

Sorption of cesium and iodide ions onto KENTEX-bentonite

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Abstract The sorption of cesium and iodide ions onto KENTEX-bentonite was investigated using batch test and in-diffusion test methods. The cesium ions were highly sorbed on the bentonite, and the experimental data fit the Freundlich isotherm well. The distribution coefficient, K_d , of the cesium ions was variably affected by the chemical conditions of the solution (initial ion concentration, pH, salinity) and temperature. An increasing pH of solution increased the K_d . However, there were different K_d values that decrease with an increase in the initial ion concentration, salinity, and temperature. The iodide ions, on the contrary, were negligibly sorptive. The K_d values obtained from the in-diffusion tests were quite lower than those from the batch tests, which could be explained by changes in the pore water chemistry and surface area available for sorption.

Keywords Sorption · Cesium and iodide · Distribution coefficient (K_d) · Sorption-affecting factors · Bentonite buffer · Radioactive waste repository

Introduction

A reference disposal system (KRS) for high-level waste (HLW) management in Korea will be constructed in bedrock several hundred meters below the ground surface and its engineered barrier system (EBS) will consist of a HLW-encapsulating container, a bentonite buffer between the container and the wall of a borehole, and a backfill in the

space of an emplacement room to isolate the HLW from the surrounding rock masses (Kang et al. 2002). The Korea Atomic Energy Research Institute (KAERI) carried out a large-scale test to investigate the thermal-hydro-mechanical (THM) behavior in the bentonite buffer using an experimental apparatus (KENTEX), which was designed to be at one-third scale of the KRS (Lee et al. 2008), and currently, has been performing a subsequent test with the same experimental apparatus to investigate the transport of cesium and iodide ions through the bentonite buffer under a steady-state THM condition (Cho et al. 2012). It is essential, in this connection, to understand the sorption of cesium and iodide ions onto the bentonite because the sorption dominates the transport of both ions through the bentonite buffer.

Sorption includes sorptive and non-sorptive processes that retard the migration of aqueous species through physical adsorption, electrostatic adsorption, chemisorptions, and other mechanisms such as precipitation, co-precipitation, and substitution. The mechanism of sorption depends on the chemical properties of the ions in solution, i.e., ion speciation, and both the chemical and physical properties of the sorbent (bentonite) (Allard et al. 1983). Sorption studies have been performed by measuring the distribution coefficient (K_d) that describes the interaction of bentonite–solution in the sorption. The distribution coefficients of ions for the bentonites were measured through many investigations. Most of their measurements (Galambos et al. 2009a, b, 2010, 2011, 2012; Chen et al. 2011a, b; Hamidpour et al. 2011; Kufcaková et al. 2005; Bradbury and Baeyens 2003; Hurel et al. 2003; Bereket et al. 1997; Auboiroux et al. 1996; Zhang and Sparks 1996; Jeong et al. 1995; Khan et al. 1994, 1995; Puls 1988; Inskip and Baham 1983; Garcia-Miragaya and Page 1976), which were done by means of a batch test method, were focused on investigating the effects of experimental variables (initial ion concentration, pH, salinity, temperature,

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etc.) upon the distribution coefficient as well as the detailed mechanisms leading to those effects. These investigations showed that the distribution coefficients of ions had a wide range of values and different trends depending on the experimental variables. On the other hand, some measurements (Oscarson et al. 1994; Sato et al. 1992; Miyahara et al. 1991; Muurinen et al. 1987) of the distribution coefficients of ions for bentonites have been done by means of a diffusion test method which, even if the test procedure is more complicated than the batch test method, has an advantage in that the distribution coefficient can be directly measured under realistic conditions as in the compacted bentonite buffer of a repository. Oscarson et al. (1994), Sato et al. (1992), and Miyahara et al. (1991) reported that the distribution coefficients obtained from the diffusion test method were lower than those from the batch test method. This difference requires more care to use the distribution coefficient as an input data for transport models, as the distribution coefficient obtained from the batch test method may underestimate the transport of ions through the bentonite buffer.

The present study carries out the batch test with KENTEX-bentonite to investigate the effect of initial ion concentration, pH, salinity, and temperature on the distribution coefficient of cesium and iodide ions. Also a comparison is made between the distribution coefficients obtained from the batch test and in-diffusion test methods to assess the limitations on their application to the transport of cesium and iodide ions through the bentonite buffer.

