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Kaolinite and Cd^{2+} Transport and Interaction in Sand Media: Batch and Column Experiments

Rakkreat Wikiniyadhanee[1, 2], Srilert Chotpantarat[2, 3, 4, *], and Say Kee Ong[5]

[1] *International Postgraduate Programs in Environmental Management, Graduate School, Chulalongkorn University, Bangkok, Thailand*
[2] *Research Program of Toxic Substance Management in the Mining Industry, Center of Excellence on Hazardous Substance Management (HSM), Chulalongkorn University, Bangkok, Thailand*
[3] *Department of Geology, Faculty of Science, Chulalongkorn University, Bangkok, Thailand*
[4] *Research Unit of Site Remediation on Metals Management from Industry and Mining (Site Rem), Chulalongkorn University, Bangkok, Thailand*
[5] *Department of Civil, Construction and Environmental Engineering, Iowa State University, Ames, IA, USA*

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ABSTRACT

Batch sorption and column transport experiments were used to investigate kaolinite colloids and Cd(II) ions (Cd^{2+}) co-transport in water-saturated sand media. Batch sorption was used to evaluate Cd^{2+} sorption onto kaolinite clay as a function of the initial Cd^{2+} concentrations and solution ionic strengths as well as determining the equilibrium sorption isotherms. For very low ionic strength (~0 mM), the Langmuir isotherm was the more suitable isotherm for the experimental results while at higher ionic strength (> 0.45 mM) the Freundlich isotherm was the more suitable isotherm. The sorption parameters, K_F, K_L, n, and q_m, were found to decrease with increasing ionic strength. For column transport experiments, Cd^{2+}-bearing kaolinite colloids were found to be retained more in the column for an increase in the solution ionic strength. For example, the steady state breakthrough rate of Cd^{2+}-bearing kaolinite colloids was 0.2 C_i/C_0 for an ionic strength of 0.0 mM compared to about 0.05 C_i/C_0 for ionic strengths of 0.45 and 0.9 mM. This increased Cd^{2+}-bearing kaolinite colloid retention trend with increased solution ionic strength was likely due to the reduced zeta-potential and increased size of the colloid particles at higher ionic strengths. Consequently, the kaolinite-facilitated Cd^{2+} concentration decreased with increasing solution ionic strength. In summary, solution ionic strength had a strong effect on the transport behavior of kaolinite-facilitated Cd^{2+} and Cd^{2+}-bearing kaolinite colloids through the sand column.

Key words: Cadmium, Colloids, Facilitated, Ionic strength, Kaolinite

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1. INTRODUCTION

Colloids are ubiquitous in the subsurface environment (Sen et al. 2004). Most natural porous media, such as aquifer materials, contain some colloidal particles stuck onto the surfaces of bigger immobile particles and/or those in a flocculated state (Roy and Dzombak 1996). Colloids are generally considered to be particles of approximately 10 nm to 10 μm diameter in size (DeNovio et al. 2004) and are commonly found in natural subsurface environments at concentrations ranging from 35 - 100 mg L^{-1} (Zhuang et al. 2003).

Soil colloids transport in the subsurface environment can potentially enhance the migration of contaminants that are adsorbed onto colloids (McCarthy and McKay 2004), playing a significant role in facilitating the transport of these contaminants. These contaminants may be cations, anions, non-polar and/or polar organic compounds. Metal cations are especially influenced by this "colloid-facilitated transport" (Kersting et al. 1999) as they have an affinity to be adsorbed onto soil colloids, such as clay minerals, which then act as a carrier (Sen et al. 2004). Heavy metals adsorbed onto colloid particles can be transported through subsurface media at a similar or greater rate than the mobile aqueous phase (McGechan and Lewis 2002). Under normal water flow conditions colloidal

