Adsorption of Cadmium(II) Ions from Aqueous Solution onto Kaolinite and Metakaolinite

**Jean Serge ESSOMBA, Julius NDI NSAMI, Placide Desire BELIBI BELIBI,
Guy Merlain TAGNE and Joseph KETCHA MBADCAM***

Physical and Theoretical Chemistry Laboratory
Department of Inorganic Chemistry, Faculty of Science
University of Yaoundé I, P.O Box 812 Yaoundé, Cameroon
* Corresponding author

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Abstract

The removal of cadmium(II) ions from aqueous solution by adsorption on kaolinite (KAO.1) and metakaolinite (MKB) was investigated depending on the initial concentration, adsorbents dosage, initial pH of solution, and contact time. The influences of those factors have been experimentally verified by a batch method at (27±3 °C). These results have showed that the amount of cadmium(II) ions adsorbed increases with increased contact time and that equilibrium adsorption is reached in 10 minutes, the optimum value of pH was 8.0 and the effect of absorbent dose for the uptake of cadmium(II) ions by kaolinite and metakaolinite was found to decrease by increasing the adsorbent dose. The experimental results obtained are described by Langmuir, Freundlich and Dubinin-Kaganer-Radushkevich (D-K-R) isotherm models. The Langmuir and D-K-R adsorption isotherms described the adsorption data very well. The maximum adsorption capacity; $Q_{\max}$, determined from the Langmuir adsorption isotherm studies was found to be 7.407 and 9.174 mg/g for KAO.1 and MKB respectively. Pseudo-first order, pseudo-second order, Elovich and the intraparticle diffusion kinetic models were used to describe the kinetic data obtained. The experimental data fitted well to the pseudo-second order kinetic model, which indicates that

chemical adsorption is the rate-limiting step. The results indicate that kaolinite and metakaolinite adsorb cadmium(II) ions efficiently and could be employed as a low-cost alternative in waste water treatment for the elimination of cadmium(II) ions.

Keywords: adsorption, kaolinite, metakaolinite, cadmium, kinetic model, isotherm model

1. INTRODUCTION

Most of the metals are known to be toxic and half of these including cadmium, chromium, copper, lead, mercury, nickel, selenium, silver and zinc. These metals are released into the environment in quantities that pose a risk to human health (Rao et al., 2010).

Health metal contamination exist in aqueous waste streams from diverse industries such as metal plating manufacturing, batteries, as well as agricultural sources where fertilizers and fungidal sprays are intensively used (Shih et al., 2010). Cu, Zn, Hg and Cd are harmful waste introduced by industries that pose a risk of contamination groundwater and other water resources (Achour and Youcef, 2003). Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders (Tekere and Imamoglu, 2005). As far as cadmium is concerned, it causes serious renal damage anemia and hypertension (Mahvi et al., 2007).

The reduction of the pollutant to an acceptable level is necessary when toxic metals are present in solution (Cheng et al., 2012). Decontamination of surface and underground water supplies is a major concern and conventional methods such as: Chemical precipitation, Membrane Filtration (Reverse Osmosis and Electro dialysis), Electrolytic processes, Biosorption and Adsorption can be used for decontamination of effluents (Igwe et al., 2008; Zohre et al., 2010). These techniques which have been mentioned above, with the exception of adsorption are expensive and also have disadvantages such as incomplete metal removal, high reagent, energy requirements and generation of toxic sludge or other waste products that require proper disposal (Ketcha et al., 2009). Due to the problems mentioned above this research has been interested in using Adsorption since it is cheaper, making use of low cost and local Adsorbents, which are adapted in efficiently removing heavy metal ions found in low concentrations in solution.

In the present investigation local kaolinite clay obtained from Kribi in the South Region of Cameroon, and the metakaolinite obtained from heating it at 700 °C have been used as adsorbents for the removal of cadmium(II) ion. The effect of such factors as contact time, initial pH and initial concentration was investigated. The kinetics of cadmium(II) ion adsorption on both adsorbents was analyzed by various kinetic models. Experimental equilibrium data were fitted to the Langmuir, Freundlich and Dubinin-Kaganer-Radushkevich.

