i Ni²⁺. Adsorbens je karakteriziran XRD, SEM i N₂ adsorpcijsko-desorpcijskim tehnikama. Rezultati su pokazali da se specifična površina i volumen pora adsorbensa povećavaju s povećanjem količine potrošenog katalizatora FCC. Proučavani su čimbenici utjecaja na adsorpciju adsorbensa. Prikladni uvjeti adsorpcije bili su: pH vrijednost 5,0, omjer čvrste tvari i kapljevine 1 : 600 (g:ml), vrijeme adsorpcije 4 h i sobna temperatura. Adsorpcija metalnih iona varira s tipom metalnih kationa. Adsorpcijske izoterme pokazale su da je slijed učinkovitosti adsorpcije Cu²⁺ > Zn²⁺ > Ni²⁺. Količina metalnih iona Cu²⁺, Zn²⁺ i Ni²⁺ adsorbiranih na adsorbensu iznosila je 49,17 mg g⁻¹, 46,83 mg g⁻¹, odnosno 35,72 mg g⁻¹. Podatci o adsorpciji iona Cu²⁺, Zn²⁺ i Ni²⁺ slijede model Freundlichove adsorpcijske izoterme.

Ključne riječi
Adsorpcija, ion teških metala, potrošeni FCC katalizator, diatomit

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