Materials and methods

Bentonite

The bentonite used for tests was taken from Kyeongju, Korea, which is the same as that in the KENTEX THM test (hereafter, referred to as KENTEX-bentonite) (Lee et al. 2008). It was dried below 110 °C, pulverized, and then passed through No. 200 of ASTM (American Society for Testing and Materials) standard sieves. The mineralogical composition of the KENTEX-bentonite was determined through X-ray diffraction (XRD) using monochromator-filtered, Cu-K α radiation generated at 40 kV and 20 mA. As shown in Fig. 1, it contains montmorillonite (70 %), feldspar (29 %), and small amounts of quartz (~1 %), and it was found to be of Ca-type by Chun et al. (1998). The chemical composition is listed in Table 1, and the cation exchange capacity (CEC) is 57.6 meq/100 g.

Solutions

Experimental solutions were prepared by dissolving a CsI reagent in synthetic groundwater (SGW) with the chemical composition shown in Table 2. The initial concentrations of the experimental solutions were set to 10^{-5} – 10^{-1} M for the batch tests and 10^{-2} M for the in-diffusion tests to measure the distribution coefficients for the sorption of cesium and iodide ions onto the KENTEX-bentonite.

Fig. 1 X-ray pattern of KENTEX-bentonite

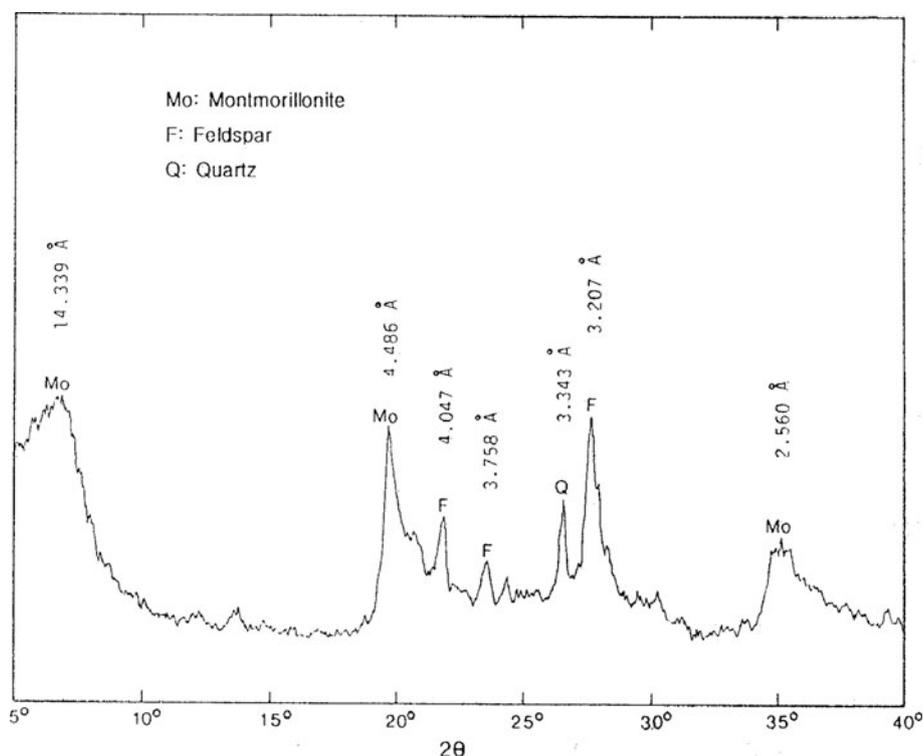


Table 1 Chemical composition of KENTEX-bentonite

Chemical constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	FeO	SO ₃	TiO ₂
Percentage	56.8	20.0	6.0	2.6	0.8	0.9	1.3	0.2	1.3	0.8

Table 2 Chemical composition of synthetic groundwater (SGW) used in batch and in-diffusion tests

Component	Concentration (mg/L)
Na	57.0
Ca	7.4
Mg	0.7
K	0.7
Si	7.2
Al	0.1
Fe	0.01
Mn	0.01
Zn	0.01
F	7.65
Cl	16.8
SO ₄	25.3
NO ₃	0.15
PO ₄	0.50
CO ₃ + HCO ₃	15.2
pH (–)	8.06
Eh (mV)	405.9

Measurement techniques of distribution coefficient

The distribution coefficient is defined as the distribution ratio of solute ions between a solution and a solid (bentonite) phase. This study employed two kinds of techniques for the measurement of the distribution coefficient: batch test and in-diffusion test methods.

