INVESTIGATION ON SOME FACTORS CONTROLLING THE SORPTION OF HEAVY METALS ON ARIAKE CLAY

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ABSTRACT: Sorption of contaminant is one of the key mechanisms in controlling transport of contaminant in clay barrier. In this study, the potential sorption of heavy metals on Ariake clay, which is a typical Japanese marine clay, is examined. Two types of heavy metals, Cd^{2+} and Pb^{2+}, were selected as key contaminants and series of batch tests were performed for investigating factors controlling the sorption of Cd^{2+} and Pb^{2+} on Ariake clay. The batch test results show that the sorption of Cd^{2+} and Pb^{2+} on the Ariake clay arrived at an equilibrium condition less than the contact time prescribed by the ASTM and US EPA standards. It is found that the sorption was significantly controlled by the solid: solution ratio. With the increase in the solid: solution ratio, the sorption of Cd^{2+} decreased. The batch test results show that the sorption of Cd^{2+} increased with the increase in the pH of solution. The influence of pH on the sorption was found to be solid: solution-dependent. At relatively lower solid: solution ratio, the sorption of Cd^{2+} increased with increasing pH, whereas it marginally increased at relatively higher solid: solution ratio. It is concluded that the solid: solution ratio could be the crucial factor for controlling the sorption of heavy metals on the Ariake clay.

Key words: Ariake clay, barrier, landfill, heavy metal, pH, sorption

INTRODUCTION

Heavy metals such as Cd^{2+} and Pb^{2+} are commonly found in leachate from landfills. Due to their toxicity and potential migration, heavy metals may impact the aquifer underlying the landfill and thereby may pose potential risk to human health. An effective way for reducing this contamination risk is to use soil barriers (e.g., natural clayey soils) to highly adsorb and control the migration of heavy metals. To evaluate the application of clays for barrier materials, some parameters, such as sorption of heavy metals, need to be evaluated. A simple and easy way to investigate sorption behaviors of clays is to use batch test (ASTM 1993; US EPA 1987; Shackelford and Daniel 1991). Factors controlling sorption of heavy metals on clays, such as soil-solution contact time, solution chemistry and solid: solution ratio should be investigated (Manassero et al. 1998, 2000; Chang and Wang 2002; Lecite et al. 2003).

In Japan, there is a tendency to build waste sites in costal areas (Kamon and Katsumi 2001). Therefore, regional marine clays may provide potential source of soil barrier materials. Du et al. (2000) and Du and Hayashi (2004) showed that the Ariake clay, which is a typical regional marine clay located in Kyushu of Japan, seemed to be a good barrier material because of its high sorption capacity. However, rather relatively little information has been published with regard to the sorption of heavy metals on the Ariake clay. This aspect results in considerable uncertainty in using Ariake clay as a barrier in landfill, especially landfill for industrial waste, where heavy metals are commonly encountered.

The main objective of this study is to investigate the sorption of heavy metals on the Ariake clay. For this purpose, a series of batch test results has been presented, and the effects of soil-solution contact time, soil: solution ratio and pH of solution on the sorption of two heavy metals Cd^{2+} and Pb^{2+} are discussed, respectively.

BACKGROUND

Sorption

In this study, the term sorption refers to the process by which chemicals are partitioned between the liquid phase and the soil particle interface (Yong et al. 1992). It includes electrostatic and specific sorption and precipitation. The determination of the distribution of electrostatically sorbed ions can be obtained by the Double Diffuse Layer (DDL). The specific sorption involves inner-sphere surface complextion, which refers to the situation where ions are sorbed by forces other than the electric potential. The precipitation is the converse of dissolution, which can be a major factor in the retention of heavy metals in soils. All of

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these processes are directly or indirectly associated with the surface charge of soils.

