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Evaluating temperature effects on diffusive releases from clay particles

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ABSTRACT

In order to investigate how temperature affects the leaching characteristics of hazardous substances, leaching tests were conducted at four different temperature conditions (10, 20, 35, and 50°C) using clay containing geogenic heavy metals and other substances. The results showed that the larger the temperature difference, the more pronounced the effect on the amount of substances leached from the soil; in the comparison between the 10°C and 20°C cases, the diffusion coefficients obtained for the 20°C case were larger with less than twofold. Between the case of 10°C and 50°C, diffusion coefficients obtained at 50°C were 5 to 20 times larger than at 10°C. Leaching tests conducted by varying the liquid-solid ratio indicated that the total mass involved in adsorption/desorption increased with increasing temperature. These results indicate the importance of considering the temperature conditions at the site of utilization in evaluating the soil leaching ratio when using clay containing geogenic hazardous substances.

Keywords: diffusion, leaching characteristics, temperature, liquid-solid ratio,

1 INTRODUCTION

In recent years, the global movement towards a low-carbon society has accelerated, and the use of geothermal heat as a renewable energy source has been attracting attention. In the geotechnical field, a number of applications of energy piles have been reported (e.g., Akrouch et al, 2014; Rao et al, 2015; Yavari et al, 2016). Other attempts have been made to accelerate ground improvement by actively changing ground temperature (e.g. Pothiraksanon et al, 2010; Sakr et al, 2022). On the other hand, it has been pointed out that changes in the temperature of the aquifer itself and the ground in contact with it may cause deformation of the ground, increased leaching of toxic substances, and changes in the microbial environment (e.g. Bonte et al, 2011; Saito & Komatsu, 2014). Recent studies have also reported that temperature affects the leaching behavior of heavy metals and other substances in ground and waste, with leaching generally increasing at higher temperatures (e.g. Saito et al, 2018; Liu at al, 2019; Takai et al, 2020; Lu et al, 2021; Ogawa et al, 2022). Thus, it is important to clarify the effect of temperature on the leaching behavior from the ground in order to safely apply geothermal technology. In Europe, which is ahead of Japan in the use of geothermal heat, regulatory limits have been set for the temperature of reducing water when geothermal heat pump systems are used, and consideration has been given to avoid adverse effects on the surrounding groundwater environment (e.g. Haehnlein et al, 2010). On the other hand, in Japan, the temperature of ground and groundwater at a depth of 10 m or more below the ground surface has been regarded as generally constant and independent of the season. Currently, leaching tests are conducted at room temperature of approximately 20°C to evaluate the leaching of hazardous substances. However, in actual geothermal heat utilization sites, temperature changes are expected to occur due to heat extraction and heat dissipation, and leaching tests conducted at room temperature may not be adequate to reproduce the actual phenomena and thus may not be able to evaluate environmental impact appropriately. Therefore, this study attempted to understand the difference in leaching behaviour at different temperatures by conducting leaching tests under different temperature conditions. Two types of leaching tests were conducted as follows. One was a static leaching test to evaluate leaching by diffusion from soil particles to pore water, where pore water was

collected by consolidating the specimens. The other was a shaking batch test by varying the liquid-solid ratio to identify the total mass of substance involved in adsorption/desorption reactions.

2 MATERIALS AND METHODOLOGIES

2.1 Materials

Alluvial clay distributed in the Osaka Plain of Japan was used for each experiment (Figure 1). This clay is known to contain geogenic arsenic, fluorine, boron, and other elements, and their eluted amounts often exceed environmental standard values. In recent years, there have been increasing opportunities for excavation in conjunction with redevelopment projects in Osaka City. In this study, clay exposed as a result of construction work was collected and used for testing. The basic physical properties of the clay used are shown in Table 1. The particle size distribution indicates that this material consists mainly of silt fractions. Table 2 shows leaching concentration and acid-eluted concentration. Both test methods are defined by the Japanese Ministry of the Environment. For the leaching test, pure water and soil were mixed to a liquid-solid ratio of 10 and the sample was shaken for 6 hours at an amplitude of 4 cm and 200 times/minute. The supernatant solution obtained by centrifugation at 3000 G for 20 minutes was filtered through a 0.45-μm membrane filter and used as the test solution. The test solution was analyzed quantitatively following JIS K 0102. Acid-eluted concentration was measured in accordance with the Sediment Survey Method. For arsenic and boron, 5mL of nitric acid and 2mL of hydrochloric acid were added to 0.2g dry weight of soil and decomposed under pressure using a microwave. The decomposed solution was then diluted 2-fold and quantitatively analyzed using ICP-MS. For fluorine, 0.5 g dry weight of soil was steam-distilled with perchloric acid, and the fluorine in the sample was distilled out as fluorosilicic acid (H_2SiF_6), and the fluoride ions in the distillate were measured by ion electrode method. In the leaching test, arsenic, fluorine, and boron were detected below the environmental standard values. Acid-eluted concentration were high for fluorine, boron, and arsenic, in that order. Boron had a lower acid-eluted concentration than fluorine, but eluted more.