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* *Corresponding author*
E-mail: csrilert@gmail.com

particles in the subsurface are attached mainly to porous media. However, they may be released into the aqueous phase and be transported through a porous medium as the electrostatic repulsion between particles is increased, such as with a lowering of the solution ionic strength (Roy and Dzombak 1996). In recent years, nanoscale particles such as carbon nanotubes (Zhang et al. 2011), titanium dioxide (Fang et al. 2011) and hydroxyapatite nanoparticles (nHAP; Wang et al. 2011), have attracted attention due to their potential toxicity and possible colloid-facilitated contaminant transport in the subsurface environment. Wang et al. (2011) reported the ionic composition and ionic strength effects on the facilitated transport of Cu^{2+} with nHAP, where the amount of nHAP attached to the sand column increased with increasing NaCl and $CaCl_2$ concentrations in the solution.

Over the last ten years colloids from mine wastes and soils have attracted interest due to their high sorption affinity for heavy metals and possible risks to the environmental and human health. However, there are only a few articles on the transport and fate of natural colloids (Zhou et al. 2011), the influence of colloids in the transport of heavy metals in groundwater and the effects of the ionic strength on the co-transport of kaolinite colloids and Cd(II) ions (Cd^{2+}). An understanding of colloid-facilitated transport is necessary for predicting the transport of heavy metals in the groundwater systems of contaminated sites such as acid mine drainage sites. The objective of this study was to evaluate the solution ionic strength effects on the transport of kaolinite-facilitated Cd^{2+} and kaolinite colloids.

2. MATERIALS AND METHODS

2.1 Composition of Ottawa Sand and Kaolinite Clay

Ottawa sand and kaolinite clay were purchased from Fisher Chemical (Loughborough, England) and analyzed for their mineral composition using X-ray diffraction (XRD) (Bruker AXS D8, Karlsruhe, Germany). Prior to use Ottawa sand was cleaned to remove any metal oxides and adsorbed particles on the sand surface. Kaolinite clay was analyzed for its chemical composition in terms of oxide contents using X-ray fluorescence (XRF) (Bruker AXS S4, Karlsruhe, Germany).

2.2 Preparation of Kaolinite Suspension

Kaolinite clay (0.25 g) was added to 500 mL of ultrapure water (18.2 MΩ) in a 500 mL volumetric flask, shaken and ultrasonicated in an ultrasonic bath for 30 min and then left to stand for 24 h. The upper half of the suspension was then siphoned into a new container.

2.3 Preparation and Characterization of Cd^{2+}-Bearing Kaolinite Suspension

Cd^{2+}-bearing kaolinite suspensions in solution of different ionic strengths were prepared as follows. The kaolinite colloids concentration in the kaolinite suspension (section 2.2) was gravimetrically measured, diluted to 250 mg L^{-1} and then 200 mL of this 250 mg L^{-1} kaolinite suspension was added to a 500 mL volumetric flask and ultrasonicated for 1 min. Five mL of 1000 mg L^{-1} Cd^{2+} [as $Cd(NO_3)_2$] and 295 mL of varying $CaCl_2$ solution concentrations were added, giving a final concentration of kaolinite colloids and Cd^{2+} of 100 and 10 mg L^{-1}, respectively, in solution ionic strengths of 0.00, 0.45, 1.8, and 3.6 mM. $CaCl_2$ was used as the background ions because Ca^{2+} is one of the most common divalent cations found in natural waters. In addition, Cd is predominantly present as Cd^{2+} in the acidic pH range for low Cl^- (1 mM) concentrations (Basualto et al. 2006). The highest chloride ion concentration in the co-transport experiments was 0.6 mM. Therefore, the Cd-Cl complexes formation effects on Cd adsorption were minimal and can be ignored. The zeta-potential and particle sizes of the kaolinite colloids under different solution ionic strengths were measured using a Zetasizer nano (ZS, Malvern, England).