SOIL DESCRIPTION

Ariake clay is a typical marine sedimentation product accumulated in the Ariake Bay over the past 10 000 years (Ariake Bay Research Group 1965). The details of its geological history and depositional geochemistry are provided by Ariake Bay Research Group (1965) and Ohtsubo et al. (1995), respectively. In this study, Ariake clay was sampled at 3 m depth from the ground surface at the Kawasoe Machi, Saga Prefecture, Kyushu, Japan. Some geotechnical and chemical properties are shown in Table 1.

The pH was measured in a 1:3 (by weight) mixture of soil and distilled water. The cation exchange capacity (CEC) and exchangeable cation concentrations were measured using the Schollenberger method (Schollengerger and Simon 1945).

To determine the clay mineral composition, moist soil samples were treated with 7% hydrogen peroxide (H_2O_2) to remove organic matters, followed by deflocculation by adjusting pH to 10 with 1 M sodium hydroxide (NaOH) after sonification. The <2μm clay fractions were collected by repeated sonification-sedimentation-siphoning.

For X-ray diffraction (XRD), duplicate clay suspensions containing 50 mg clay were prepared. One was washed with 0.5 M magnesium chloride ($MgCl_2$), the other was washed with 1.0 M potassium chloride (KCl), followed by washing with distilled water to remove excess salt. 1 cm^3 of water was added and an aliquot of the suspension containing 30 mg of clay was dropped onto a glass slide, air-dried and X-rayed. The potassium-saturated specimen was heated at 550°C, and the magnesium-saturated specimen was solvated with glycerol, followed by X-raying, respectively. Filtered CoK_α radiation from a Rigaku diffractometer was used for the XRD. In this study, the monochromatic radiations, K_α and K_β with different wave length, were produced by the X-ray tube. To identify the

Table 1 Some geotechnical and chemical properties of Ariake clay

Specific gravity, G_s	2.68
Natural water content, w_n (%)	173
Liquid limit, w_L (%)	115
pH	7.87
Grain size distribution	
Clay particle fraction (<2μm) (%)	46
Silt particle fraction (%)	49
Sand particle fraction (%)	5
Clay mineral compositions in clay size (%)	
Smectite	84
Illite	9
Kaolinite	7
Pore water concentrations (mg/L)	
Na^+	32
K^+	20
Ca^{2+}	17
Mg^{2+}	38
Cd^{2+}, Pb^{2+}	ND
CEC (meq/100 g)	36
Exchangeable cation concentrations (meq/100 g)	
Na^+	14
K^-	4
Ca^{2+}	7
Mg^{2+}	12
Cd^{2+}, Pb^{2+}	ND
Specific surface area (m^2/g)	69
Ignition loss (%)	10.2

Note: ND: not detected

Table 2 Properties of the soils and solutions used for the batch test for investigating the effect of contact time

Solid: solution (g/mL)	Soil mass (g)	Contact time (hour)	C_0 (mg/L)		pH		ORP (mV)	
			Cd^{2+}	Pb^{2+}	(a)*	(b)**	(a)*	(b)**
1: 20	5	1, 2, 4, 6, 9, 24 and 48	58.1	30.2	6.01	5.82~6.19	-14	-35~ -57

Note: *initial ** at equilibrium

Table 3 Properties of the soils and solutions used for the batch test for investigating the effect of solid: solution

Solid: solution (g/mL)	Soil mass (g)	Contact time (hour)	C_0 (mg/L)		C_e (mg/L)		pH		ORP (mV)	
			Cd^{2+}	Pb^{2+}	Cd^{2+}	Pb^{2+}	(a)*	(b)**	(a)*	(b)**
1: 1.3	100	24	10.7, 25.1, 40.1, 69.6	1.4, 16.2 19.7, 27.1	1.6~ 17.7	0	5.64~ 6.08	4.90~ 5.09	-11~ -52	-31~ -45
1: 4	50	24	10.7, 25.1, 40.1, 69.6	1.4, 16.2 19.7, 27.1	2.3~ 30.1	0	5.64~ 6.08	4.66~ 5.24	-11~ -52	-31~ -52
1: 10	20	24	10.7, 25.1, 40.1, 69.6	1.4, 16.2 19.7, 27.1	2.5~ 43.3	0	5.64~ 6.08	4.75~ 5.34	-11~ -52	-35~ -54
1: 20	10	24	10.7, 25.1, 40.1, 69.6	1.4, 16.2 19.7, 27.1	3.2~ 51.1	0	5.64~ 6.08	5.11~ 5.47	-11~ -52	-28~ -42