The batch test method was performed by shaking 1×10^{-3} kg of the bentonite powder with 20×10^{-6} m³ of CsI solution in a high-density polyethylene (HDPE) vial for 4 weeks at room temperature (25 °C). The shaking rate was 100 oscillations per minute. At the end of the shaking period, the aqueous phase was separated from the bentonite particles by centrifuging for 30 min at 12,000 rpm and was then filtered through a ϕ 25-mm, 0.45- μ m membrane filter. An aliquot of the filtrate was sampled and its concentration was analyzed using an Atomic Absorption Spectrophotometer (PERKIN ELMER #400) for cesium ions, and an Ion Selective Electrode Meter (ISTEK #735P) for iodide ions. The pH of the solutions was also measured before and after the batch tests. In the batch tests, the K_d of the cesium and iodide ions was determined from the following equation:

$$K_d = \frac{\text{Mass of an ion sorbed on unit mass of bentonite}}{\text{Mass of an ion in solution per unit volume of solution}} = \frac{(w_o - w_1)/w}{w_1/V} \text{ (m}^3\text{/kg)}, \tag{1}$$

where w_o is the mass of an ion in the initial solution, w_1 the mass of an ion in the solution after sorption equilibrium, w the mass of the solid, and V is the volume of the solution. The same batch test was also performed using blank vials that did not have bentonite powders to check if any of the cesium or iodide ions might be removed by the HDPE vials. The results of these blank tests showed that the cesium and iodide ions remained completely in solution without any removal by the HDPE vials.

The in-diffusion test method (Fig. 2, upper), which consists of two layers of compacted bentonite with each layer saturated with an ion-spiked solution and ion-free water, respectively, has been used to determine the diffusion coefficient of ions in compacted bentonite (Lever 1989). This test method allows the ions in the spiked compacted bentonite to diffuse across the ion-free compacted bentonite. Figure 2 (lower) shows an experimental apparatus of an in-diffusion test, which was conducted for the cesium ions in this study. The diffusion cells were made of two stainless steel cylinders with a 3×10^{-2} m inner diameter and 3×10^{-2} m height. The dry densities of the compacted bentonite plugs were set to 1,600 and 1,700 kg/m³. These tests were carried out according to the following experimental procedure: air-dried and weighed bentonite was mixed with a pre-determined amount of SGW or 10^{-2} M CsI solution required to saturate the compacted bentonite. The mixture was kneaded for a uniform distribution of the liquid phase in the bentonite and then compacted in each cylindrical cell to a target dry density using a hydraulic press. The compacted bentonite plugs were left in a zip-bag for about 4 weeks for complete saturation. When the two bentonite-compacted cylinders were prepared, they were then contacted and combined firmly together with restrain rams. The in-diffusion tests were conducted in a temperature-controlled water bath (25 °C). After a certain time period, the diffusion cells were disassembled and the bentonite plugs were extruded and sliced into a 2×10^{-3} m thickness using a thin blade. The bentonite slices were divided into two parts. One was used to determine the water content and the other used to analyze the concentration of cesium. The cesium ions were extracted from the sliced bentonites with 1 M HCl

solution, and their concentration was measured using the same analyzer, as in the above batch tests. The measured values of concentration (C) were regarded as average at the center of each slice. The apparent diffusion coefficient (D_a) of cesium ions was determined by curve-fitting the measured concentration with the calculated values of Eq. (2):

$$C/C_o = \frac{x_o}{L} + \frac{2}{\pi} \sum_{n=1}^{\infty} \frac{1}{n} \exp\left(-\frac{D_o n^2 \pi^2}{L^2} t\right) \cos\left(\frac{n\pi}{L} x\right) \quad (2)$$

where C_o is the initial ion concentration in the ion-spiked bentonite plug, x_o the location of the interface between the two bentonite plugs in the diffusion cell, and L is the overall length of the diffusion cell. Details of the in-diffusion test and the determination of the apparent diffusion coefficient have been described in Lee et al. (2002). Finally, the distribution coefficient, $K_{d,\text{in-diffusion}}$ for the in-diffusion test was calculated from the above-determined apparent diffusion coefficient using the following Eqs. (3) and (4) (Miyahara et al. 1991):

$$D_a = D_o f / \left(1 + \frac{\rho_d}{\varepsilon} K_d\right), \quad (3)$$

$$K_{d,\text{in-diffusion}} = \left(\frac{D_o f}{D_a} - 1\right) \frac{\varepsilon}{\rho_d} \quad (4)$$

where D_o is the diffusion coefficient of cesium ions in pure water, D_a the apparent diffusion coefficient of cesium ions in the compacted bentonite plug, and ε , f , ρ_d are the porosity, impedance factor, and dry density of the compacted bentonite, respectively.

Results and discussion

Chemical speciation

The speciation of cesium and iodine in the solutions was calculated for the range of pH 7–14 using a chemical equilibrium speciation program, PHREEQE (Parkhurst et al. 1993). The calculation results showed that for the cesium Cs^+ was a major species, and its portion was absolutely dominant in the whole pH range. For the iodine, on the other hand, I^- and IO_3^- were present, in which I^- was dominant at $\text{pH} < 11$, and beyond this pH, its portion was reduced with IO_3^- existing as a major species.

