Measurements of Apparent Diffusion Coefficients (Da) for Cs (I), Ni (II)and Se(IV) in Bentonite with Silica Sand

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Measurements of Apparent Diffusion Coefficients (Da) for Cs(I), Ni(II) and Se(IV) in Bentonite with Silica Sand

Haruo Sato[*]

Abstract

Apparent diffusion coefficients (Da) of Cs (Cs^+), Ni (Ni^{2+}) and Se (SeO_3^{2-}) in a Na-bentonite (Kunigel-V1®) were measured for a dry density of 1.8 $Mg \cdot m^{-3}$ with silica sand of 30 wt% at room temperature by in-diffusion method to evaluate the effect of the mixture of silica sand on Da in bentonite. The experiments for Cs and Ni were carried out under aerobic condition, but those for Se which is redox sensitive were carried out in an Ar glove-box (O_2 concentration < 0.1 ppm). Consequently, no significant effect of silica sand mixture to the bentonite on Da values of Cs and Se was found, and the obtained Da values were approximately the same as those in the system without silica sand reported so far. On the other hand, Da values of Ni in bentonite with silica sand were 2 orders of magnitude lower than those in bentonite without silica sand obtained to date. The Da values of Ni reported so far were obtained using stable isotopic tracer and a tracer solution with fairly high Ni concentration compared with concentration used in this study was introduced. Additionally, it is known that distribution coefficient (Kd) of Ni on Na-montmorillonite which is the major constituent clay mineral of Kunigel-V1® decreases with increasing Ni concentration. Based on this, the abrupt decrease in Da values of Ni for bentonite with silica sand is considered to be due to the difference of sorption caused by the difference of Ni concentration in the porewater of bentonite.

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珪砂混合系ベントナイト中の Cs(I), Ni(II), Se(IV)の 見掛けの拡散係数（Da）の取得

（研究報告）　　　　　　佐藤治夫[*]

要　旨

　ベントナイト中の見掛けの拡散係数（Da）に及ぼす珪砂混合の影響を評価するため、Na 型ベントナイト（クニゲル V1）を対象として、Cs (Cs^+), Ni (Ni^{2+}), Se (SeO_3^{2-})の Da を乾燥密度 $1.8 Mg \cdot m^{-3}$、珪砂混合率 30wt%、室温の条件にて in-diffusion 法により測定した。Cs 及び Ni については大気雰囲気下で、また、酸化還元電位に鋭敏な Se については Ar 雰囲気の低酸素条件（酸素濃度 0.1ppm 未満）で測定した。その結果、Cs 及び Se の Da には珪砂混合の影響は認められず、得られた Da は、これまでに報告されている珪砂が混合されていない系での値と同程度であった。一方、Ni については、珪砂混合系での Da は混合されていない系での値より 2 桁小さい値が得られた。これまでに報告されている Ni の Da は安定同位体のトレーサを用いて測定されており、本研究で用いた Ni 濃度よりかなり高い濃度のトレーサが使用された。さらに、クニゲル V1 の主要粘土鉱物である Na モンモリロナイトに対する Ni の分配係数（Kd）は、Ni 濃度の増加に伴って小さくなることが報告されている。このことから、珪砂混合系でのベントナイト中の Ni の Da が大きく減少したのは、間隙水中での Ni 濃度の違いによる収着の違いによるものと考えられる。

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1. INTRODUCTION

In performance assessment of the geological disposal of high-level radioactive waste (HLW), the diffusion of radionuclides in bentonite composing the multibarrier system is one of the important parameters to quantitatively evaluate the nuclide migration. Particularly since apparent diffusion coefficient (Da) also includes retardation, it is important to derive distribution coefficient (Kd) of nuclide in compacted state.

For Da of nuclide in compacted bentonite, much data have been reported for Na-bentonites (Kunigel-V1® and Kunipia-F®) so far[e.g. 1-5]. However, these data were all obtained in the system without silica sand and the effect of the mixture of silica sand in bentonite is unclear. For the effect of the mixture of silica sand in bentonite, Da values of U and Fe in Kunigel-V1® have been reported for a composition of 40 wt% by Idemitsu et al. and no effect of the mixture of silica sand is found[6, 7]. Additionally Da values of HTO in Kunigel-V1® have been reported for a composition range of 30 to 50 wt% and it is reported that no effect of the mixture of silica sand in bentonite for this range is found[8]. For the effect of the mixture of silica sand in bentonite on effective diffusion coefficient (De), De values of ^{125}I (I^-), ^{36}Cl (Cl^-), ^{14}C ($CO_3^{2-}+HCO_3^-$)[9] and HTO[10] in Kunigel-V1® have been reported as a function of the composition of silica sand for a dry density of 1.6 Mg•m^{-3} by Kato et al. The De values showed a tendency to increase with increasing the composition of silica sand in the bentonite, but it was insignificant up to the composition 70 wt%. This indicates there is a possibility that the composition of silica sand in bentonite insignificantly affects pore structural parameters in diffusion.

As described above, it is predicted that the effect of the mixture of silica sand in bentonite on diffusion is insignificant. However, for the effect of that on Da which includes also the effect of sorption, not so much data have been reported so far.

In this study, Da values of Cs, Ni and Se in bentonite with silica sand which no datum exists, were obtained and the effect of the mixture of silica sand in the bentonite on these Da values was discussed.