Note: *initial ** at equilibrium

clay minerals, K_β was filtered by using a metal, cobalt (Co) and only K_α ray was used. The percentage of the respective clay minerals in clay fractions were estimated based on the peak areas of the clay minerals in the X-ray diffraction patterns (Wada 1966). The results show that the Ariake clay is dominated by smectite.

TEST METHOD

In this study the batch-contact test was performed according to the ASTM (1993) standard. Generally, the sampled Ariake clay was air-dried, ground and passed through a 2-mm sieve. Sieved soils with size <2 mm were adopted for batch tests. The synthetic leachate, a solution containing $CdCl_2$, $PbCl_2$ and KCl, were prepared by dissolving the commercial $CdCl_2$, $PbCl_2$ and KCl solids in powder form in the distilled pure water. The batch tests consist of placing soil samples and solutions at certain solid (soil mass, g): solution (volume, mL) ratio (this ratio is termed *SD/SN* throughout the text) into a 2 L Erlenmeyer flask. The flask was top-sealed and placed in an end-over-end rotary mixer and shaken at 29 rpm for a given number of hours. At the end of the mixing period, the equilibrium pH and Oxidation-Reduction Potential (ORP) of soil-solution slurry were recorded. A sample of the soil-solution slurry was poured from the flask into a 50-mL centrifuge tube and centrifuged for 30 minutes at 3000 rpm. After that, the concentrations of Cd^{2+} and Pb^{2+} in the supernatant sampled from the tube were measured using a HITACHI Z-6000 atomic adsorption spectrophotometer, and termed equilibrium concentrations, C_e. To investigate the influence of soil-solution contact time, the soil: solution ratio was set

as 1: 20 and contact time was arranged as 1h, 2h, 4h, 6h, 9h, 24h and 48h. The initial concentrations of Cd^{2+} and Pb^{2+} were 58.1 mg/L and 30.2 mg/L respectively and the initial pH of solution was about 6 (Table 2). To investigate the influence of *SD/SN* on sorption, four cases were considered by setting the solid: solution ratio at 1: 20, 1: 10, 1: 4 and 1: 1.3 with contact time of 24 h. For each case, the initial concentrations of Cd^{2+} and Pb^{2+} were (10.7, 25.1, 40.1 and 69.6 mg/L) and (1.4, 16.2, 19.7 and 27.1 mg/L) respectively, and the initial pH of solution varied from 5.64 to 6.08 (Table 3). All of the tests noted above were performed at a laboratory temperature of 22°C.

The chemical analysis of the batch tests was plotted in the form of the adsorption isotherm or equilibrium concentration, C_e (mg/L), versus sorbed concentration, q (μg/g). The sorbed concentration, q, was determined according to the following equation

$$q = \frac{(C_0 - C_e)V_{sol}}{M_s} \times 1000 \tag{1}$$

where C_0 = the initial concentration of Cd^{2+} or Pb^{2+} (mg/L), V_{sol} = the volume of the solution (L) and M_s = the soil mass (oven-dried basis).

The percent of sorption for a unit mass of soil, *PS* (%/g), is defined as

$$PS = \frac{(1 - C_e/C_0) \times 100}{M_s} \tag{2}$$

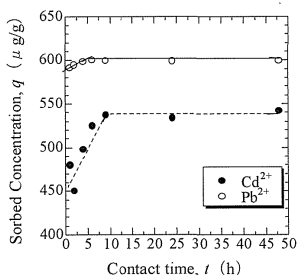in which all of the symbols are defined in Eq.(1).