2. MATERIALS AND METHODS

2.1 Adsorbent

The kaolinite clay was collected from Kribi in the South Region of Cameroon. Particles of ore that passed through an 80 μm sieve were extensively washed with tap water to remove dirt and other particulate matter, followed by washing in distilled water. The resulting slurry was allowed to sediment and particles of size less than or equal to 2 μm were obtained. After complete drying in the sun, one part of the kaolinite was used to prepare metakaolinite. This was done by heating the part in a furnace at the rate of 5 $^{\circ}\text{C}/\text{min}$ to a temperature of 700 $^{\circ}\text{C}$. Heating was maintained at this temperature for 6 hours before being allowed to cool in the furnace to 40 $^{\circ}\text{C}$. The dried adsorbent was ground in a laboratory mortar and sieved in standard test sieves for use during adsorption experiments. The adsorbents were heated in an oven at 110 $^{\circ}\text{C}$ for 24 hours and allowed to cool in a desiccator containing CaCl_2 (drying agent) for 1 hour before being used in the adsorption process.

2.2 Adsorbent Characterization

Diffractometer model Bruker and Philips equipment were used to obtain the X-ray diffraction (XRD) pattern of kaolinite and metakaolinite samples respectively, to identify the crystalline compounds. Infrared spectra of the both samples were measured from 400 to 4000 cm^{-1} using a Bruker Alpha-P spectrometer with ethanol as solvent. The BET specific area and total pore volume of kaolinite and metakaolinite were determined by standard multipoint techniques of nitrogen adsorption, using TriStar 3000 V6.05A equipment. The chemical analysis of kaolinite and metakaolinite is obtained by Harrison method and X fluorescence. The specific area of the kaolinite and metakaolinite is obtained by volumetric equipment as described by Haul and Dumbgen.

2.3 Preparation of cadmium(II) Ion Solution

All reagents used in this study were of analytical grade. A stock solution of copper (II) ion of concentration 1000 ppm (1000 mg/L) was prepared by dissolving 1.8158 g of CdSO_4 in a 1000 mL volumetric flask. This was swirled until all the salt dissolved, transferred into a 1-L volumetric flask and completed with distilled water up to the mark. This solution was stirred on a magnetic stirrer for one hour to obtain homogeneity. All experimental solutions were prepared by diluting the stock solution to the required concentration.

2.4 Batch adsorption measurement

Batch adsorption was carried out at room temperature. The effect of contact time, mass of adsorbent, pH of solution and the initial concentration of cadmium (II)

ions were studied by using kinetic and equilibrium models. In each experiment, 20 mL of cadmium(II) ion solution of known concentration was mixed with a known mass of adsorbent. The pH of the mixture was adjusted either with 0.1 N HCl or 0.1 N NaOH as required before agitating for the required length of time. The concentration of cadmium(II) ion after adsorption was determined by using a direct complexometric titration method.

2.5 Adsorption Equilibrium Studies

For each run the adsorbent is mixed with 20 mL solution of nickel ion at different initial concentrations (50–150 mg/L). The suspensions were stirred for 15 minutes by using a magnetic stirrer. The amount of Cd^{2+} ion adsorbed at equilibrium, Q_e (mg/g) was calculated using equation:

$$Q_e = \frac{(C_0 - C_e)}{m} \times V \quad (1)$$

Where, C_0 and C_e are Cd^{2+} ion concentrations initially and at equilibrium respectively, V the volume of the solution (L) and m the adsorbent mass. The equilibrium data were then fitted by using the Langmuir, the Freundlich and Dubinin-Kaganer-Radushkevich adsorption isotherm models.

Langmuir Adsorption Isotherm Studies

The Langmuir adsorption equation is one of the most common isotherm equations for modeling equilibrium data in solid-liquid systems. This equation is valid for monolayer adsorption onto a surface with a finite number of identical sites which are homogeneously distributed over the adsorbent surface. The general form of the Langmuir equation is (Ketcha Mbadcam et al., 2011):

$$Q_e = \frac{Q_m K_L C_e}{(1 + K_L C_e)} \quad (2)$$

Where C_e is the equilibrium concentration of cadmium(II) ions (mg/L), Q_e is the amount of cadmium(II) ions adsorbed per unit mass of the adsorbent, K_L is the Langmuir adsorption constant (L/mg) and Q_m is the maximum amount of per unit mass of adsorbent to form a complete monolayer on the surface (mg/L). The linear form of this equation is as follows:

$$\frac{1}{Q_e} = \frac{1}{Q_m K_L C_e} + \frac{1}{Q_m} \quad (3)$$

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L , which is defined as:

$$R_L = \frac{1}{1 + KC_o} \quad (4)$$

The R_L value indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$) as shown in Table 3 in this work.