Sorption of cesium and iodide ions

The results of the equilibrium test, which depicts the concentration of ions in a solution as a function of time, are shown in Fig. 3 (upper). As shown in the figure, the sorption of cesium ions onto the bentonite almost reached equilibrium in a couple of days.

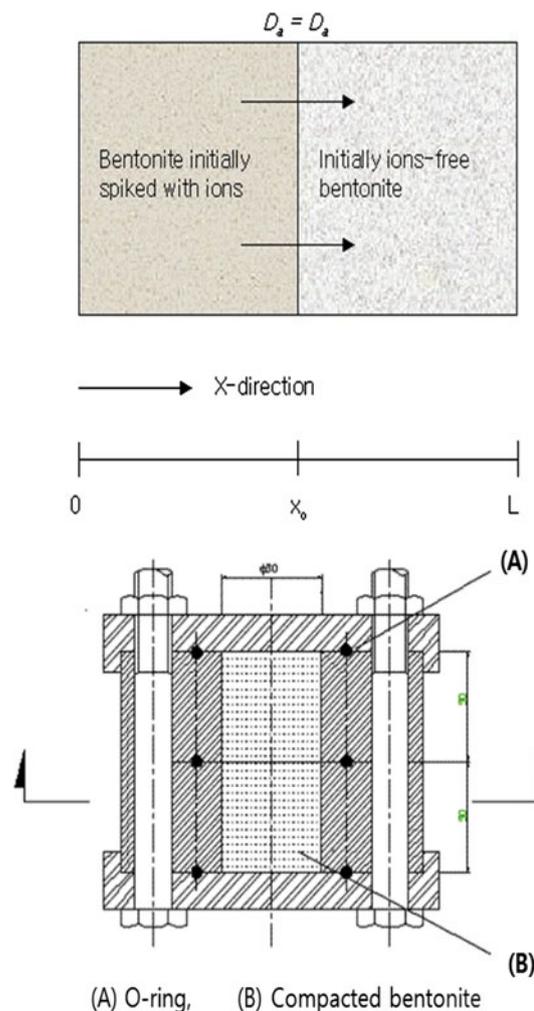


Fig. 2 Schematic diagram (upper) and experimental apparatus (lower) of in-diffusion test method

The experimental data were fitted to the Freundlich isotherm. This isotherm can be written as

$$q = K_F C^{1/n}, \quad (5)$$

where q is the amount of ions sorbed on the solid (bentonite) in mol/kg, C the amount of ions remaining in the solution at equilibrium in mol/m^3 , and K_F and $1/n$ = constants that provide rough estimates of the sorption capacity and the intensity of the sorption, respectively (Adamson 1976). As shown in Fig. 3 (lower), the test data fitted the Freundlich isotherm well. The fitting resulted in $K_F = 142.56$ and $n = 1.292$.

The distribution coefficient, K_d , for the cesium sorption showed high values under given experimental conditions. Figure 4 (upper) is a plot of the K_d as a function of the initial concentration in the range of 10^{-5} – 10^{-1} M. The K_d was highly dependent on the initial concentration beyond 10^{-4} M. It decreased with an increase in the initial concentration of cesium in the solution. This is probably

