

Heavy Metal Removal from Aqueous Solutions by Adsorption using Montmorillonite

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Removal of Cu(II), Zn(II), Cd(II) and Cr(III) from aqueous solutions by sorption on the clay from south west region of Anatolia was investigated. Experiments were carried out as a function of solution pH (2-8), initial solute concentration (1×10^{-4} - 1×10^{-3} mol/L), temperature (20-65°C), amount of adsorbent dosage (0.1-0.9 g) and time (5-2880 min) by batch process. Sorption characteristics of montmorillonite were evaluated by using Freundlich, Langmuir and Dubinin-Radushkevich (D-R) adsorption isotherms. X-Ray diffraction, FTIR, SEM and elemental analysis were performed for characterization of clay. Concentrations of heavy metals in solution are determined by flame atomic absorption spectrometry.

Key Words: Clay, Heavy metals, Langmuir isotherm, Freundlich isotherm, Dubinin-radushkevich model.

INTRODUCTION

Many toxic heavy metals have been discharged to the environment through natural phenomena and human activities, such as industrial wastes, agricultural practices, transport and waste disposal, causing serious water pollution¹⁻⁴. Heavy metals are well known for toxicity and their disposal is a significant industrial waste problem⁵. Their amount in the environment not only increases every year, but also they are not biodegradable and tend to accumulate in living organisms³. Although many heavy metals are necessary in small amounts for the normal development of the biological cycles, most of them become toxic at high concentrations. Furthermore, many are biologically essential but toxic with increasing dosage⁶. Cadmium and chromium show high toxicity to humans as well as to animals, while copper and zinc show moderate toxicity to humans and animals. All these metals are also toxic to plants, being cadmium the strongest phototoxic elements^{7,8}.

Various physicochemical and biological methods have been studied for metal removal. Treatment processes for the removal of heavy metal ions from wastewater include coagulation, carbon adsorption, ion exchange,

precipitation, reverse osmosis, *etc.* The sorption processes are probably the most attractive among these methods, since their application is simple and they require relatively mild operating conditions, although the cost of substrate materials and regeneration is the limiting factor⁹⁻¹¹.

The adsorption models deal with the equilibrium of the metal ions between the aqueous solution and the solid phase. It is well understood that the adsorption phenomena for a given pair of metal ion and solid surface is mainly a function of metal ion concentration and the concentration of other ions¹², especially pH 4.5.

Most of the previous studies focused on using ion exchange resins to remove chromium¹³. In addition, the sorption of heavy metals or chromium on brown coals^{14,15}, *Ocimum basilicum*¹⁶, IRN77 cation-exchange resin¹⁷, bio-polymeric beads of cross linked alginate and gelatin¹⁸, pretreated biomass of *Phanerochaete chrysosporium*¹⁹, eelgrass (*Zostera noltii*)²⁰, fern²¹ and clinoptilolite^{2,5,9} were presented.

Use of clays as sorbents to remove contaminants has been increasingly paid attention because they are cheaper than other materials such as activated carbon, synthetic resins and synthetic zeolite. Clays are widely applied in many fields of technology and science¹. Clay linings have been used as barriers in landfills to prevent contamination of groundwater and subsoil by leachates containing metals. These clays are chosen to avoid pollutant release into the environment owing to their high specific surface areas, high chemical and mechanical stability, a variety of surface and structural properties, the degree of surface heterogeneity due to the existence of crystal edges, broken bonds and imperfection on the surfaces, soil organic matter, low cost and ubiquitous presence in most soils^{1,2,12}. In other word, the chemical nature and pore structure generally determine the sorption ability of clay.

Montmorillonite can adsorb heavy metals *via* two different mechanisms: (1) cation exchange in the interlayers resulting from the interactions between ions and negative permanent charge and (2) formation of inner-sphere complexes through Si-O⁻ and Al-O⁻ groups at the clay particle edges. Both mechanisms are pH dependent because in acid conditions (pH < 4) most silanol and aluminol groups are protonated; therefore, in particular for the latter, an acidification can lead to an increase in mobility of metals bound to soil. For this reason, it is necessary to improve the knowledge of the effect of pH on the sorption capacity of montmorillonite in soil-solution system².