Fig. 1 Change of sorbed concentrations of Cd^{2+} and Pb^{2+} with contact time

Fig. 2 Adsorption isotherms of Cd^{2+} at four solid: solution ratios

To predict the relationship between the sorbed and equilibrium concentrations obtained from batch tests, the Freundlich isotherm equation is adopted as expressed by

$$q = K_f C_e^{N} \tag{3}$$

in which K_f and N are the Freundlich sorption parameters adjusted to fit Eq. (3) to the experimental data.

RESULTS AND DISCUSSIONS

Effect of Contact Time

Figure 1 shows the sorbed concentrations versus contact time for Cd^{2+} and Pb^{2+}. It can be seen that it took about 9h and 6h for Cd^{2+} and Pb^{2+} to reach the equilibrium conditions respectively. The 24 h of contact time, which is prescribed in the ASTM (1993) standard test, is enough for Ariake clay to reach a satisfactory equilibrium. Due to this observed fact, for the following batch tests which focused on investigating the influence of solid: solution ratio, the contact time was set as 24 h.

The observed time scale required to reach the equilibrium condition indicates that: (1) sorption of heavy metals on Ariake clay was an instantaneous process. The hypothesis of sorption being instantaneous, which is usually assumed for modeling transport of contaminant in soil, is acceptable. When the sorption kinetics is concerned for modeling the transport of Cd^{2+} and Pb^{2+} in Ariake clay barrier, 24 h would be sufficient to reach an equilibrium condition; (2) using adsorption isotherm, which is an equilibrium model, to model the sorption of heavy metals onto Ariake clay would be acceptable. With this verification, the Freundlich isotherm model was used to model the sorption of Cd^{2+} and Pb^{2+} on Ariake clay as presented later in this paper.
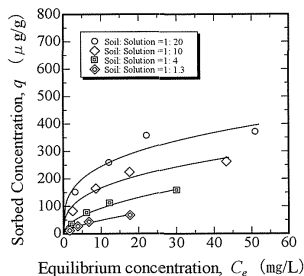
From Fig. 1, it is also clear that Ariake clay sorbed larger amount of Pb^{2+} than Cd^{2+} indicating that the selectivity of Pb^{2+} was higher than that of Cd^{2+}. This result is consistent with previous finding (Elliot et al. 1986; Roehl and Czurda 1998).

Effect of Solid: Solution Ratio

The adsorption isotherms for Cd^{2+} at four different *SD/SN* values are depicted in Fig. 2. All of the isotherms appear non-linear, which is consistent with the literature study (Shackelford and Daniel 1991; Lecite et al. 2003). From Fig. 2, it is also clear that sorption of Cd^{2+} largely depended on the value of *SD/SN*. With the increase in *SD/SN*, the amount of sorbed Cd^{2+} decreased. The largest amount of sorbed Cd^{2+} was observed at *SD/SN* of 1: 20 whereas the lowest was at a ratio of *SD/SN* of 1: 1.3. The Ariake clay sorbed Cd^{2+} about 4 times more in 1: 20 compared to 1: 1.3. The change in pH at equilibrium for different *SD/SN* values are shown in Fig. 3. It is clear that pH drop increased with increasing *SD/SN*. When *SD/SN* was relatively low, <1: 4, the pH dropped sharply, whereas pH dropped slightly when *SD/SN* was relatively high (e.g., >1: 4). For Cd^{2+}, the change of *PS* with *SD/SN* are depicted in Fig. 4. The shapes of the curves are well consistent with that of pH drop at equilibrium, indicating that *SD/SN* had played an important role in controlling the sorption of Cd^{2+} on Ariake clay.