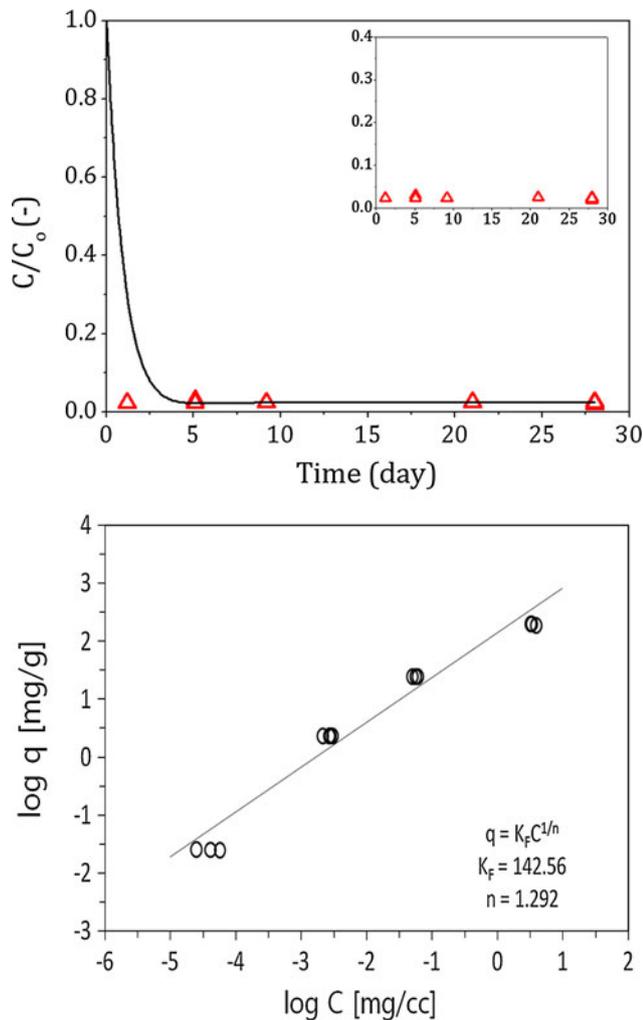


Fig. 3 Equilibrium test results as a function of time (*upper*) and Freundlich isotherm (*lower*) for cesium ion

because a significant ion competition, with an increasing initial concentration, occurs among the cesium ions and thus sorption sites available on the surface of bentonite particles are reduced. However, below the initial concentration of 10^{-5} M, the K_d value is expected to be nearly constant similar to experimental results of Salter et al. (1981), Ames and McGarrah (1980), and Lynch and Dosch (1980) in their sorption tests.

When an HLW repository is located in a coastal area, the salinity of porewater in the bentonite buffer increases owing to seawater intrusion. The effect of salinity on the distribution coefficient was examined using NaCl solution. The presence of sodium ions and their concentration had a large influence on the sorption of cesium onto the bentonite (Fig. 4, lower). The K_d decreased with an increase in the concentration of NaCl solutions ranging from 0.04 to 1 M. This can be explained by the following two processes: a change in the density of ions in diffusive double layers

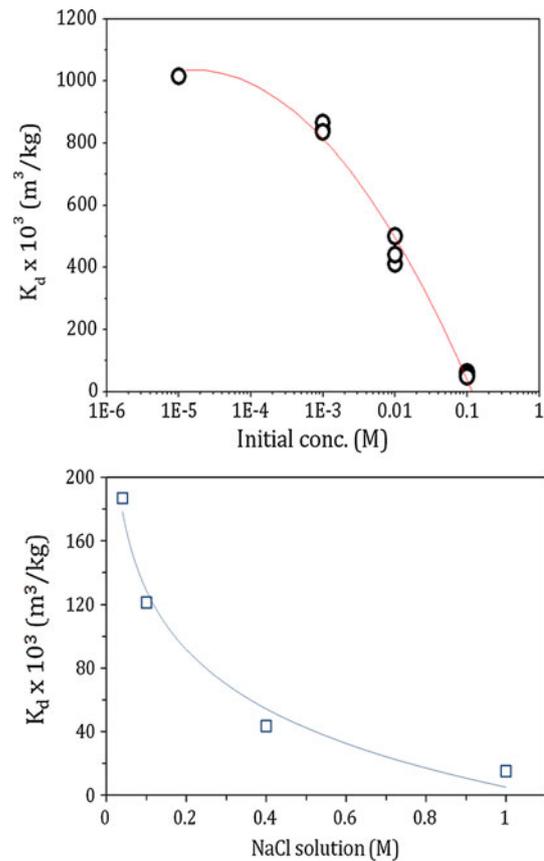


Fig. 4 Effect of the initial concentration of cesium ion (*upper*) and salinity (*lower*) on the K_d

(DDL) (Yong and Warkentin 1976) on the surfaces of bentonite particles and the ion competition effect on the sorption sites. An increasing concentration of NaCl solution increases the density of ions in the DDL, and thereby the sorption of cesium ions onto the bentonite is hindered. Also, the increasing concentration of NaCl solution leads to a large ion competition. That is, the increasing presence of sodium ions in the solution prevents cesium ions from filling sorption sites by an ion exchange because those ions may take up the sorption sites.

The effect of the pH of the solution on K_d is shown in Fig. 5 (*upper*). The solution pH was adjusted by adding NaOH solution. The K_d for the cesium ions increased with an increase in the pH in the range of 9.5 and 14. This is attributed to a change in the surface charge of bentonite particles. As shown in Fig. 5 (*lower*), the surface charge increases negatively with an increasing solution pH, which results in an increase of K_d by the electrostatic adsorption such as an ion exchange reaction (Hayes and Leckie 1987; Jeong et al. 1995). However, the present study did not show a sharp increase in the alkaline range of pH owing to precipitation of hydroxide compounds as reported for alkaline earth metal or transition metal ions (Goh et al.