2. EXPERIMENTAL

2.1 Experimental Conditions

Table I shows, summarizing experimental conditions for the diffusion experiments of Cs, Ni and Se. All measurements have been carried out for a bentonite dry density of 1.8 Mg•m^{-3} with silica sand of 30 wt%.

Table I Experimental conditions for the diffusion experiments

Bentonite	Kunigel-V1® (composition of Na-smectite, 46~49wt%)
Dry density	1.8 Mg•m^{-3}
Composition of silica sand	30 wt% (mixture of silica sand with particle sizes of 1~5 mm and 0.1~1 mm)
Temperature	room temperature
Tracer	Cs : 5x10^4 Bq•ml^{-1} ^{137}CsCl solution
	Ni : 5x10^4 Bq•ml^{-1} ^{63}NiCl$_2$ solution
	Se : 1M Na$_2$SeO$_3$ solution
Introducted tracer quantity	^{137}Cs : 1.25 kBq ^{63}Ni : 1.25 kBq
	Se : 2.5x10^{-5} mol
Atmosphere	Cs, Ni : aerobic conditions
	Se : anaerobic conditions (Ar atmosphere) (O$_2$ concentration < 0.1 ppm)
Producibility	n=2

The experiments for Se were all carried out using stable isotopic tracer.

2.2 Diffusion Experiments for Cs and Ni

Apparent diffusion coefficients of Cs and Ni in a Na-bentonite (Kunigel-V1®) were measured for a dry density of 1.8 Mg•m^{-3} with silica sand of 30 wt% at room temperature by in-diffusion method[e.g. 2]. The bentonite was dried at 110 °C more than 24 hours and filled in the cylindrical space of a diffusion column together with silica sand to obtain a dry density of 1.8 Mg•m^{-3} with the size of φ20x20 mm. The column with bentonite was saturated with distilled water more than 10 days under atmospheric pressure after degassed a half hour in a vacuum chamber.

Figure 1 shows the sectional view of a pair of difusion columns. The diffusion column is made of stainless steel and a cylindrical space of 20 mm in diameter and 20 mm in thickness is cored inside of the diffusion column so that bentonite can be filled in it. A small amount of tracer (25 μl: 1.25 kBq) with a concentration of 50 kBq•ml^{-1} was pipetted on the surface of one end of each bentonite and 2 columns were connected at the surface of the bentonite where the tracer was pipetted as shown in **Figure 1** and allowed to diffuse for 25 to 56 days (25 days for Cs, 48 and 56 days for Ni).

At the end of all experiments, the cylindrical bentonite was pushed out and cut with a knife into 1 mm pitched slices. Each slice was immediately weighed and dried at 110 °C over 24 hours to obtain water content and to calculate accurately distance sliced. The slices were immersed in a 2 ml sodium hexametaphosphate solution for 3 hours shaking to extract tracer from the slices and then a liquid scintillator (PICO-FLOUR 40, PACKARD) of 18 ml was added. The concentrations of Cs-137 and Ni-63 were analyzed with a liquid scintillation counter and the concentration profiles in the bentonite were determined based on the analyzed data.
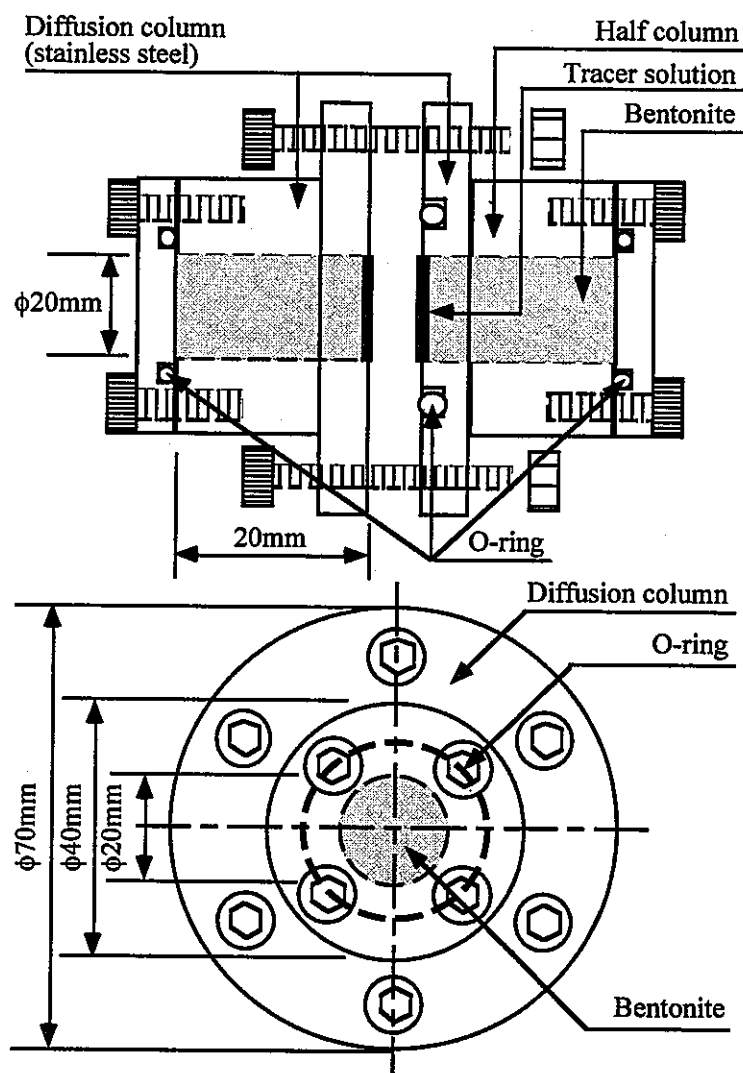
Although the slicing of bentonite