Freundlich Adsorption Isotherm Studies

Freundlich equation is an empirical equation based on the adsorption on a heterogeneous surface (Augustine et al., 2007):

$$Q_e = K_f C_e^{1/n} \quad (5)$$

Where, C_e is the equilibrium concentration of Cd^{2+} ion (mg/L), Q_e is the amount of Cd^{2+} ion bound to per gram of the adsorbent at equilibrium (mg/g), k_f and n are the Freundlich constants related to sorption capacity and sorption intensity of the sorbent respectively. The linear form of the Freundlich isotherm equation is:

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (6)$$

Dubinin-Kaganer-Radushkevich (D-K-R) Isotherm Studies

Langmuir and Freundlich isotherms are insufficient to explain the physical and chemical characteristics of adsorption. D-K-R isotherm is commonly used to describe the sorption isotherms of single solute systems. The D-K-R isotherm, apart from being analogue of Langmuir isotherm, is more general than Langmuir isotherm as it rejects the homogeneous surface or constant adsorption potential. The D-K-R isotherm is expressed as (Ketcha Mbadcam et al., 2012):

$$Q_e = Q_{\max} \exp \left[\frac{\left(\frac{RT \ln \left(1 + \frac{1}{C_e} \right) \right)^2}{-2E_a} \right] \quad (7)$$

Where, E_a : is the main energy of adsorption and gives information about the physical and chemical features of adsorption. The linear form of the D-K-R isotherm equation is:

$$\ln Q_e = \ln Q_{\max} - \beta \varepsilon^2 \quad (8)$$

Where, $\varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right)$ is called the Polanyi Potential.

2.6 Kinetic Adsorption Experiments

Kinetic adsorption experiments were conducted using a series of 20 mL solutions containing fixed amount of adsorbent and cadmium(II) ions (50 ppm). The solutions were vigorously agitated with a magnetic stirrer for increasing time intervals. At the end of each run, the solution is filtered and the Cd^{2+} ion concentration in the filtrate is determined. Kinetics models have been used to fit experimental data.

The Pseudo-First Order Model

The pseudo-first order equation of Lagergren is generally expressed as follows (Ketcha Mbadcam et al, 2012):

$$\frac{dQ_t}{dt} = k_1(Q_e - Q_t) \quad (9)$$

Where, Q_e and Q_t are the sorption capacity at equilibrium and at time t respectively (mg.g^{-1}) and K_1 is the rate constant of pseudo-first order sorption (L.min^{-1}). After integration and applying boundary conditions, $t = 0$ to $t = t$ and $Q_t=0$ to $Q_t=Q_t$ the integrated form of equation is:

$$\ln (Q_e - Q_t) = \ln(Q_e) - K_1 t \text{ or } \ln C_t = -K_1 t + \ln C_o \text{ after reduction} \quad (10)$$

Where,

K_1 is the rate constant of pseudo first order adsorption ($\text{mg}^{-1} \text{ min}^{-1}$).

The Pseudo-Second Order Model

If the rate of the sorption is a second order mechanism, the pseudo-second order chemisorption kinetic rate equation is expressed as (Augustine et al., 2007):

$$\frac{dQ_t}{dt} = k_2(Q_e - Q_t)^2 \quad (11)$$

Where k_2 is the rate constant of pseudo-second order sorption ($\text{g.mg}^{-1}.\text{min}^{-1}$). The integrated and rearranged form of equation is:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (12)$$

The Elovich Equation

The Elovich equation is general expressed as follows (Zora and Snezana, 2006):

$$\frac{dQ_t}{dt} = \alpha \exp(-\beta Q_t) \quad (13)$$

Where α is the initial sorption rate ($\text{mg}^{-1}.\text{g}.\text{min}^{-1}$) and β is the desorption constant (g.mg^{-1}) during any one experiment. The integrated and simplified equation is:

$$Q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \quad (14)$$

The Intraparticle Diffusion Model

The linear form of this equation is expressed as follows (Zora and Snezana, 2006):

$$\ln P = \ln k_{id} + a \ln t \quad (15)$$

Where, P is the percent removal of cadmium ions at time t , K_{id} is the intraparticle diffusion rate constant and a is a gradient whose value depends on the adsorption mechanism.