2000, 2001). The results of the speciation calculation for the cesium support this. As mentioned above, the cesium exists almost in Cs^+ in the whole range of pH, and thus there are no hydroxide compounds for the sorption of cesium ion onto the bentonite particles.

Figure 6 shows the effect of temperature on the K_d . For the cesium, the K_d decreased with an increasing temperature of up to 60 °C and then remained almost constant. Khan et al. (1994) and Kenna (1980) also reported results similar to ours. Generally a temperature increase, for an endothermic reaction, causes the reaction rate to increase. However, this study indicates that the sorption of cesium ions onto bentonite is exothermic [its enthalpy (ΔH°) < 0 (Shahwan and Erten 2002)]. It is supposed that the exothermic reaction resulted in the process of desorption favored at higher temperatures and thus a decrease in the K_d of the cesium ions.

Figure 7 (upper) shows the results of iodide equilibrium test for the initial concentration of 10^{-3} M. The concentration of iodide ions in the solution was reduced quite a

little over time and then reached equilibrium in a couple of days. Figure 7 (lower) shows a plot of K_d as a function of the initial concentrations. This also shows that the sorption of iodide ions onto the bentonite was quite small. The K_d values were below about 5×10^{-3} m³/kg. Muurinen (1989) also reported similar results in his sorption tests with a sodium bentonite–chloride system. Negligible or little sorption of iodide ions (I^-) onto the bentonite is possibly attributed to anion exclusion from the negative surface charge of bentonite particles. Also, the slight dependency of K_d on the initial concentration, in this figure, is supposed to be within the experimental error range.

Distribution coefficients ($K_{d,\text{in-diffusion}}$) determined using an in-diffusion test method

Investigators (Meier et al. 1987; Alberts et al. 1986; O'Connor and Connolly 1980) have reported that the solution-to-solid (bentonite) ratio affects the sorption of ions onto the bentonite. For a repository, the solution-to-bentonite ratio would be much lower because the groundwater is limited to narrow pores within the compacted bentonite buffer. The in-diffusion tests in this study were carried out to determine the distribution coefficient under realistic solution-to-bentonite ratios as in the compacted bentonite buffer.

The in-diffusion test method premises complete saturation in the bentonite plugs. Figure 8 (upper), which is an example of the distributions of water content (ω/ω_0) in the bentonite plugs, shows that the water content was more than 98 % of the moisture content at a saturation state. This indicates that the bentonite plugs were in nearly full saturation during the in-diffusion test.

Figure 8 (lower) displays the concentration profiles of cesium ions for the dry densities of 1,600 and 1,700 kg/m³ obtained in the in-diffusion tests. The apparent diffusion

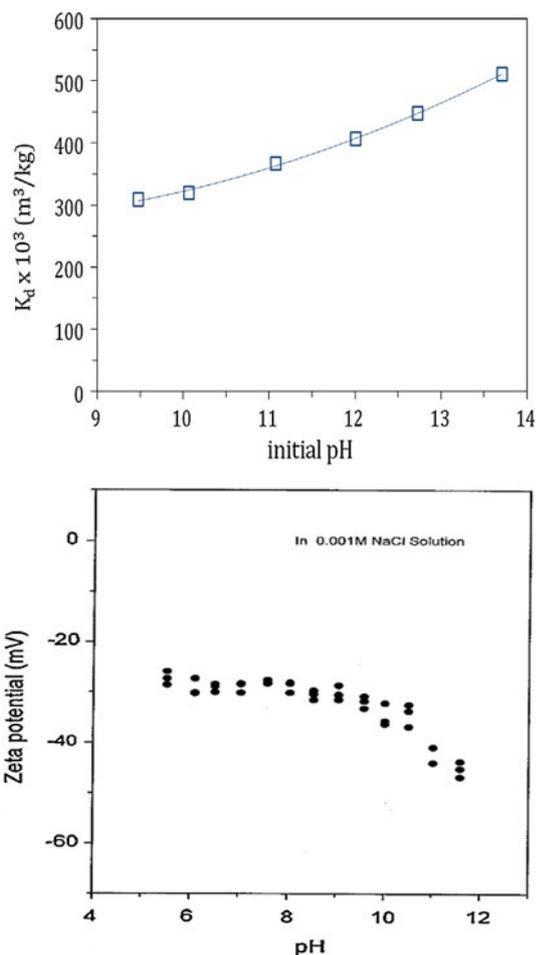


Fig. 5 Effect of the initial pH of solution on the K_d (upper) and zeta potential of the bentonite as a function of pH under 0.001 M NaCl solution (lower)