The Freundlich sorption parameters (K_f and N) obtained from this study for Cd^{2+} are listed in Table 4. The tendency of the changing of the two parameters with *SD/SN* (Fig. 5) is found to be well consistent with those of the pH drop at equilibrium and *PS* (see Figs 3 & 4, respectively). At low value of *SD/SN*, <1: 4, K_f and N changed significantly, whereas at high value, >1: 4, K_f and N changed slightly. The relationship between the Freundlich sorption parameters and *SD/SN* presented in this study can be expressed as

Table 4 Freundlich sorption parameters at different solid: solution ratios for Cd2+

Parameters	Solid: Solution (g/mL)			
	1: 20	1: 10	1: 4	1: 1.3
K_f	123.97	75.06	28.70	12.23
n	0.295	0.346	0.508	0.602
R^2	0.883	0.937	0.978	0.972

$$K_f = 10.272(SD/SN)^{0.836} \quad (R^2{=}0.994) \qquad (4)$$

$$N = 0.662(SD/SN)^{0.258} \qquad (R^2{=}0.952) \qquad (5)$$

The dependence of sorption behavior on *SD/SN* noted above indicates that with respect to the determination of the sorption parameters (K_f and *N*) from the batch tests to be used for modeling transport of heavy metals in the Ariake clay barrier, the effect of *SD/SN* must be considered.

The reason that the Ariake clay displayed solid: solution-dependent sorption behavior can be attributed to the findings made by Voice et al. (1983) and Gschwend and Wu (1985). They indicated that the behavior was due to microparticulate molecular material being washed off from the soil particles during the course of the batch test, and not being removed from the liquid phase during the separation procedure. These "non-settling microparticles" were found to be able to accommodate solutes and tend to increase the capacity of the liquid phase to accommodate solutes. Therefore, the apparent equilibrium concentration (C_e) of a specific solute measured in the batch test increased, and the sorbed concentration (q) as well as the percent sorbed solute per unit soil mass (*PS*) decreased based on Eq. (1) and (2), respectively. The amount of the non-settling microparticles increases with the soil phase in the system (i.e., the solid: solution ratio), and thereby the values of q and *PS* for Cd^{2+} increased with the increase in the solid: solution ratio, as shown in Fig. 2 and 4, respectively.

Generally it is thought that sorption of metal ions to soils involves concurrent release of protons (H$^+$) as a reaction in the surface function of the soil particles, which may decrease pH of the solution (Sposito 1989). For a given volume solution phase, with the increase in the solid: solution ratio, the solid concentration will increase and there will be more sites available on the soil particles for releasing H$^+$. Therefore the decrease in pH of solution will be more significant as that observed in this study (Fig. 3). However, under the condition of relatively high solid: solution ratio, the buffer potential of soil tends to play significant role in resisting the change in pH when more H$^+$ are potentially being released. As a result, the change in pH of the solution tends to become slight even though the solid: solution ratio increases. This may explain the observation presented in this study that pH drop at equilibrium became stable at relatively high solid: solution ratio, > 1: 4 (Fig. 3).
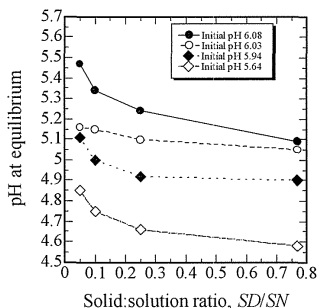

Fig. 3 Change of pH at equilibrium with solid: solution

For Pb^{2+}, it was observed that the equilibrium concentrations in the batch tests at different soil solution ratios were almost zero (Table 3). Therefore it was not able to construct the adsorption isotherms. As a result, the Freundlich sorption parameters for Pb^{2+} were not determined. The reason that the equilibrium concentrations of Pb^{2+} were almost zero may be due to that Pb^{2+} were highly sorbed on the Ariake clay during the batch tests. Since the lower the equilibrium concentration, the higher the sorbed concentration will be, based on Eq. (1), this observation indirectly indicates that the Ariake clay has higher retention ability for Pb^{2+} than for Cd^{2+}.