Figure 1 Sectional view of a pair of diffusion columns

is carried out by pushing out using a pushing tool with gauge, it is difficult to accurately slice with constant thickness due to pushing error. Therefore, depth sliced from the surface of bentonite was determined based on each slice weight in this study. In this case, distance from the surface of bentonite where tracer was pipetted, was calculated by the following equation.

$$X_i = X_{i-1} + \frac{W_i + W_{i-1}}{2 \sum_{i=1}^{n} W_i} \cdot L \qquad (1)$$

Where X_i is the distance from the surface of bentonite where tracer was pipetted for the i-th slice (mm), W_i is the weight before dried for the i-th slice (g), L is the thickness of the bentonite specimen (mm), and n is the number of total slice (–).

2.3 Diffusion Experiments for Se

Since Se is sensitive to redox condition, diffusion experiments for Se were carried out under anaerobic conditions to keep Se(IV) (species: SeO_3^{2-}). Selenium is found in oxidation states between –II and VI. In the pH range of natural waters, SeO_4^{2-} is predominant under strongly oxidising conditions, while HSe^- dominates under reducing conditions[11]. For Se, all experiments were carried out in an Ar glove-box, in which oxygen concentration was kept < 0.1 ppm. In this condition, Se is considered to take SeO_3^{2-} in the porewater of bentonite. The drying and filling of bentonite were carried out in the same procedure as that of Cs and Ni. Diffusion column filled bentonite and silica sand was removed for air by exchanging with Ar gas in a
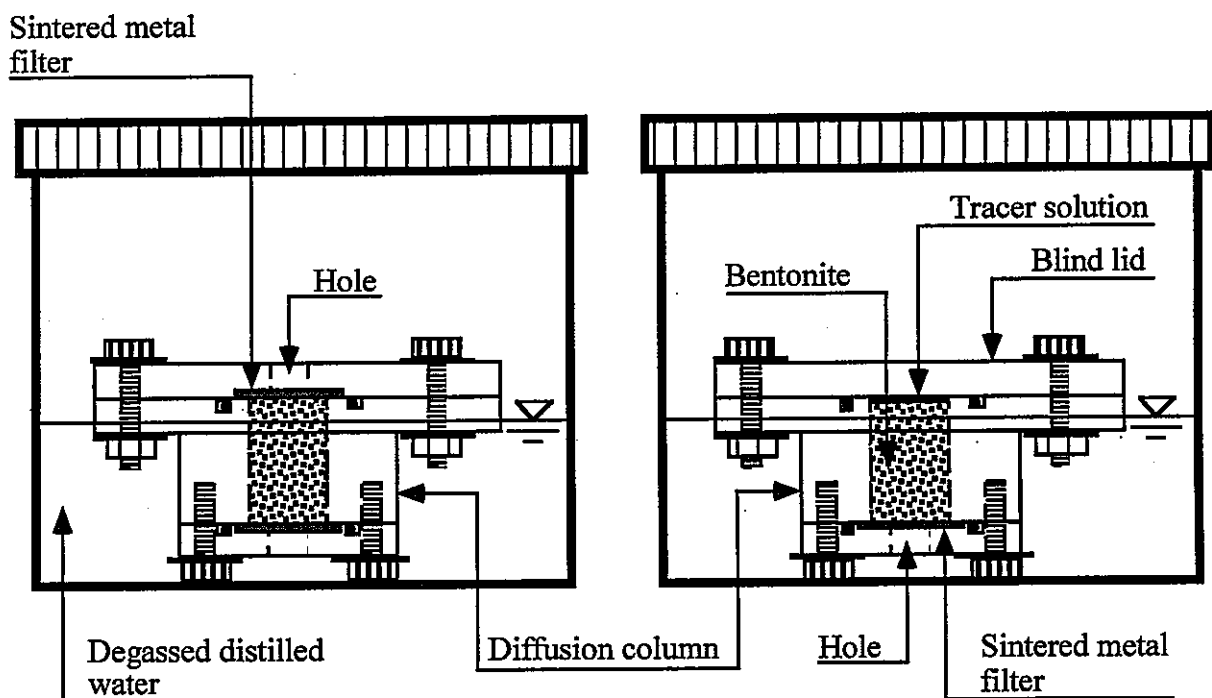

Figure 2 An image of the saturation of bentonite (left side) and diffusion experiment (right side)

vacuum chamber and then transferred into the glove-box. The column with bentonite was then saturated with degassed distilled water more than 10 days under atmospheric pressure after degassed a half hour in a vacuum chamber. The degassed distilled water was prepared by bubbling with atmospheric gas in the glove-box.