2.4 Batch Sorption of Cd^{2+} onto Kaolinite

A stock solution of 1000 mg L^{-1} of Cd^{2+} was prepared from $Cd(NO_3)_2$. Batch sorption, as per the method reported previously (Jiang et al. 2010), were performed using 60 mL capped polypropylene bottles on a shaker at 200 rpm at room temperature (25°C). The bottles contained 20 mL of varying concentrations of Cd^{2+} solutions (0 - 150 mg L^{-1}) in different ionic strength solutions (0.0 - 3.6 mM) by adding an appropriate volume of $CaCl_2$ solution to the Cd^{2+} solution (all at pH 5.6) and 0.5 g of kaolinite clay. The polypropylene bottles were agitated for 24 h. Batch sorption experiments for each ionic strength were conducted in triplicate (Wikiniyadhanee 2012). The bottles were then centrifuged at 9000 rpm for 15 min, and the supernatant harvested and filtered through 0.22 μm Polytetrafluoroethylene (Teflon) (PTFE) syringe filters. The Cd^{2+} concentration in the filtrate (aqueous phase) was then measured using atomic absorption spectrophotometry (AAS) (Perkin Elmer AAnalyst 800, Waltham, MA, USA). Kinetic experiment (using 25 g L^{-1} kaolinite clay) was performed for IS of 0.0 mM with an initial Cd^{2+} concentration of 10 mg L^{-1} without any buffer for 24 h (Wikiniyadhanee 2012). The amount of Cd^{2+} adsorbed by kaolinite clay was determined using the following equation [Eq. (1)].

$$q_e = \frac{V(C_0 - C_e)}{m} \tag{1}$$

Where q_e is the Cd^{2+} concentration adsorbed onto the kaolinite clay (mg g^{-1}) at equilibrium time, C_0 is the initial Cd^{2+} concentration (mg L^{-1}), C_e is the Cd^{2+} concentration in aqueous phase at equilibrium time (mg L^{-1}), V is the volume of

Cd^{2+} solution (L), and m is the mass of kaolinite clay (g).

The sorption data were regressed and fitted using the Freundlich and Langmuir isotherms. The Freundlich isotherm is based on sorption onto heterogeneous surfaces (Freundlich 1906) and is represented in a linear form by Eq. (2);

$$\log(q_e) = \log(K_F) + \frac{1}{n}\log(C_e) \tag{2}$$

where, C_e is the equilibrium concentration of Cd^{2+} (mg L^{-1}), q_e is the amount of adsorbed Cd^{2+} per mass of kaolinite clay (mg g^{-1}), n is the Freundlich equation exponent, and K_F is the Freundlich constant.

The Langmuir model is based on sorption onto a homogeneous surface with no interaction between adsorbed ions (Langmuir 1918). The equation may be rearranged in a linear form as shown in Eq. (3);

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \tag{3}$$

where q_e is the amount of adsorbed Cd^{2+} per mass of kaolinite clay (mg g^{-1}), C_e is the Cd^{2+} concentration at equilibrium time (mg L^{-1}), q_m is the maximum amount of adsorbed Cd^{2+} per mass of kaolinite clay (mg g^{-1}), and K_L is the Langmuir constant.

The Chi-square test, the sum of square of the differences between data estimated from the models and data observed from experiments (Ho and Ofomaja 2006), was used to assess the appropriate isotherm for adsorption. Equation (4) is shown below:

$$\chi^2 = \sum\frac{(q_e - q_{e,\,model})^2}{q_{e,\,model}} \tag{4}$$

Where $q_{e,\,model}$ is the amount of adsorbed Cd^{2+} per mass of kaolinite clay [calculated from the model (mg g^{-1})] and q_e is the experimental data at equilibrium (mg g^{-1}). The value of χ^2 should be a small number if the observed data and data calculated from models are the same. On the other hand, if the data obtained from the experiments and models are different the value of χ^2 will be a large number.