3. RESULTS AND DISCUSSIONS

3.1 Characterization of Adsorbents

The XRD (Fig.1.) shows that the major constituents of the KAO.1 are Anatase, illite, Quartz, Kaolinite, and Lepidocrocite. In the case of MKB there is complete disappearance of the more intense pics of kaolinite ($2\theta = 9^\circ$ and $2\theta = 67^\circ$), the amorphous phase on MKB confirmed the presence of metakaolinite. The chemical analysis (Table 1) shows that the major elements are SiO_2 and Al_2O_3 . BET measurements on the kaolinite and metakaolinite showed their surface area to be 20.1819 and 33.8331 m^2/g respectively. The pore volume of 0.0814 and 0.0935 cm^3/g for KAO.1 and MKB respectively. This means that when heating kaolinite, there is increasing in surface area and the pore volume

The transformation of kaolinite to metakaolinite is proved by the disparition of the band at $3688\text{-}3618\text{ cm}^{-1}$ on MKB sample wich corresponds to the dexhydroxylation of kaolinite and the presence of the band at $1698\text{-}1521\text{ cm}^{-1}$ of the molecules H_2O on the MKB sample is due to the hygroscopic property of metakaolinte after heating it at $700\text{ }^{\circ}\text{C}$ (Tchakouté et al., 2012).

Fig.2. FTIR Spectra of KAO.1 and MKB Samples

Table 1: Chemical Composition of the KAO.1 and MKB

OXIDES (%)	KAO.1	MKB
SiO ₂	49.52	46.99
Al ₂ O ₃	32.04	34.66
Na ₂ O	0.05	10.55
TiO	0.99	0.94
Fe ₂ O ₃	0.75	0.79
K ₂ O	0.69	0.68
MgO	0.13	0.16
CaO	0.04	0.06
l.i	15.73	5.05
Total	99.94	99.91

3.2 Effect of Contact Time

In order to determine the effect of the contact time, 0.5 g of the adsorbent was stirred with a 20 mL solution of cadmium (II) ions of initial concentration 50 mg/L for a time interval between 5 to 40 minutes for KAO.1 and MKB with pH=8. The experimental results obtained for the adsorption of cadmium ions

during varying contact times are shown in (Fig.3). It is clear from the graph that amount of uptake Q_t (mg gm^{-1}) increased with increased contact time and after certain period of time; it reached to a constant value beyond which no further adsorption took place. The results showed that, the adsorption was fast at initial stage of contact period and after that near the equilibrium it became slower. With the lapse of time, the surface adsorption sites were exhausted. The remaining vacant sites were difficult to be occupied by the cation due to repulsive forces between adsorbate present in solid and bulk phases (Soug et al., 2009). Adsorptions reach equilibrium within 10min for KAO.1 and MKB as represented by Fig.3.

Figure 3: Effect of Agitation Time on the Adsorption of Cadmium(II) Ions on KAO.1 and MKB.

3.3 Effect of the Amount of Adsorbents

To study the effect of adsorbent dosage on the adsorption of cadmium(II) ions, a series of adsorption experiments were carried out with different adsorption dosages varying from 0.1 to 1.1 g at initial concentration of 50 ppm. The effect of adsorbent dose for the uptake of cadmium(II) ions by kaolinite and metakaolinite was found to decrease by increasing the adsorbent dose. This may be allocated to the fact that as the amount in grams of adsorbent is increased the total surface area available for the adsorption of cadmium(II) ions reduces as a result of overlapping or aggregation of adsorption sites (Ndi Nsami and Ketcha Mbadcam, 2013). Furthermore, maximum quantity adsorbed, $Q_e = 6.875$ and 7.638 mg/g for KAO.1 and MKB respectively, was reached by 0.1 g of each of the adsorbents (see Figure 4).