Figure 2 shows an image of the saturation of bentonite (left side) and diffusion experiment (right side). The saturation of bentonite and introduction of tracer were carried out in the same way as that in diffusion experiments of Se(−II) for reducing condtions[11]. Where no reductant is added in this study, because the experiment is not under reducing condition. After the saturation of the bentonite, a small amount of a 1M Na_2SeO_3 solution (25 μl: 2.5×10^{-5} mol) was pipetted on the surface of one end of each bentonite and a blind lid was then sealed shut. The other end of the column remained in contact with the degassed distilled water via a sintered metal filter as shown in **Figure 2** (right side) and allowed to diffuse for about 13 days. At the end of the experiment, the bentonite in the column was pushed out and cut with a knife into 1 mm pitched slices. Each slice was immediately weighed and dried at 110 °C over 24 hours similarly to procedure of Cs and Ni. The distance from the surface of bentonite where tracer was pipetted, was determined similarly to that for Cs and Ni. The slices were immersed in distilled water of a volume of 80 ml over 7 days to extract Se from the slices. The extracted solutions were then passed through a 10000 MWCO (Molecular Weight Cut Off) ultrafilter and analyzed for Se concentration using atomic adsorption spectroscopy (AAS).

3. DIFFUSION THEORY

The calculations of Da values were based on Fickian law[12]. The diffusion equation for a one-dimensional non-steady state is generally expressed by the following equation.

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x}\left(Da \cdot \frac{\partial C}{\partial x}\right) \tag{2}$$

Assuming that Da is independent of position, equation (2) is rewritten as the following equation.

$$\frac{\partial C}{\partial t} = Da \cdot \frac{\partial^2 C}{\partial x^2} \tag{3}$$

Where C is the concentration of the tracer in the bentonite ($kg \cdot m^{-3}$), t is the diffusing time (s), Da is the apparent diffusion coefficient ($m^2 \cdot s^{-1}$), and x is the distance from the source (m).

For soluble elements, the analytical solution in case of an instantaneous planar source can be applied for the calculation of Da. On the other hand, for elements which solubilities in the porewater of bentonite are low, there is a possibility that precipitation occurs on the surface of the bentonite after the tracer solution was pipetted. In this case, the boundary concentration is presumed to be controlled by the solubility of the tracer.

For one-dimensional diffusion of a planar source consisting of a limited amount of substance in a cylinder of infinite length, the analytical solution for equation (3), based on initial and boundary conditions, is given by the following equation[12].

Initial condition

$C(t, x) = 0, t = 0, x \neq 0$

Boundary condition

$C(t, x) = 0, t > 0, x = \pm \infty$

$$M = \int_{-\infty}^{\infty} C \, dx$$

$$C = \frac{M}{2\sqrt{\pi Da \cdot t}} exp\left(-\frac{x^2}{4Da \cdot t}\right) \tag{4}$$

Where M is the total amount of tracer added per unit aera (kg•m^{-2}). This analytical equation is applied for diffusion in 2 directions from one source.

From equation (4), taking log C and x^2 as the vertical and horizontal axes, respectively, the slope can be used to derive Da at the measurement time. The Da values for Cs and Se were determined by a least squares fitting based on this method.

If bentonite sample is assumed to be a semi-infinite medium, the analytical solution for the boundary condition of constant concentration is derived as the following equation on the basis of initial and boundary conditions[12].

Initial condition

$C(t, x) = 0, t = 0, x > 0$

Boundary condition

$C(t, x) = Co, t > 0, x = 0$

$$\frac{C}{Co} = erfc\left(\frac{x}{2\sqrt{Da \cdot t}}\right) = 1 - erf\left(\frac{x}{2\sqrt{Da \cdot t}}\right) \tag{5}$$

Where Co is the boundary concentration (kg•m^{-3}), *erf* is the error function, and *erfc* is the complementary error function.

The error function is defined by the following equation.

$$erf(z) = 1 - erfc(z) = \frac{2}{\sqrt{\pi}} \int_0^z exp\left(-\eta^2\right) dx \tag{6}$$

The Da values for Ni were calculated by fitting equation (5) to the concentration profiles of Ni in bentonite.

4. RESULTS AND DISCUSSION

4.1 Diffusion Behaviour of Cs and Se

The concentrations of Cs and Se in bentonite gradually decreased with increasing distance from the surface of bentonite where those tracer solutions were pipetted (see **Appendices I and II** for Cs and Se, respectively). **Figure 3** shows Da values of Cs and Se as a function of

bentonite dry density and the composition of silica sand in the bentonite. **Table II** shows Da values of Cs and Se obtained for a bentonite dry density of 1.8 $Mg \cdot m^{-3}$ with silica sand of 30 wt%. The plots simultaneously show also Da data obtained in the system without silica sand so far to compare[5]. Incidentally Da values quoted from the literature of [5] were also obtained in the same condition. Therefore, the dominant species of Se in the porewater of bentonite is presumed to be SeO_3^{2-}. Although Da values of Se for the system with silica sand of 30 wt% are a little lower than those for the system without silica sand, significant effect of the mixture of silica snad to Kunigel-V1® on Da was not found.

Similar tendency is reported also for the diffusion of HTO[8]. Namely Da values for HTO in bentonite were obtained as a function of bentonite dry density and the composition of silica sand in the bentonite. No effect of the composition of silica sand (0~50 wt%) on Da of HTO in bentonite was found over the density. Since HTO is a non-sorbing nuclide, the results described above show that the pore structure of compacted bentonite is not significantly affected by the mixture of silica sand to bentonite.