Brown coals and different ion-exchange resins as sorbents for heavy metals from aqueous solutions were used^{13,14}. The aim of this work was to provide fundamental equilibrium and kinetic information in the sorption of Cu(II), Zn(II), Cd(II) and Cr(III) from aqueous solutions on montmoril-

lonite. Sorption characteristics were investigated using Freundlich, Langmuir and Dubinin-Radushkevich adsorption isotherms. Finally, many parameters including sorption capacity were determined. These parameters would be very useful in the clays from an aqueous environment.

EXPERIMENTAL

The clay used was collected from a deposit in the south western part of Turkey (Yalvac-Isparta), which had an idealized formula of $(\text{Na}, \text{Ca})_{0.3} (\text{Al}, \text{Mg})_2 \text{Si}_4\text{O}_{10} (\text{OH})_2 \cdot x\text{H}_2\text{O}$. It was grounded and sieved to a grain size of -65 mesh ASTM before use in the sorption experiments. The samples were washed prior to the adsorption experiments in order to remove the surface dust and finally kept in bottles for future usage. Samples were dried in a vacuum oven at 60°C for 1 h before use. The surface structures of the clay with metals loaded were determined by SEM measurement. SEM (Jeol 5600-LV Model instrument) photographs of montmorillonite were given as Figs. 1a-d.

The results of elemental compositions (%) of the clay are as: C = 0.560, H = 1.900, N = 0.020 and S = 0.045. A Perkin Elmer BX Model FTIR spectrometer for FTIR spectrum of clay was used.

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ were supplied by Merck as analytical grade reagents. The solution pH was adjusted by adding a small amount of 0.1 M HCl or NaOH.

For the sorption of metal ions, 0.5 g of montmorillonite was used in a batch vessel at $20 \pm 1^\circ\text{C}$. The montmorillonite was contacted with different concentrations of Cu(II), Cd(II), Zn(II) and Cr(III) ranging from 1×10^{-4} to 1×10^{-3} mol/L. A Memmert WB29 model thermostated water bath shaker was used to mix the solution for a period of 2 h. The mixture was filtered and washed with distilled water until the filtrate was reached 50 mL. The concentration of metals in the solution was determined by a Perkin Elmer atomic absorption spectrophotometer and the pH was measured with a Crison Basic20 Model pH meter. The amount of metals sorbed on the clay was calculated from a mass balance equation. The solid phase compositions were calculated from the mass balance by taking into account the initial and equilibrium compositions of the liquid phase, according to the equation:

$$q = V/W (C_0 - C) \quad (1)$$

where C_0 and C are the initial and equilibrium concentration of metal in the liquid phase (mol/L), respectively, q is the clay phase concentration of metal (mol/g), V is the volume of solution (L) and W is the mass of dry clay (g)^{13,22}. Each experiment was performed twice at least under identical conditions.