2.5 Sand Column Transport Experiments

The acrylic column used had an inner diameter and length of 2.5 and 10 cm, respectively. The influent solution was pumped into the bottom of the column. The column was packed with Ottawa sand with an effective porosity of about 0.32 and a bulk density of 1.51 g cm^{-3}. The seepage velocity was set to approximately 1.56 ± 0.07 m day^{-1}. Prior to use, the packed columns were equilibrated by flushing with several pore volumes (PVs) of ultrapure water, followed by at least 5 PVs of the Cd^{2+}- and kaolinite-free background solution at the same pH (pH ~5.6) as the Cd^{2+}-bearing kaolinite suspension. In this acidic pH range the major Cd species in the solution was Cd^{2+} (Naidu et al. 1994). Cd^{2+}-bearing kaolinite suspension in the influent tank was stirred while being pumped into the column in an upward direction via a piston pump (FMI lab QG6, Syosset, NY, USA) at a constant velocity of 1.56 ± 0.07 m day^{-1} for 13 PVs. After that several PVs of the Cd^{2+}- and kaolinite-free background solution (same pH and ionic strength) were applied into the column. The effluents were collected in 16 mL tubes at regular time intervals using a fraction collector (GE Frac-920, Buckinghamshire, England).

Bromide was used as a conservative tracer using the same experimental procedures as the column transport experiments above. An electrical conductivity meter (HACH sensION5, Loveland, CO, USA) was used to measure the bromide concentrations. A calibration curve (NaBr concentration against electrical conductivity) was established from serial dilutions of 1 M NaBr and was linear with a coefficient of determination (R^2) of 0.999.

2.6 Analytical Procedures

The Cd^{2+}-bearing kaolinite colloid concentrations in the effluent were measured using a UV/vis spectrophotometer (GENESYS 10S, Thermo Scientific, Loughborough, England) at a wavelength of 350 nm. A calibration curve was established by diluting the 250 mg L^{-1} Cd^{2+}-bearing kaolinite suspension to various concentrations. To determine the total Cd^{2+} concentration 1 mL of the sample was added to 2 mL of 14.5 M HNO_3 to digest the kaolinite colloids in the solution. Aliquots of the effluent were centrifuged at 9000 rpm for 15 min. The supernatant was then harvested and filtered through 0.22 μm PTFE syringe filters to measure the Cd^{2+} concentration in the filtrate (aqueous phase) using AAS. The kaolinite-facilitated Cd^{2+} concentration was then determined as the difference between the total and dissolved Cd^{2+} concentration.

3. RESULTS AND DISCUSSION

3.1 XRD and XRF Analyses

XRD analysis revealed that the kaolinite clay and Ottawa sand used comprised of kaolinite mineral and quartz mineral, respectively (Fig. 1). In addition, the XRF analysis revealed that the major oxides in the kaolinite clay were SiO_2 and Al_2O_3 (total ~84.7%) with 13.6% loss on ignition and several minor components (Table 1).

3.2 Batch Sorption of Cd^{2+} by Kaolinite

Results from the kinetic sorption experiment found that sorption equilibrium was reached in 24 hours (data not shown). The batch sorption assays revealed that Cd^{2+} sorption onto kaolinite clay was non-linear and the amount of Cd^{2+} adsorbed by the kaolinite clay increased with increasing initial Cd^{2+} concentrations (0 - 100 mg L^{-1}). In constrast, the amount of Cd^{2+} adsorbed by the kaolinite clay decreased with increasing solution ionic strength (Fig. 2). The final pHs of the batch experiments were generally acidic (pH < 7), suggesting that the dominant Cd^{2+} species in the suspensions under different ionic strengths was Cd^{2+} (Srivastava et al. 2004; Naidu et al. 1994). Based on the initial and final pHs, buffering was not necessary in our batch experiments.