Effect of pH

From Fig. 4, it seems that at the same *SD/SN*, the sorption of Cd^{2+} increased with increasing pH. To better understand the influence of pH, the relationship between the values of *PS* and initial pH, is depicted in Fig. 6. Generally, it can be seen that with the increase in pH, the value of *PS* increased. For the case of low *SD/SN* (i.e., 1: 20 and 1: 10), *PS* increased significantly with increasing pH whereas for the case of high *SD/SN* (i.e., 1: 4 and 1: 1.3), *PS* increased only marginally with increasing pH. This result indicates that the effect of pH on sorption may largely depend on the solid: solution ratio. A report of the influence of pH on sorption should be along with the presence of value of *SD/SN*.

The reason of the aforementioned phenomenon may be explained by the buffer potential of the Ariake clay to resist the change in pH. As discussed in the earlier part of this paper, at relatively high solid: solution ratio, > 1: 4, when more H$^+$ are being potentially released, the buffer potential of the Ariake clay may begin to play considerable role in resisting the change in pH. Therefore, the change in pH will not be considerable at relatively high solid: solution ratio, as shown in Fig. 3. As a result, at relatively high solid: solution ratio, *PS* changed slightly with the increase of initial pH of solution since the retention of Cd^{2+} on soils is generally significantly affected by the change in pH of solutions (Yong et al. 1992).
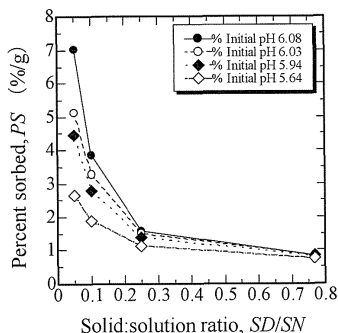
Fig. 4   Change of percent sorbed Cd^{2+} with solid:
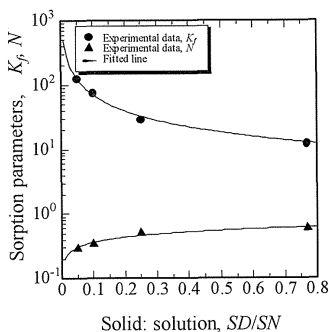solution ratio

Fig. 5   Change of sorption parameters with solid:
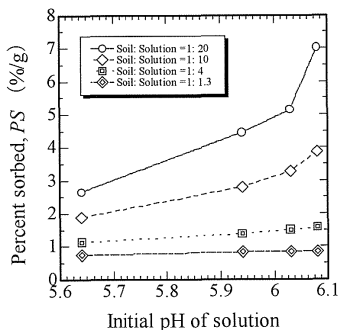solution ratio for Cd^{2+}

Fig. 6   Change of percent sorbed Cd^{2+} with pH of
solution

CONCLUSIONS

To assess the potential use of Ariake clay as a soil barrier material, the sorption performance of Cd^{2+} and Pb^{2+} on the Ariake clay has been investigated via the batch test. The batch test results are presented and the effects of contact time, solid: solution ratio and pH of solution on sorption have been discussed. Following conclusions can be drawn from this study:

1)   The time for the sorption of Cd^{2+} and Pb^{2+} on Ariake clay to reach an equilibrium condition is rather short. In terms of practice, when sorption kinetics is needed in modeling transport of Cd^{2+} and Pb^{2+} in the Ariake clay, the standard contact time is enough to reach a satisfied equilibrium condition.

2)   The equilibrium concentrations of Pb^{2+} in the batch tests were found almost zero, indirectly indicating that the Ariake clay has higher retention ability on Pb^{2+} than Cd^{2+}.

3)   With the increase of the solid: solution ratio, the amount of sorbed Cd^{2+} decreased. The Freundlich sorption parameters presented in this study are found to have good relationship with solid: solution ratio. These results indicate that when performing batch test to derive sorption parameters, the influence of solid: solution ratio must be considered.

4)   The sorption of Cd^{2+} increased with increasing pH. The effect of pH was found to be solid: solution ratio-dependent. At relatively higher solid: solution ratio, the sorption only increased marginally with increasing pH.

5)   The batch test results reveal that among the three targeted factors, the solid: solution ratio seems to be the crucial one controlling the sorption of Cd^{2+} on the Ariake clay.

ACKOWLEDGEMENT

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