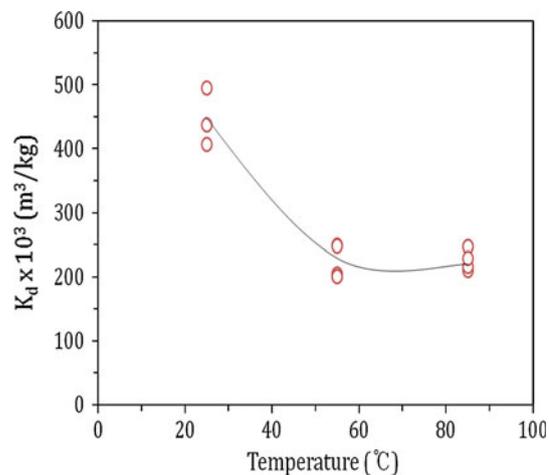


Fig. 6 Effect of temperature on the K_d

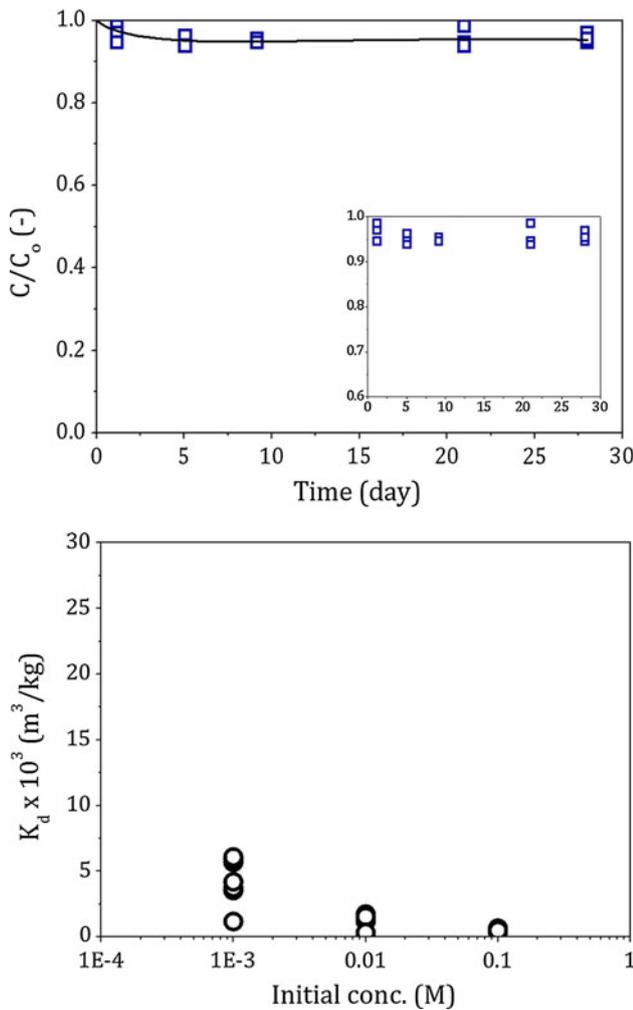


Fig. 7 Iodide equilibrium test results (*upper*) and the effect of the initial concentration of iodide ion on the K_d

coefficients (D_a) were determined by curve-fitting the concentration profiles in this figure with the calculated values of Eq. (2), and the distribution coefficient for the in-diffusion test method, $K_{d,in-diffusion}$, was then calculated from the determined apparent diffusion coefficient using Eq. (4). The calculated $K_{d,in-diffusion}$ values are listed together with the other parameter values of Eq. (4) in Table 3. When the dry densities of compacted bentonite are 1,600 and 1,700 kg/m³, the $K_{d,in-diffusions}$ are 4.5×10^{-3} and 4.3×10^{-3} m³/kg, respectively. They both are expected to be negligibly different. However, they are much lower than the K_d value obtained from the batch test, which was conducted at the solution-to-bentonite ratio of 20×10^{-3} m³/kg. The batch K_d value was 446×10^{-3} m³/kg. The in-diffusion $K_{d,in-diffusion}$ is about a hundred times as low as the batch K_d value. This difference can be explained by changes in the pore water chemistry and surface area available for the sorption. The lower solution-to-bentonite ratio affects the chemistry of