The Freundlich and Langmuir isotherm models were used to analyze the experimental data and the results are summarized in Fig. 2 and Table 2. With respect to the Freundlich isotherm model, data for Cd^{2+} sorption onto kaolinite clay fitted very well to the Freundlich equation (R^2 ranged from 0.876 - 0.973). The K_F value for an ionic strength of 0.0 mM was 1.70-, 2.60-, and 4.09-fold higher than that in the 0.45, 0.9, and 1.8 mM solutions, respectively, (K_F declined from 0.794 - 0.194 L g^{-1}), suggesting that the sorption capacity of Cd^{2+} by kaolinite clay decreased with increasing solution ionic strength. This is likely due to a reduction in

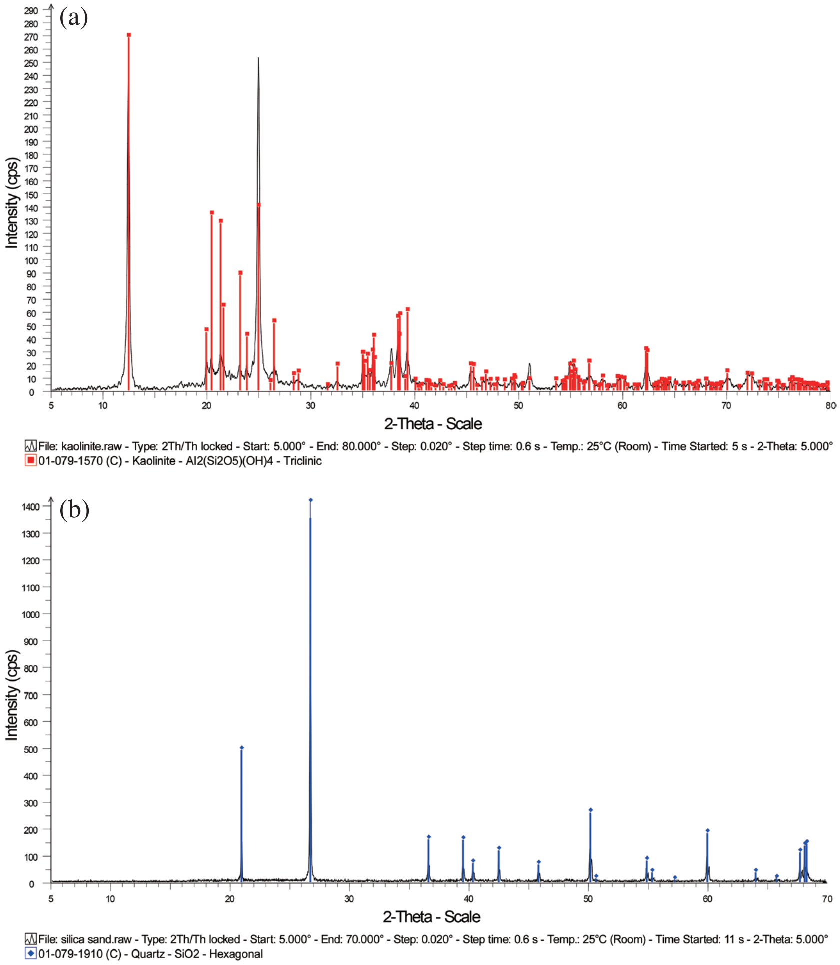

Fig. 1. X-ray diffraction pattern of kaolinite clay (a) and Ottawa sand (b). (Color online only)

Table 1. Chemical composition of kaolinite clay.