Additionally Da values of U and Fe in Kunigel-V1® are reported for a silica sand composition of 40 wt% in a bentonite dry density range of 1.6 to 1.8 $Mg \cdot m^{-3}$ by Idemitsu et al.[6, 7]. These both studies were carried out for aerobic and reducing conditions, and no effect of the mixture of silica sand to bentonite on Da values of U and Fe was found over the dry density and redox condition. Since the effect of the mixture of silica sand in bentonite
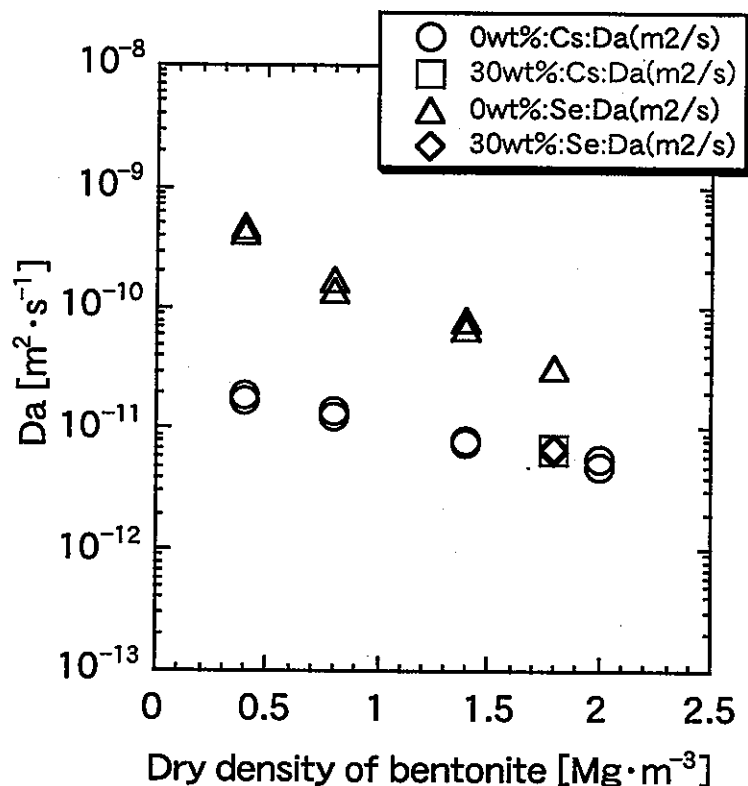

Figure 3 Da values of Cs and Se as a function of bentonite dry density and the composition of silica sand in the bentonite

$\bigcirc$, $\triangle$: [5], $\square$, $\diamondsuit$: this work

Table II Da values of Cs and Se obtained for a bentonite dry density of 1.8 $Mg \cdot m^{-3}$ with silica sand of 30 wt%

Element (nuclide)	Species	Atmosphere	Diffusing time[day]	Da $[m^2 \cdot s^{-1}]$
Cs-137	Cs^+	aerobic	24.9	2.7E–12
Cs-137	Cs^+	aerobic	25.0	6.6E–12
Se	SeO_3^{2-}	anaerobic	13.1	1.1E–11
Se	SeO_3^{2-}	anaerobic	13.1	1.2E–11

on the pore structure of compacted bentonite is insignificant from the results of diffusion experiments of HTO[8], it is presumed that no effect of silica sand mixture on Da values of U and Fe was found is because the effect of the mixture of silica sand on the sorption of U and Fe was small. Similarly, since no effect of silica sand mixture to bentonite on Da values of Cs and Se was found, it is presumed that the effect of the mixture of silica sand on the sorption of Cs and Se is also small.

4.2 Diffusion Behaviour of Ni

The concentrations of Ni in bentonite abruptly decreased with increasing distance from the surface of bentonite where tracer was pipetted (see **Appendix III**). Nickel scarcely diffused in the bentonite and the concentrations of Ni approximately became background level around 3 mm deep from the surface of the bentonite. Duplicate experiments were carried out for the same condition and similar profiles were obtained. **Figure 4** shows Da values of Ni as a function of bentonite dry density and the composition of silica sand in the bentonite. **Table III** shows Da values of Ni obtained for a bentonite dry density of 1.8 $Mg \cdot m^{-3}$ with silica sand of 30 wt%. The plots simultaneously show also Da data for Ni (Ni^{2+}) obtained in the system without silica sand so far to compare both[4]. The Da values of Ni quoted from the literature of [4] were also obtained in the same condition. Where although radioactive tracer (Ni-63) was used in this study, stable isotopic tracer ($NiCl_2$
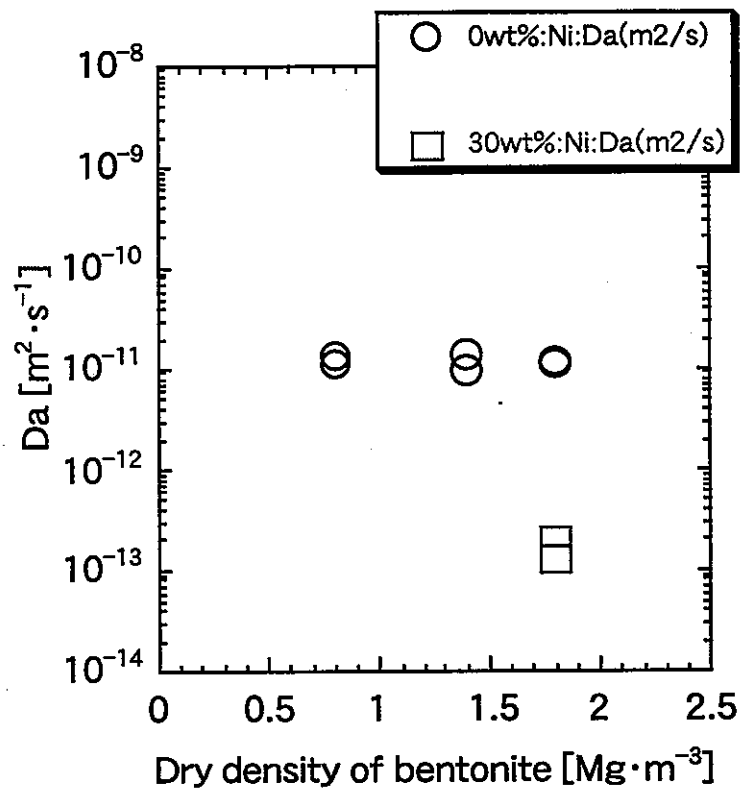