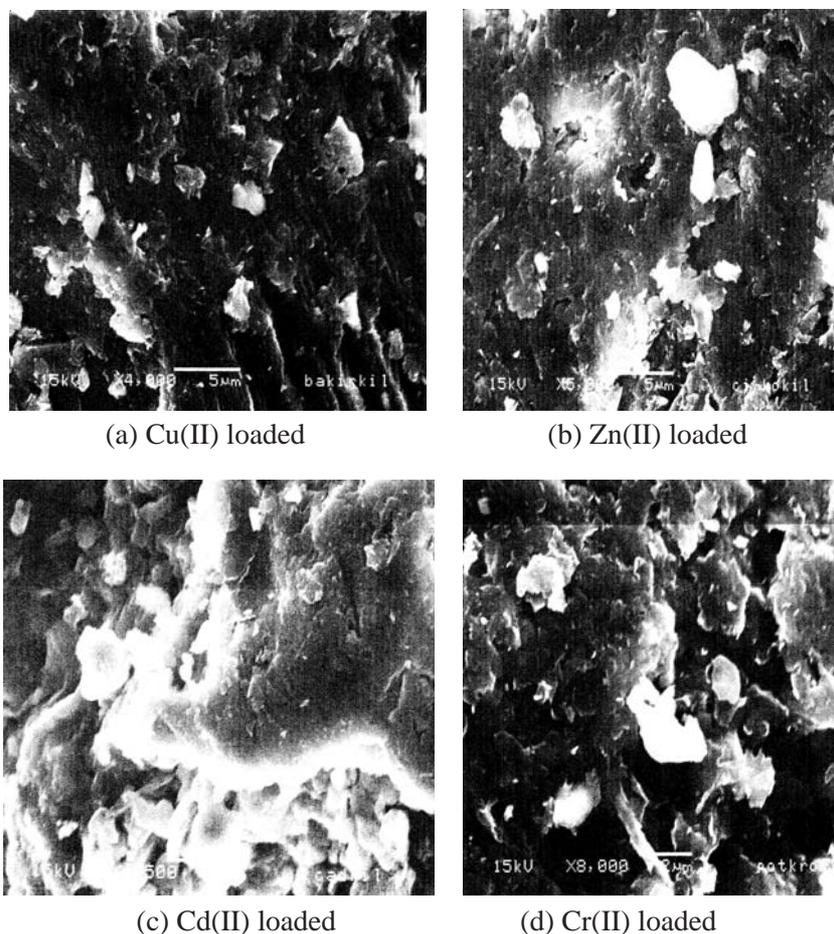


Fig. 1. Scanning electron microscopy of clays with metal loaded

For the effect of adsorbent dosage, a measured quantity of montmorillonite (0.1-0.9 g) was added into a vessel, containing measured volumes of metal solutions (30 mL) at 1×10^{-3} mol/L initial concentration.

To evaluate effect of pH on the efficiency of the clay, solutions pH was initially adjusted ranging from 2-8 using 0.1 M HCl or NaOH. The equilibrium studies were conducted as follows: A measured quantity of clay (0.5 g) was added in a vessel containing a measured volume of metal solutions (30 mL) at 1×10^{-3} mol/L initial concentration with pH initial adjustment, as above. In order to check for precipitation of metal hydroxides, control samples were acidified at the end of each experiment and analyses were performed for the evaluation of heavy metals in the solution. Cu(II), Cd(II), Zn(II) and Cr(III) solutions of appropriate concentrations were prepared by dissolution of the appropriate salts in distilled water.

For the effect of time, samples were withdrawn at several time intervals and analysis of metal remaining in the solution was performed. To evaluate adsorption rates of the studied cations by the clay, 0.5 g clay was mixed with cations solution and stirred for 0-2880 min at $20 \pm 1^\circ\text{C}$.

Starting concentrations of the solutions were adjusted on the basis of preliminary sorption experiments, so that the expected concentrations of metal ions in the solution during the sorption process are in the range suitable for measuring by atomic absorption spectroscopy and concentration of metal ions in wastewater are similar.

RESULTS AND DISCUSSION

SEM images of clays with Cu(II), Zn(II), Cd(II) and Cr(III) loaded were given in Figs. 1a-d. Clay surfaces have favourable homogeneous structure for metal sorption. The bands observed from FTIR spectrum are listed as follows: 468, 521, 796, 873, 1447, 1637, 3436, 3628 cm^{-1} . The bands at 3628, 3436, 1637 and 1447 cm^{-1} are generally attributed to the (OH) vibration of clays. The bands of metal oxides groups are 1035-468 cm^{-1} spectral region as given in Fig. 2.

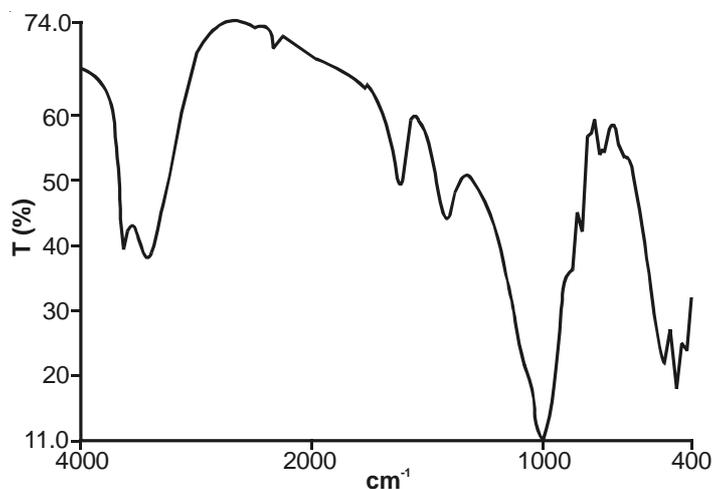


Fig. 2 FTIR spectrum of montmorillonite

Depending on the type of the clay, clay minerals show a variety of surfaces which exhibit electrical charge properties having a strong influence on the sorption of ionic and polar species. The charge on the surface of the soils must be counterbalanced in the aqueous phase to maintain electro neutrality. As a result, electrical double layer exists at the clay/water interfaces.