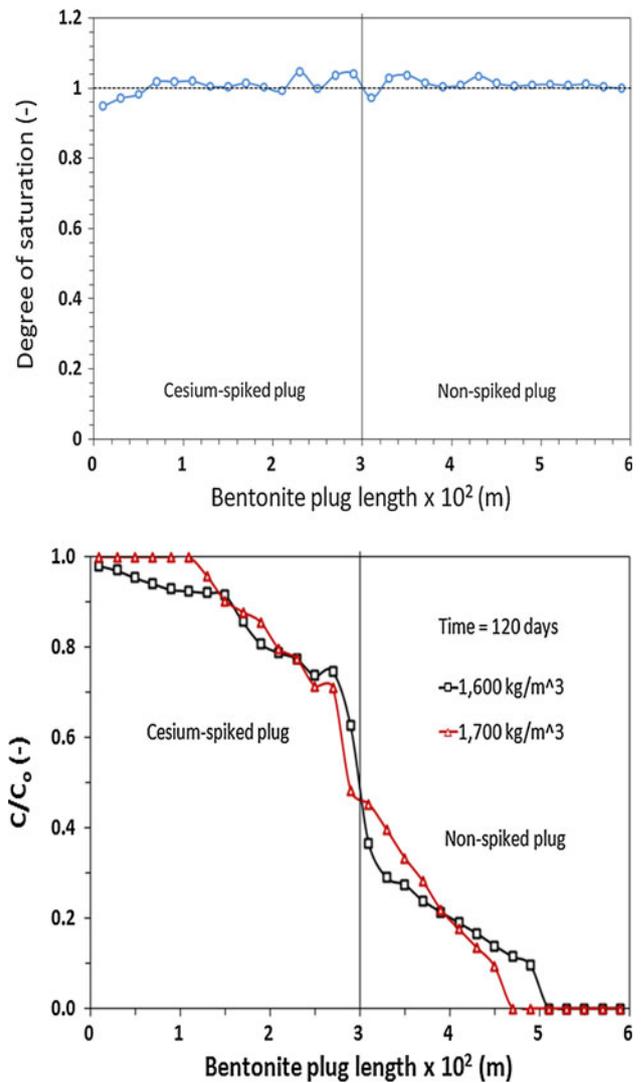


Fig. 8 Water content distribution (*upper*) in the compacted bentonite plugs and concentration profiles (*lower*) of cesium ions for the dry densities of 1,600 and 1,700 kg/m³, after the in-diffusion test

the pore water as additional ions are leached or dissolved from the bentonite (Oscarson and Dixon 1989), and it also reduces the surface area available for the sorption of cesium ions onto the bentonite by its compaction (Lee et al. 1997; Oscarson et al. 1994). Thereby, the lower distribution coefficients are obtained at a lower solution-to-bentonite ratio.

From the aspect of predicting the transport of ions for a safety and performance assessment of the compacted bentonite buffer, this is significant as the use of the distribution coefficient obtained from the batch test may underestimate the ion transport prediction. Therefore, it will be necessary for better confidence in the transport prediction to ensure that the distribution coefficients obtained at higher solution-to-bentonite ratios (in the loose bentonite of batch test) do not differ markedly from those

Table 3 Distribution coefficients ($K_{d,\text{in-diffusion}}$) obtained from the in-diffusion tests

Dry density, ρ_d (kg/m ³)	Porosity, ε (-)	Impedance factor, f (-)	D_a (m ² /s)	$K_{d,\text{in-diffusion}}^*$ (m ³ /kg)	Remarks
1.6	0.407	0.091	2.18×10^{-11}	4.5×10^{-3}	Initial cesium concentration = 10^{-2} M Solution-to-bentonite ratio = 0.25×10^{-3} m ³ /kg
1.7	0.370	0.077	2.18×10^{-11}	4.3×10^{-3}	Initial cesium concentration = 10^{-2} M Solution-to-bentonite ratio = 0.22×10^{-3} m ³ /kg

* $K_{d,\text{in-diffusion}} = \left(\frac{D_o f}{D_a} - 1\right) \frac{\varepsilon}{\rho_d}$, where D_o is the diffusion coefficient of cesium ions in pure water, and D_a is the apparent diffusion coefficient of cesium ions in compacted bentonite plug

at lower ratios (in the compacted bentonite of in-diffusion test), and if they do, to understand how and why they differ.

Conclusions

Batch tests and in-diffusion tests were carried out to investigate the sorption of cesium and iodide ions onto the KENTEX-bentonite. The cesium ions were highly sorbed on the bentonite and the experimental data fitted the Freundlich isotherm well. The distribution coefficient, K_d , of the cesium ion was variably affected by the chemistry of the solution and temperature. An increasing pH of the solution increased the K_d . However, there were different K_d values that decrease with an increase in the initial ion concentration of the solution, salinity, and temperature. The iodide ions, on the contrary, were negligibly sorptive owing to anion exclusion from the negative surface charge of bentonite particles. The K_d values obtained from the in-diffusion tests were quite lower than those from the batch tests. The difference was explained by changes in the pore water chemistry and surface area available for the sorption. Also this suggests that better confidence in the prediction of ion transport requires more care when the distribution coefficient is used as an input data for ion transport models.

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