Figure 4 Da values of Ni as a function of bentonite dry density and the composition of silica sand in the bentonite
○: [4], □: this work

Table III Da values of Ni obtained for a bentonite dry density of 1.8 $Mg \cdot m^{-3}$ with silica sand of 30 wt%

Element (nuclide)	Species	Atmosphere	Diffusing time[day]	Da [$m^2 \cdot s^{-1}$]
Ni-63	Ni^{2+}	aerobic	48.0	1.9E–13
Ni-63	Ni^{2+}	aerobic	56.0	1.3E–13

solution) was used in the literature of [4].

As shown in **Figure 4**, Da values of Ni in bentonite with silica sand are much lower than those in bentonite without silica sand. The gap between Da values of Ni for both bentonites is about 2 orders of magnitude, being quite large. It can not be seen that silica sand of 30 wt% added to bentonite causes such large gap. Since the pore structure of compacted bentonite is not affected by the mixture of silica sand of 30 wt%, it is presumed that Da values of Ni abruptly decreased is because the sorption of Ni on bentonite was drastically affected.

For the diffusion experiments of Ni in bentonite without silica sand described above[4], a tracer solution with relatively high Ni concentration (0.01 M $NiCl_2$) was introduced on the surface of bentonite specimen, because stable isotopic tracer was used. For the sorption of Ni, concentration dependencies (sorption isotherms) for Kd on Na-montmorillonite which is the major constituent clay mineral of Kunigel-V1[®], are reported for various pH and Kd values of Ni decrease from around 10^{-8} M with increasing Ni equilibrium concentration[13]. Although the sorption behaviour of Ni depends on pH, Kd values decrease by approximately one order of magnitude while Ni equilibrium concentration increases by 3 orders of magnitude. In the diffusion experiments in the literature of [4], 10^{-6} mol Ni per sample was pipetted on the surface of bentonite specimen. On the other hand, 2.9×10^{-12} mol Ni per sample was introduced in this study. It is clear from each quantity of Ni tracer pipetted on the surface of bentonite specimen that the difference between both Ni concentrations in the porewater of bentonite is very large. From this matter, such large gap between Da values of Ni in both bentonites (Kunigel-V1[®], Kunigel-V1[®] with silica sand of 30 wt%) is considered to be not due to the mixture of silica sand and due to the difference of sorption caused by the difference of Ni concentration in the porewater of bentonite. For this problem, it will be experimentally studied in the future.

5. CONCLUSIONS

Apparent diffusion coefficients of Cs (Cs^+), Ni (Ni^{2+}) and Se (SeO_3^{2-}) in bentonite (Kunigel-V1[®]) with silica sand of 30 wt% were obtained for a dry density of 1.8 Mg•m^{-3}. Consequently, no significant effect of silica sand mixture to the bentonite on Da values of Cs and Se was found. To the contrary, Da values of Ni were 2 orders of magnitude lower than those in the bentonite without silica sand. Since it is reported that Kd of Ni on Na-montmorillonite which is the major constituent clay mineral of Kunigel-V1[®] decreases with increasing Ni concentration, the abrupt decrease in Da values of Ni is considered to be due to the differednce of sorption caused by the difference of Ni concentration in the porewater of bentonite.