MOH^+ is formed and adsorption process is rapidly completed by interaction with negative charge on clay, but clay also absorbs metal ions by their outer sheet of hydroxyl groups:



The clay surface may also be negatively charged, providing adsorption sites for metal ions:



Effect of adsorbent dosage

Effect of the amount of clay on the adsorption for Cu(II), Cd(II), Zn(II) and Cr(III) was studied at fixed temperature ($20 \pm 1^\circ\text{C}$). It can be seen in Fig. 3 that the total amount of metal ions removed from the solutions increases by the amount of adsorbent. This is an expected result because as the adsorbent concentration increases, the number of adsorbent particles surrounding the metal ions or ratio of adsorbent particles to metal ions increases, therefore, these particles attach more ions to their surfaces^{23,24}.

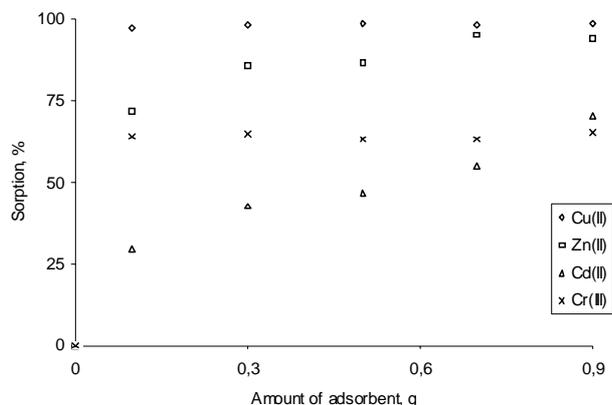


Fig. 3. The effects of the amount of clay for Cu(II), Cd(II), Zn(II) and Cr(III) ions from aqueous solutions (Adsorption conditions; initial concentration of metals: 1×10^{-3} mol/L, amount of clay: 0.1-0.9 g, volume of adsorption medium: 30 mL, temperature: $20 \pm 1^\circ\text{C}$, adsorption time: 2 h)

Effect of pH

Fig. 4 shows the effect of equilibrium pH on the amount of sorption of single metal ions by clay. Apparently, the amount of sorption $\log K$ increases with increasing pH for all metal ions. The sorption of these ions is primarily affected by the surface charge on the sorbent, which in term is influenced by solution pH. K is an equilibrium partition constant. The low $\log K$ at low pH may be explained by the competitive sorption between

proton and metal ions^{1,5,6,25} and the aluminol and silanol groups are more protonated and, hence, they are less available to retain the investigated metals. This effect is strongly evident for Cu, Pb and Cd and less pronounced for the others. The reason of this behaviour is that the surface complexation reactions are influenced also by the electrostatic attraction between the surface charge and the dissolved ions². As the pH of the solution increases, the number of negatively charged sites increases, which favours the sorption of metal cations¹.

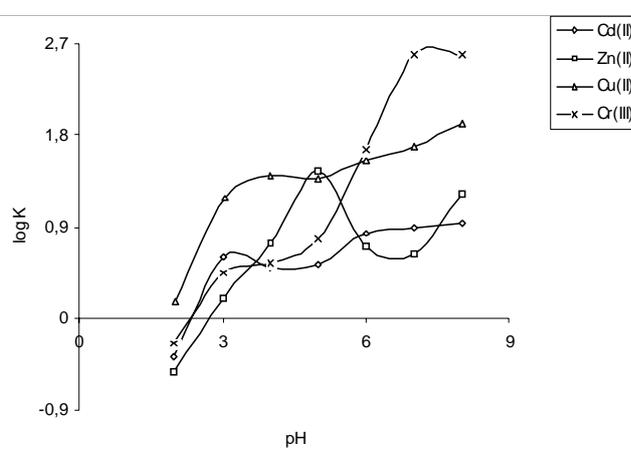


Fig. 4. The distribution coefficients of Cu(II), Cd(II), Zn(II) and Cr(III) ions on montmorillonite as a function of pH (Adsorption conditions; initial concentration of metals: 1×10^{-3} mol/L, amount of clay: 0.5 g, volume of adsorption medium 30 mL, temperature: $20 \pm 1^\circ\text{C}$)