Figure 4: The effect of Absorbent dose for the Uptake of Cadmium(II) Ions.

3.4 Effect of Initial pH on Cadmium(II) Ion Adsorption

pH is an important parameter influencing heavy metal adsorption from aqueous solutions. It affects both the surface charge of adsorbent and the degree of ionization of the heavy metal in solution (Rao et al., 2010). (Fig.5) represents the effect of initial pH of the solution on the adsorption of cadmium(II) onto KAO.1 and MKB using 50 mg/L initial cadmium (II) concentration and 0.5 g of adsorbent, in the present study the pH was varied between 3 and 10.

Fig.5. Effect of pH on Cadmium(II) Ion Adsorption on KAO.1 and MKB

The lower adsorption at pH less than 6 this may be attributed to the competition between the hydrogen and cadmium ions on the sorption sites (Malferrari et al., 2007). At pH values higher than 8.0 insoluble cadmium hydroxide starts precipitating from the solutions making true sorption studies impossible (Yang et al., 2001; Yu et al., 2007). According to the Fig.5 we saw that the optimum adsorption pH was between 7 and 8 for KAO.1 and MKB, the working pH value for cadmium removal onto KAO.1 and MKB was chosen as 8.0 and the other adsorption experiments were performed at this pH value.

3.5 Kinetic Modeling of Cadmium(II) Ions Adsorption

The kinetics of the adsorption of cadmium(II) ions has been studied to analyze the adsorption rates of cadmium(II) ions onto Kaolinite and Metakaolinite. Four simple kinetic models were tested. The straight-line plots are presented on Figures 6 to 9, while the parameters for these models are given in Table 2. It can be noted that with Kaolinite and Metakaolinite, the elimination of the cadmium(II) ions adequately follows the kinetic model of the pseudo second order, giving a very good coefficient of correlation, $R^2=0.999$, this implies that cadmium (II) ions adsorption on both adsorbent may occur through a chemical process involving the valence forces of the shared or exchanged electrons (Ecboon et al., 2010). This means that the chemisorption reaction or an activated process becomes more predominant in the rate-controlling step for the cadmium system.

Figure.6: Linearized Pseudo-First Order Plots for $C_0=50$ mg/L, $V=20$ mL, $pH=8.0$, $m=0.5$ g Adsorbent

Figure.7: Linearized Pseudo-Second Order Plots for Co=50 mg/L, V=20 mL, pH=8.0, m=0.5 g Adsorbent

Figure.8: Linearized Elovich plots for Co=50 mg/L, V=20 mL, pH=8.0, m=0.5 g Adsorbent

Figure.9: Linearized Intraparticle Diffusion Plots for Co=50 mg/L, V=20 mL, pH=8.0, m=0.5 g Adsorbent.

Table 2: Kinetic Models Parameters of Adsorption

Models	Parameters	Adsorbents	
		KAO.1	MKB
Pseudo-first order	R^2	0.241	0.320
	K_1 (min^{-1})	0.007	0.005
Pseudo-second Order	R^2	0.999	0.999
	h (mg/g min)	3.663	3.185
	K_2 (g/mg. min)	1.663	1.189
	Q_e (mg/g)	1.484	1.637
Elovich	R^2	0.142	0.205
	β (g/mg)	20.833	16.667
	α (mg/g min) $\times 10^{-9}$	7.163	0.803
Intra-particle Diffusion	R^2	0.904	0.900
	K_{id} (min^{-1})	64.715	72.313
	a (mg/g)	0.036	0.030

3.6 Adsorption Isotherms Studies

Data obtained from these studies have been tested with the Langmuir, Freundlich and Dubinin-Kaganer-Raduskevich linearized equations. These results are summarized in table 3. The studies were conducted with 0.5 g of each adsorbent. The results are shown in Figure 10. The isotherm for the adsorption of cadmium(II) ions on kaolinite and metakaolinite is of type II. It represents the formation of a monolayer followed by multi-layer. This type of isotherm is a characteristic of adsorption on adsorbents that are nonporous solids.