Constituents	Quantity (%)	Constituents	Quantity (%)
Al_2O_3	39.63	P_2O_5	0.121
SiO_2	45.08	CaO	0.017
Fe_2O_3	0.499	SO_3	0.0791
K_2O	0.127	TiO_2	0.646
Na_2O	0.195	Loss on ignition	13.6

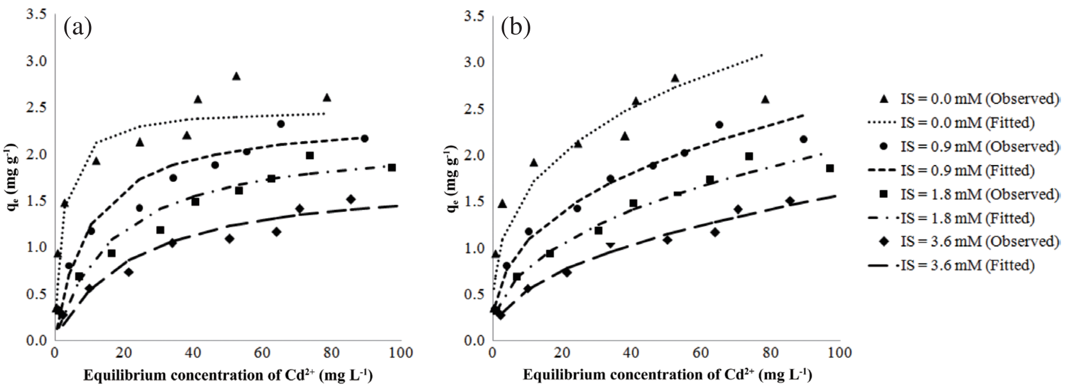

Fig. 2. Measured and fitted curves of Freundlich isotherm for adsorbed amount of Cd^{2+} on kaolinite clay under the ISs of 0.0, 0.9, 1.8, and 3.6 mM (a), and measured and fitted curves of Langmuir isotherm for adsorbed amount of Cd^{2+} on kaolinite clay under the ISs of 0.0, 0.9, 1.8, and 3.6 mM (b).

Table 2. Freundlich and Langmuir isotherm parameters for Cd^{2+} sorption onto kaolinite clay.

Ionic strength (mM)	**Freundlich isotherm**				**Langmuir isotherm**			
	1/*n*	**K_F (L g^{-1})**	**R^2**	**χ^2**	**q_m (mg g^{-1})**	**K_L (L g^{-1})**	**R^2**	**χ^2**
0.0	0.300	0.7940	0.876	0.488	2.477	0.4660	0.937	0.289
0.9	0.367	0.4660	0.973	0.053	2.369	0.1110	0.927	2.667
1.8	0.414	0.3050	0.961	0.059	2.108	0.0680	0.947	0.260
3.6	0.454	0.1940	0.963	0.045	1.817	0.0420	0.939	0.147

negative charges on the adsorbent surface for an increase in ionic strength, resulting in a reduction in the sorption of metal ions (Unuabonah et al. 2008). In addition, *1*/*n* values tended to increase with increasing solution ionic strength, being 1.22-, 1.38-, and 1.51-fold larger at 1.8 mM than at 0.9, 0.45, and 0.0 mM, respectively, indicating that the preferential sorption of Cd^{2+} was decreased at higher ionic strengths (Shahmohammadi-Kalalagh et al. 2011).

The maximum sorption capacity of kaolinite clay declined by 1.36-fold and the K_L value was reduced by 11.0-fold (from 2.477 - 1.817 mg g^{-1}) with increasing ionic strength from 0.0 - 3.6 mM, indicating that an increase in solution ionic strength reduced the sorption of Cd^{2+} by kaolinite clay. This is in accord with previous results where, for example, Cd^{2+} removal decreased as the ionic strength increased (Reed et al.1992).