Cd ions are adsorbed less on clay even if they have same valence as Cu and Zn because they are characterized by larger ionic radii and, then, their introduction in interlayer and complexation with surface sites is limited by steric hindrance and lower electrostatic attraction. Cr(III) ion, instead, has a higher charge density and, then, the columbic attraction towards the superficial sites of the clay is greater. Thus, the total capacity of montmorillonite towards the trivalent ion considered is the highest. In general, montmorillonite was found to have a good total capacity towards all considered metals².

Dubinin-Radushkevich (D-R), Langmuir and Freundlich equations

The single-solute sorption isotherms for Cu(II), Zn(II), Cd(II) and Cr(III) by montmorillonite at $20 \pm 1^\circ\text{C}$ are shown in Fig. 5. The Freundlich, Langmuir and Dubinin-Radushkevich adsorption isotherms were used to analyze the results and these isotherms have been proved to be useful in describing adsorption behaviour of the clays for metal ions.

The Dubinin-Radushkevich^{1,26}, Langmuir¹³ and Freundlich²⁷ equations have been used to describe the sorption of metal ions on montmorillonite. The D-R equation has the following form:

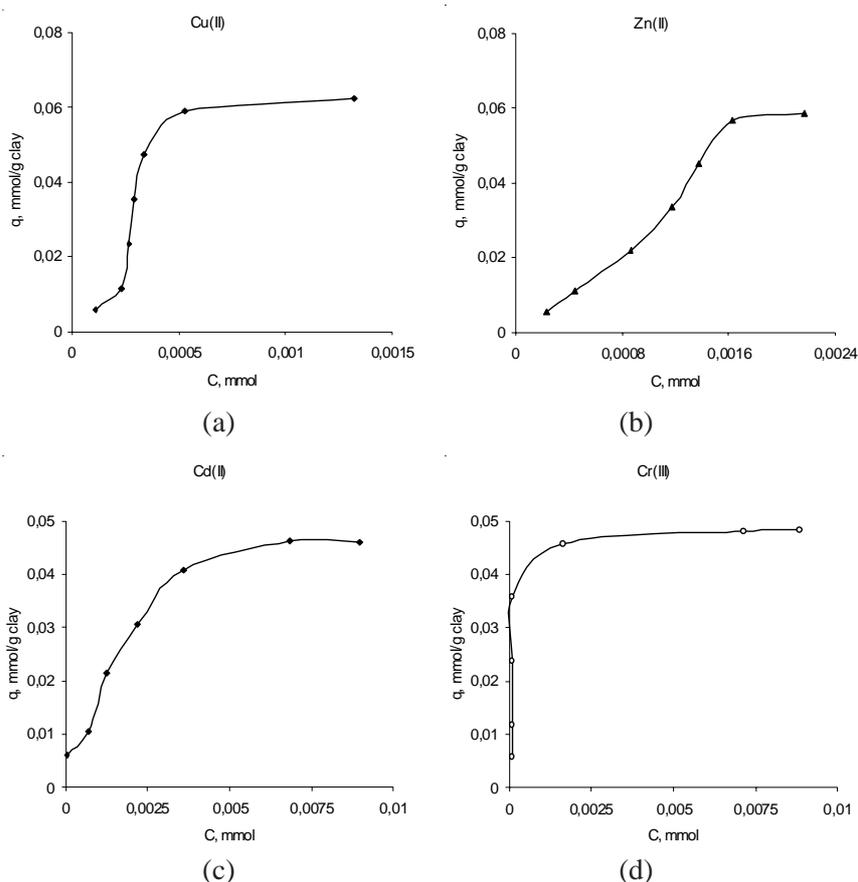


Fig. 5. Sorption isotherm of Cu(II), Cd(II), Zn(II) and Cr(III) ions on montmorillonite (Adsorption conditions; initial concentration of metals: 1×10^{-4} - 1×10^{-3} mol/L, amount of clay: 0.5 g, volume of adsorption medium: 30 mL, temperature: $20 \pm 1^\circ\text{C}$, adsorption time: 2 h)