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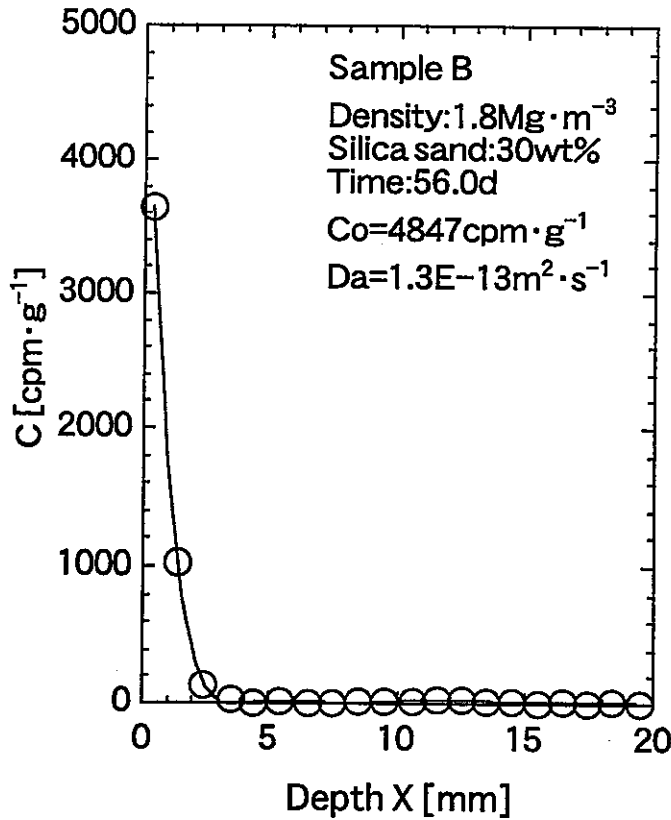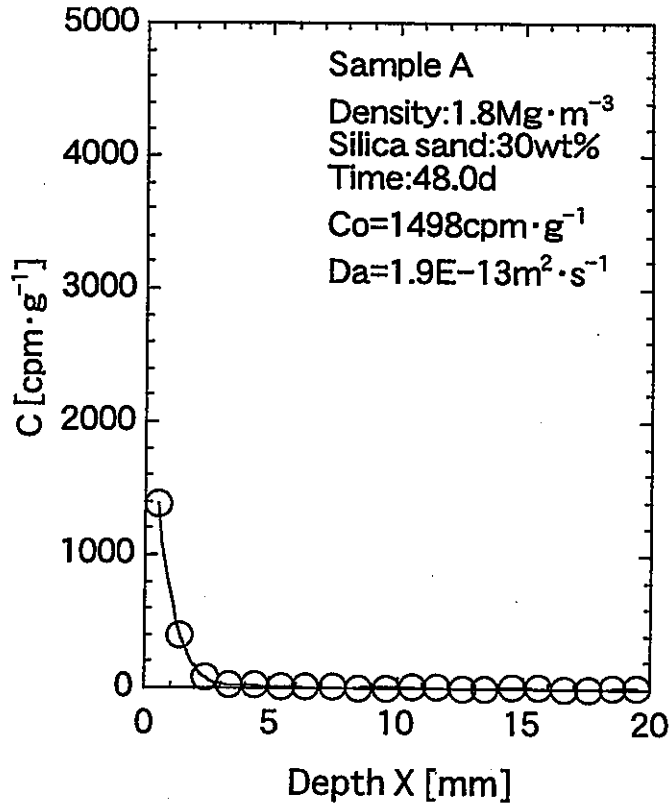
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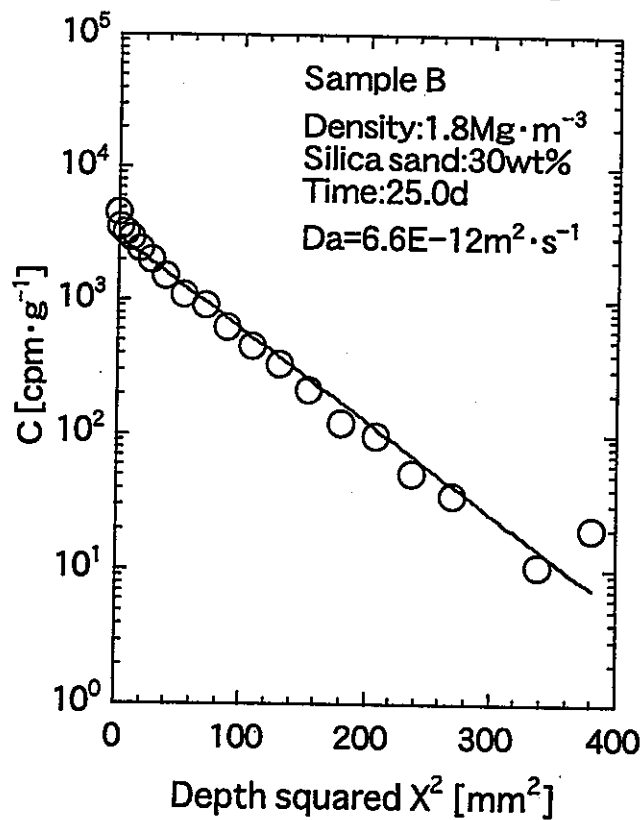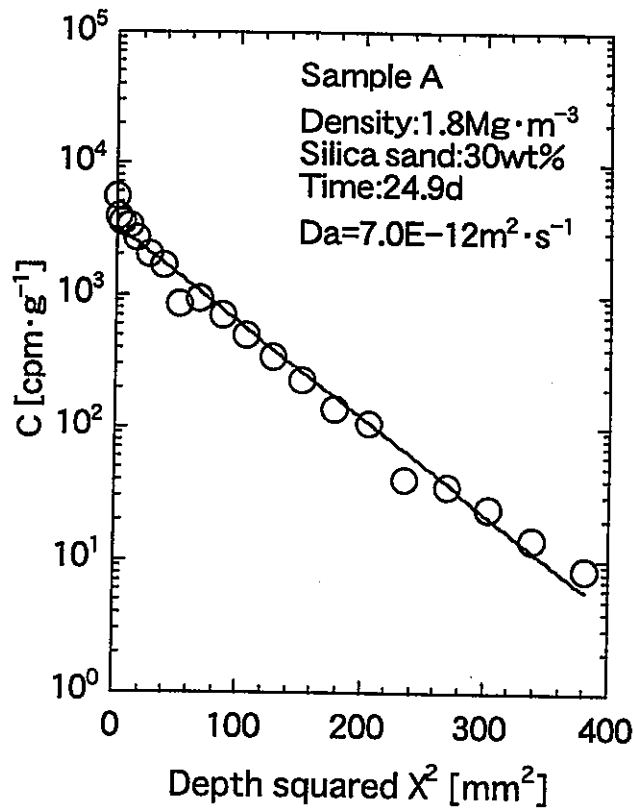
Correlations between the concentrations of Cs-137 in bentonite with silica sand of 30 wt% and depth squared from the surface of the bentonite where tracer was pipetted for a dry density of 1.8 Mg•m^{-3}
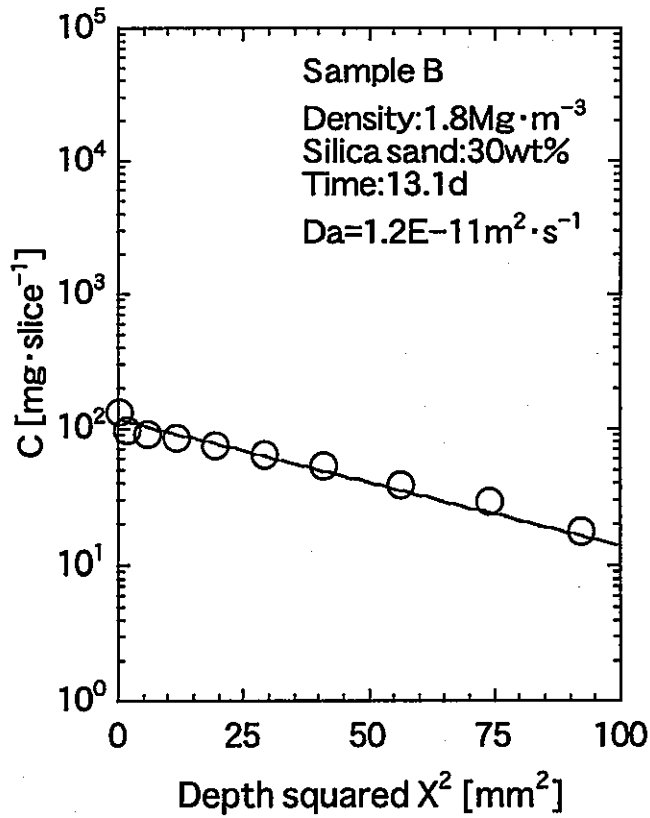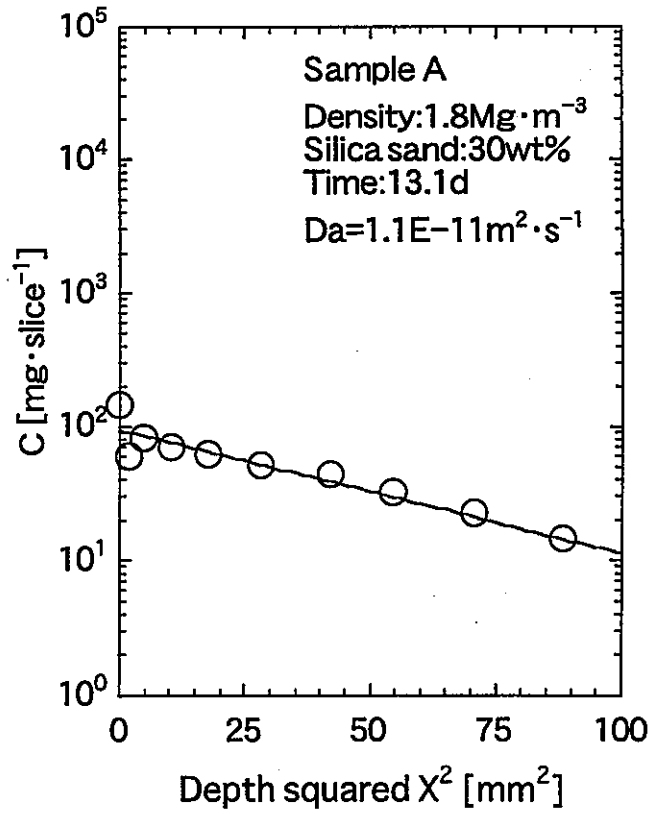
Appendix II

Correlations between the concentrations of Se in bentonite with silica sand of 30 wt% and depth squared from the surface of the bentonite where tracer was pipetted for a dry density of 1.8 Mg·m^{-3}

Appendix III

Sample A

Density:1.8Mg·m^{-3}
Silica sand:30wt%
Time:48.0d
Co=1498cpm·g^{-1}
Da=1.9E−13m^2·s^{-1}

Sample B

Density:1.8Mg·m^{-3}
Silica sand:30wt%
Time:56.0d
Co=4847cpm·g^{-1}
Da=1.3E−13m^2·s^{-1}

Correlations between the concentrations of Ni-63 in bentonite with silica sand of 30 wt% and depth from the surface of the bentonite where tracer was pipetted for a dry density of 1.8 Mg•m^{-3}