For zero or low ionic strength (0.0 mM), the Langmuir isotherm fitted the experimental data of Cd^{2+} sorption better than the Freundlich isotherm. This may be due to no ions competing with Cd^{2+} for certain sites, suggesting that the sorption of Cd^{2+} appeared to be monolayer coverage or less (Naidu et al. 1994). However, increasing the solution ionic strength is often attributed to competition between Ca^{2+} and Cd^{2+} for surface sites, leading to a decrease in the sorption of Cd^{2+}. As a result, under high solution ionic strengths the Freunlich isotherm fitted well the sorption data, indicating that more than one type of active sites (heterogeneous and nonspecific surfaces) may be involved in the sorption of Cd^{2+} (Ho et al. 2002; Adebowale et al. 2006; Jiang et al. 2010). The Chi-square test was applied to confirm the best-fit isotherm for the batch sorption experiments (Ho and Ofomaja 2006). In addition, data for sorption experiments at IS = 3.6 mM were added to confirm that the Freundlich isotherm was a better isotherm than the Langmuir isotherm at high ionic strength. The results of χ^2 were in line with the R^2 value, for example, the Freudlich isotherm gave a better fit at ionic strength of 3.6 mM with a higher R^2 (0.963) and lower χ^2 (0.045) values.

3.3 Column Transport Experiment

Cd^{2+}-bearing kaolinite colloids transport through sand columns under different solution ionic strengths (0, 0.45, and 0.9 mM) are summarized in Fig. 3a. The Cd^{2+}-bearing kaolinite colloids showed a steady state breakthrough rate of 0.2 C_i/C_0 for a solution of 0.0 mM ionic strength and decreased at higher ionic strengths (0.45 and 0.9 mM) to a concentration of Cd^{2+}-bearing kaolinite colloids of less than 0.05 C_i/C_0. The zeta-potentials of Cd^{2+}-bearing kaolinite colloids decreased (less negative) and the particle size increased markedly (2.70- and 2.92-fold) in size as the ionic strength increased from 0.0 to 0.45 and 0.9 mM, respectively (Table 3). This leads to Cd^{2+}-bearing kaolinite colloids retarding Cd^{2+} transport through the packed sand column.

Increased solution ionic strength promoted Cd^{2+}-bearing kaolinite colloid retention in the sand column. This is probably due to a decrease in repulsive forces between the kaolinite colloids and sand resulting from diffused double layer compression by divalent ions (Akbour et al. 2002). Similar results were previously reported in the co-transport study of nHAP and Cu^{2+} in the saturated sand column (Wang et al. 2011), where the amount of nHAP retained in the sand column increased with increasing NaCl concentrations and, especially, $CaCl_2$. In addition, hyper-saline solutions (Dead Sea water) of increasing ionic strength decreased the transport of fluorescent carboxylate-modified latex microspheres (1 μm diameter) in porous media (Magal et al. 2011).

Breakthrough curves for kaolinite-facilitated Cd in the different ionic strength solutions are shown in Fig. 4. Althougth the concentration of kaolinite-facilitated Cd in the effluent fluctuated for each ionic strength, an increase in the solution ionic strength clearly decreased the concentration of kaolinite-facilitated Cd^{2+} in the effluent, by up to 2.93-fold at 0.9 mM as compared to that at 0.0 mM (Fig. 4 and Table 3). Similar results were previously reported by Walshe et al. (2010). As mentioned above, cations can compete with the metal ion for vacant sites on the kaolinite colloids and this leads to a decline in the concentration of kaolinite-facilitated Cd^{2+}. Therefore, the increased retention of Cd^{2+}-bearing kaolinite colloids and decreased amount of Cd^{2+} adsorbed onto kaolinite colloids accounted for the decreased kaolinite-facilitated Cd^{2+} mobilization for an increase in solution ionic strength. However, dissolved Cd^{2+} in the aqueous phase should not be ruled out because dissolved Cd^{2+} accounted for a higher percentage of the total Cd^{2+} in the solution than kaolinite-facilitated Cd^{2+}.