$$\ln q = \ln q_m - \beta \varepsilon^2 \quad (6)$$

where q is the amount of sorption of metal ions in mmol/g, q_m the D-R monolayer capacity, β a constant related to sorption energy and ε is the Polanyi which is related to the equilibrium concentration as follows:

$$\varepsilon = RT \ln (1/C_e) \quad (7)$$

where T is the temperature in K and C_e is the equilibrium solution concentration of solute in mmol/mL. The slope of the plot of $\ln q$ vs. ε gives β (mol^2/J^2) and of the intercept yields the sorption capacity, (mmol/g). The

D-R parameters are listed in Table-1. According to following relationship, we could further obtain the sorption energy (E)

$$E = -1 / (-2\beta)^{1/2} \quad (8)$$

As shown in Table-1, the E values are 7.07 kJ/mol for Cu(II) sorption, 7.07 kJ/mol for Zn(II), 10.00 kJ/mol for Cd(II) sorption and 12.91 kJ/mol for Cr(III) sorption on the clay. They are the orders of an ion-exchange mechanism, in which the sorption energy lies within 8-16 kJ/mol, indicating that chemisorptions may play a significant role in the sorption process. The sorption capacity q_m in the D-R equation is found to be 1.82 mmol/g for Cu(II), 1.17 mmol/g for Zn(II), 0.08 mmol/g for Cd(II), and 0.08 mmol/g for Cr(III).

TABLE-1
SOME PARAMETERS CALCULATED FROM THE
DUBININ-RADUSHKEVICH (D-R)

Metals	q_m (mmol/g)	K (mol ² /kJ ²)	E (KJ/mol)	R ² (for D-R)
Cu(II)	1.819	1×10^{-8}	7.07	0.773
Zn(II)	1.166	1×10^{-8}	7.07	0.987
Cd(II)	0.084	5×10^{-9}	10.00	0.894
Cr(III)	0.079	3×10^{-9}	12.91	0.529

At low concentrations adsorption occurs mainly on the surface readily rather than in the pores. This fact does not conform to the volume filling assumption of D-R, therefore poor fitted results in the low concentration range¹², thus the D-R equation is mainly applicable in the low C_e region; e.g. 1×10^{-4} - 1×10^{-3} M for metal sorption in this work.

The Langmuir adsorption isotherm equation is as follows:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_0} + \frac{1}{Q_0 b} \quad (9)$$

where C_e (mg/L) is the equilibrium concentration, q_e (mg/L) is the amount of Cr(III) sorbed at equilibrium, Q_0 and b are Langmuir constants related to adsorption capacity and energy of adsorption, respectively.

A linear plot of C_e/q_e vs. C_e indicates that sorption follows the Langmuir isotherm model. Q_0 and b were calculated from the slope and intercept of the plots¹³.

The isotherms are well described by the Langmuir and Freundlich equation (Fig. 5). The values of Q_0 are 0.074 mmol/g for Cu(II), 0.109 mmol/g for Zn(II), 0.056 mmol/g for Cd(II) and 0.049 mmol/g for Cr(III) using montmorillonite. One of the essential characteristics of the Langmuir equation could be expressed by dimensionless constant called equilibrium parameter^{29,31}, R_L

$$R_L = 1/(1 + b C_0) \quad (10)$$

where C_0 is the highest initial solute concentration (mol/L). The value of R_L indicates the type of isotherm to be irreversible ($R_L = 0$), favourable ($0 < R_L < 1$), linear ($R_L = 1$) or unfavourable ($R_L > 1$). It is shown that the sorption of Cu(II), Zn(II), Cd(II) and Cr(III) metal ions on montmorillonite is favourable (Table-2).