The adsorption parameters in Table 3 show that the data both fitted Langmuir and D-K-R isotherms but fitted better in D-K-R isotherm equation by comparing the R^2 values. This implies that a monolayer sorption proceeds over a surface containing a finite number of adsorption sites and uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface. The separation factor R_L (Table 4) value indicates that cadmium(II) ion adsorption on both adsorbent is favourable for the higher initial cadmium(II) ion concentrations than for the lower ones (Zaid and Mohammed, 2009). The positive value of sorption energy ($E_a = 158.114$ and 129.100 kJ/mol) indicate that the adsorption of cadmium(II) ions by KAO.1 and MKB respectively is endothermic and the values are more than 8 KJ/mol the limited value for physical adsorption, this also indicates the strong interaction between the both adsorbents and the cadmium (II) ions (El-Said et al., 2010).

The $Q_{\max}$ values obtained for the present system in comparison with those reported earlier for sorption of cadmium(II) ions onto various adsorbents (Table 5) revealed that KAO.1 and MKB were effective adsorbent in removing cadmium(II) ions.

Figure.10: Adsorption Isotherms of Cadmium(II) Ions on KAO.1 and MKB for $C_0=50$ mg/L, $V=20$ mL, $pH=8.0$, $m=0.5$ g Adsorbent

Figure.11: Linear Plot of Langmuir Model, for $C_0=50$ mg/L, $V=20$ mL, $pH=8.0$, $m=0.5$ g Adsorbent

Figure 12: Linear Plot of the model of Freundlich for $C_0=50$ mg/L, $V=20$ mL, $pH=8.0$, $m=0.5$ g Adsorbent

Figure.13: Linear Transform of the Model of D-K-R for Co=50 mg/L, V=20 mL, pH=8.0, m=0.5 g Adsorbent

Table 3: Isotherm Models Parameters of Adsorption

Models	Parameters	Adsorbents	
		KAO.1	MKB
Langmuir	R^2	0.874	0.911
Isotherm	$Q_{\max}$ (mg/g)	7.407	9.174
	$K(x10^{-3}L/mg)$	8.706	9.790
Freundlich	R^2	0.896	0.867
Isotherm	$1/n$	1.253	1.169
	$K_F(L/g)$	0.048	0.061
D-K-R Isotherm	R^2	0.910	0.969
	$Q_{\max}$ (mg/g)	1.061	1.142
	E_a (KJ/mol)	158.114	129.100

Table 4: Langmuir Isotherm with Separation Factor (R_L)

Concentration (ppm)	50	75	100	125	150
Adsorbent	R_L				
KAO.1	0.697	0.605	0.535	0.479	0.434
MKB	0.671	0.577	0.505	0.450	0.405

Table 5: Comparison of Adsorption Capacity of Cadmium(II) Ions with others Adsorbents

Adsorbents	Q _m (mg/g)	References
Montmorillonite	0.150	Ecboon et al., 2010
Rice Husk Ash	3.84	El-Said et al., 2010
Activated carbon	63. 28	Achour and Youcef, 2004
KAO.1	7.407	Present study
MKB	9.174	Present study

4. CONCLUSION

In this study, Kaolinite (KAO.1) and Metakaolinite (MKB) have been used as adsorbents for the elimination of cadmium(II) ions from aqueous solutions. Adsorption was influenced by various parameters such as contact time, absorbent dosage, initial pH and initial cadmium(II) ions concentration. The adsorbent dosage had an effect on the adsorption process of cadmium(II) ions. The maximum uptake of cadmium(II) ions occurred at an initial pH of 8.0. Adsorption was increase with increasing initial cadmium(II) concentration 50-150 mg/L. The maximum cadmium(II) ion uptake capacity was 7.407 and 9.174 mg/g for KAO.1 and MKB, respectively. The kinetics of cadmium(II) ion adsorption nicely followed pseudo second-order for both adsorbents. Langmuir and D-K-R isotherms could be used to describe adsorption cadmium(II) by both adsorbents. Kinetic and isotherm studies revealed that KAO.1 and MKB can be effectively employed for the adsorption of cadmium(II) ions.

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