4. CONCLUSION

Sorption capacities of kaolinite clay decreased with increasing solution ionic strengths which are due to ion competition between metal ions and cations in the solution for surface sorption sites. The sorption of Cd^{2+} onto kaolinite clay was best described by the Freundlich isotherm (as compared to the Langmuir isotherm) when the ionic strength was equal or higher than 0.45 mM, but was best described by Langmuir isotherm at low ionic strength (~0.0 mM). The solution ionic strength had a strong effect on the kaolinite-facilitated transport of Cd^{2+} in quartz sand saturated with water. The kaolinite-facilitated Cd^{2+} concentration declined and kaolinite colloids retention increased with increasing solution ionic strength, leading to a decreased in Cd^{2+} sorption capacity of the kaolinite colloids for an increase in solution ionic strength. Cd^{2+} transport behavior was altered due to the differences in the Cd^{2+}sorption capacity of the kaolinite colloids for different ionic strengths and the retention of

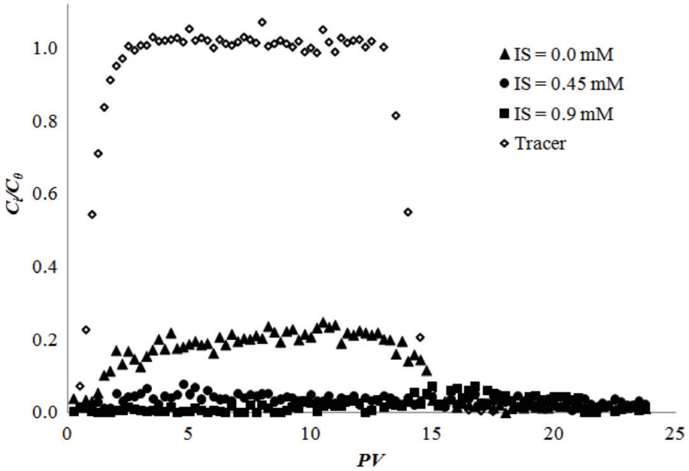

Fig. 3. Measured breakthrough curves for tracer and Cd^{2+}-bearing kaolinite colloids under the ionic strengths of 0.0, 0.45, and 0.9 mM.

Table 3. Properties of Cd^{2+}-bearing kaolinite suspension.

Ionic strength (mM)	pH	Zeta-potential* (mV)	Size* (μm)	Total Cd^{2+} (mg L^{-1})	Dissolved Cd^{2+} (mg L^{-1})	Kaolinite-facilitated-Cd (mg L^{-1})
0.00	5.59 ± 0.02	-26.3 ± 2.0	0.923 ± 0.024	9.290	8.295	0.995
0.45	5.60 ± 0.01	-22.4 ± 1.1	2.494 ± 0.032	9.290	8.540	0.75
0.9	5.61 ± 0.02	-20.7 ± 0.7	2.691 ± 0.084	9.000	8.660	0.34

*Note: *Size and surface charge of Cd bearing kaolinite colloid were measured before performing the transport test (Wikiniyadhanee 2012).*

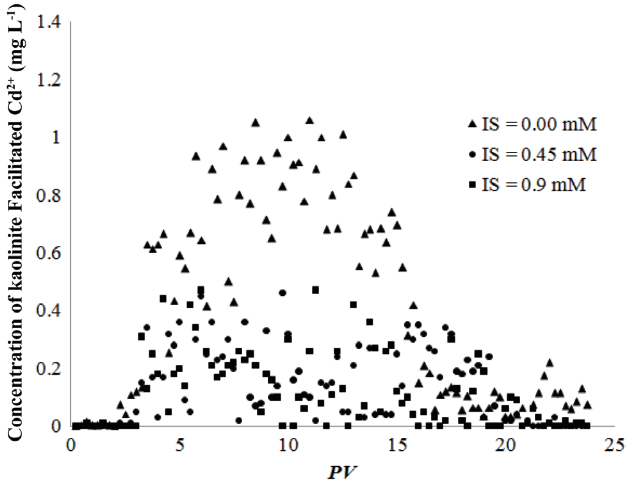

Fig. 4. Measured breakthrough curves for kaolinite-facilitated Cd under the IS of 0.0, 0.45, and 0.9 mM.

Cd^{2+}-bearing kaolinite colloids in the sand media.

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