The Freundlich model is defined by following equation^{3,27}

$$q = K C_e^{1/n} \quad (11)$$

where C_e and q are the concentrations of metal ions in solution and clay phase, respectively. Table-2 shows the adsorption coefficient K and Freundlich constant 'n'. The K in the Freundlich equation is related primarily to the capacity of the adsorbent for the adsorbate and 'n' is a function of the strength of adsorption. For the fixed values of C_e and 'n', the larger the value of K , the larger the capacity 'q'. For fixed values of K and C_e , the smaller the value of 'n', the stronger is the adsorption bond.

TABLE-2
SOME PARAMETERS CALCULATED FROM THE FREUNDLICH AND LANGMUIR ISOTHERM

Metals	Freundlich isotherms			Langmuir isotherms			
	k	n	R ²	Q _o	b	R ²	R _L
Cu(II)	0.161	0.612	0.896	0.074	4522.840	0.975	0.181
Zn(II)	0.653	0.892	0.987	0.109	207.199	0.920	0.828
Cd(II)	0.383	2.307	0.928	0.056	588.634	0.947	0.630
Cr(III)	0.182	3.919	0.515	0.049	5201.019	0.998	0.161

k, q_m , and Q_o , Adsorption capacity; mmol/g montmorillonite
n, K, E and b, constants

The Freundlich constant 'n' is also a measure of the deviation from linearity of the adsorption. If 'n' is equal to unity the adsorption is linear. This means that the adsorption sites are homogeneous in energy and no interaction occurs between the adsorbed species. These are the same as the assumptions for Langmuir model and implies that the degree of adsorption is unchanged with changes in solute concentration, *i.e.* the adsorption sites of the sorbent are not in limiting quantity and are not modified by the process of adsorption itself. If $n < 1$, this implies that increased adsorption modifies the sorbent in a manner that increases the sorption capacity, such as forming new adsorption sites. When $n > 1$ or becomes larger ($n \gg 1$), however, the adsorption bond becomes weak and the value of q changes significantly with small changes²⁸ in C_e .

It is clear that the Freundlich type isotherm is inadequate for the sorption process of Cu(II), Zn(II), Cd(II) and Cr(III) as shown by the values of correlation coefficient. Significantly, better results obtained for Langmuir type equation. Thus this simplified expression is valid not only in the cases

when cations in liquid and clay phase are singly charged, but also when they have unequal charges and compete with each other⁹.

For the concentration range studied, the Freundlich isotherm is somewhat better for Zn(II), as shown by the values of correlation coefficient. Isotherms of the adsorption process with metal ions depicted in Fig. 5 show that the experimental data fit both the Freundlich, $R^2 = 0.98$ and Langmuir, $R^2 = 0.99$, models. This suggests that adsorption tends to be in monolayer²⁹. The fact that the Langmuir equation fits the experimental data better than the Freundlich was expected, since the experimental data show a clear trend toward a value of maximum adsorption capacity (Table-2).

Effect of temperature

The adsorption mechanism (*i.e.*, chemical or physical) is often an important indicator to describe the type and level of interactions between the adsorbate and adsorbent. If adsorption decreases with increasing temperature it may be indicative of physical adsorption and reverse is generally true for chemisorption. However, there are a number of contradictory cases in the literature³⁰.

The amounts of sorption of single metal ions by montmorillonite are measured in a temperature range 20-65°C as shown in Fig. 6. The equilibrium partition constant K is calculated as follows:

$$K = q_e/C_e \quad (12)$$

K is defined as ratio of metal ion concentration on the montmorillonite to that in aqueous solution. The K for all metal ions was very high and it increased with temperature (Fig. 6). This is due to the endothermic adsorption of cations. The equilibrium constants for this reaction increase with temperature and the reaction products are favoured at high temperatures.

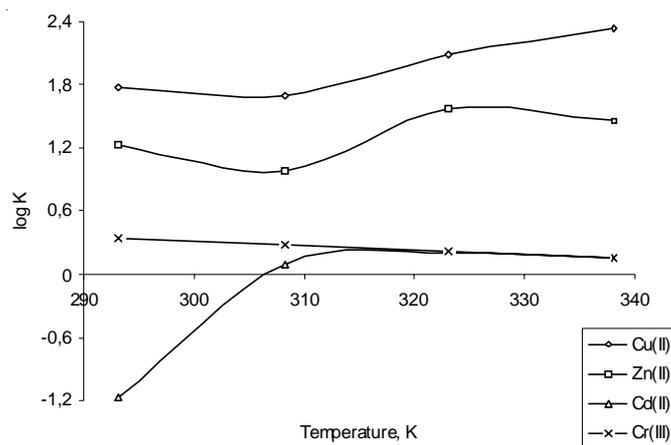


Fig. 6 The distribution coefficients of Cu(II), Cd(II), Zn(II) and Cr(III) ions on montmorillonite as a function of temperature (Adsorption conditions; initial concentration of metals: 1×10^{-3} mol/L, amount of clay: 0.5 g, volume of adsorption medium 30 mL)

Sorption kinetics of single metal ions

The kinetics of sorption of metal ions by montmorillonite was studied for its possible importance in treatment wastewater. The metal sorption on montmorillonite increased with time (Fig. 7). The sorption on montmorillonite appeared to be practically instantaneous. The metal amounts sorbed in the first half an hour correspond to 100% of equilibrium sorbed amounts. The sorption appeared insensitive to the nature of the metal ion in solution. This behaviour is mainly related to the low initial metal concentration/clay dose ratio with respect to the sorption capacities that this clay is able to achieve. Therefore, equilibrium time for metal sorption on montmorillonite appears to be dependent on initial metal concentrations as occurs for other sorbents^{7,31}.

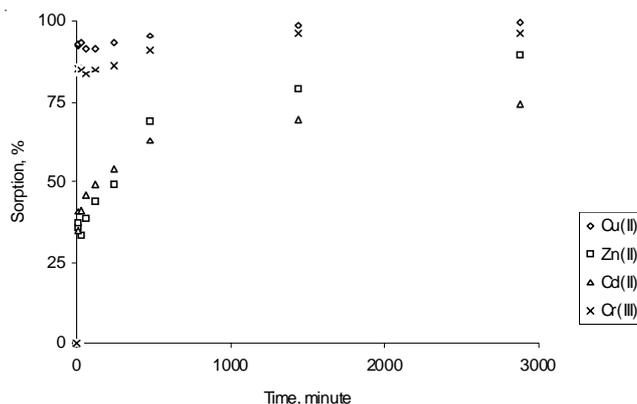


Fig. 7 Adsorption rate of Cu(II), Cd(II), Zn(II) and Cr(III) ions by montmorillonite from aqueous solution (Adsorption conditions; initial concentration of metals: 1×10^{-3} mol/L, amount of clay: 0.5 g, volume of adsorption medium 30 mL, temperature: $20 \pm 1^\circ\text{C}$)

Conclusion

In this study, the adsorption potential of Turkish clay was investigated for the removal of Cu(II), Zn(II), Cd(II) and Cr(III) ions in aqueous solutions. Sorption removal of Cu(II), Zn(II), Cd(II) and Cr(III) from single solutions using naturally available montmorillonite was studied. The following equilibrium and kinetic results were obtained. The D-R, Langmuir and Freundlich equations were used to describe the sorption isotherms of single solute systems. The sorption of metal ions on montmorillonite was rapid during the first 10 min and the equilibrium was attained within 2 h. The adsorption of metal ions on clay decreases with decreasing pH and increasing temperature. At low pH values, the hydrogen ion competes with the heavy metals towards the superficial sites and, moreover, the Si-O⁻ and

Al-O⁻ groups are less deprotonated and they form complexes with bivalent and trivalent ions in solution with greater difficulty. The results evidenced that the sorption capability of montmorillonite towards each metal ion examined is different in the various conditions and it varies as a function of pH in solution. It is necessary to consider these factors to study a real soil/solution system and effectively predict the fate of heavy metals in the environment. Sorption of Zn(II) ions on clay conformed to the linear form of Freundlich adsorption equation while Cu(II), Cd(II) and Cr(III) ions are on clay conformed to Langmuir equation. The results show that clay, which has a very low economical value, may be used effectively in removal of Cu(II), Zn(II), Cd(II) and Cr(III) ions from aqueous systems for environmental cleaning purposes. Also, montmorillonite clay minerals are relatively plentiful and they don't need any treatment. Therefore, montmorillonite may be used to remove trace amounts of heavy metals from aqueous solution.

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