2.2 Consolidation for pore water collection

In this study, pore water was collected by consolidation as shown in Figure 2. Pore water concentration was measured for clay samples cured at different temperature conditions. First, materials passed through a non-metallic sieve with a 2 mm opening to remove foreign matter such as shells from the materials. Next, the clay material and pure water were allowed to acclimate to a predetermined temperature in a thermostatic chamber adjusted to the test temperatures (10, 20, 35, and 50°C) by leaving them for one day before mixing, and then, pure water was added to achieve a water content of 55%. The water content was set to 55% to achieve sufficient workability, when placing the sample into an acrylic container. Then, 1000 g of wet clay were filled inside the acrylic container after 3, 7, and 28 days from water content adjustment to reach a height of 8.0 cm. During the filling, the sides of acrylic cylinder were tapped with a wooden hammer to eliminate air gaps and densify the soil. The specimen was then consolidated under double-sided drainage conditions with a weight set so that a load of 100

Table 1. *Basic properties of the clay used*

Test Contents	Result	Standards
Soil density (g/cm^3)	2.65	JGS0111
Natural water content (%)	36.8	JGS0121
Specific surface area (m^2/g)	6.89	JISZ8830
Particle size		
Gravel content 2-5 mm (%)	0.0	Laser
Sand content 0.075-2 mm(%)	15.0	Diffraction/
Clay content 5-75 μm (%)	72.5	Scattering
Clay content Less than 5μm (%)	12.5	



Figure 1. *Materials used*

Table 2. *Results of leaching and total content tests*

Substances	Leaching concentration (mg/L)	Environmental Standards in Japan (mg/L)	Acid-eluted Concentration (mg/kg)
As	0.008	≤ 0.01	6.8
F	0.46	≤ 0.8	230
B	0.67	≤ 1.0	31

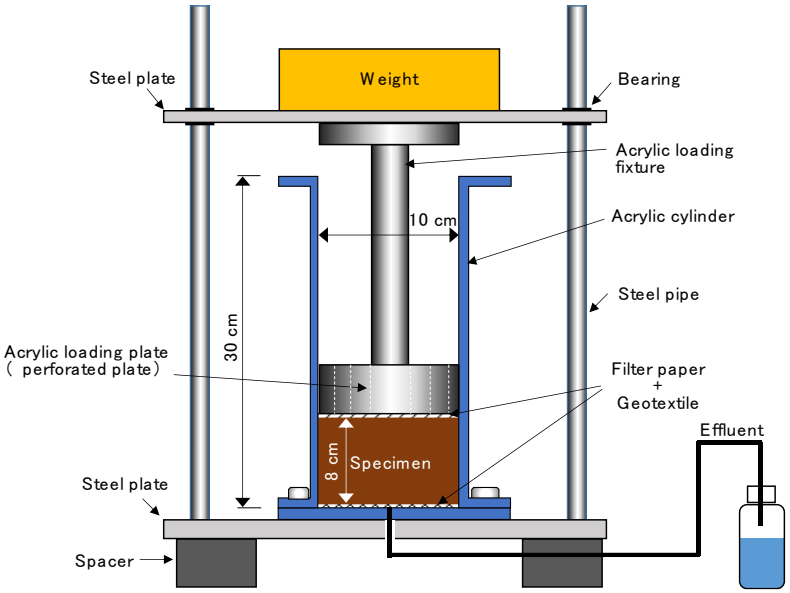


Figure 2. *Apparatus for consolidation test*



Figure 3. *Situation of shaking*

kPa was applied to the specimen. Consolidation was concluded by confirming that the amount of settlement equivalent to d_{100} was obtained by the root t method, which was theoretically considered to be the completion of primary consolidation. The pore water discharged from the consolidation test was collected by suctioning the bottom side with a PP bottle and the top side with a syringe fitted with a tube, then both were mixed in total and filtered through a 0.45 μm membrane filter and analysed according to JIS K 0102. All processes from water addition to pore water collection were conducted in a thermostatic chamber at a predetermined temperature, and only the pore water analysis was conducted at room temperature.

2.3 Leaching test

For the leaching test, a shaking batch leaching test was conducted in a thermostatic chamber controlled at predetermined temperatures (10, 20, 35, and 50°C) using consolidated specimens to see how the amount of substances released from the soil to the liquid phase changes with different temperature conditions. In addition, the liquid-solid ratio (L/S) was also varied in five patterns in this test. Sakanakura et al. (2021) proposed a method to obtain parameters related to adsorption and desorption reactions, such as distribution coefficients, by varying the liquid-solid ratio and conducting leaching tests. These parameters are very important for simulating the behavior of contaminants in the ground. In this study, we obtained the adsorption/desorption parameters from the experimental results and evaluated the effect of temperature on them. First, pure water and the specimens were placed in polyethylene bottles with a volume of 1 L so that the water/soil ratio (L/S) was 5, 10, 20, 50, and 100, respectively. The bottles were then placed in a shaking machine and shaken for 2 hours with an amplitude of 4 cm and frequency of 200 times per minute. The suspension was then divided into four 50 mL capacity centrifuge tubes and subjected to centrifugation at 3000 G for 20 minutes. The supernatant water after solid-liquid separation was filtered through a 0.45 μm membrane filter and analysed according to JIS K 0102. The work was performed in a thermostatic chamber at a predetermined temperature, except for the quantitative analysis of concentrations. Figure 3 shows the shaking equipment with vessels.

3 EXPERIMENTAL RESULT AND DISCUSSION

3.1 Effect of temperature on the concentration of substances in pore water

Figure 4 shows the relationship between temperature and pH or concentration in pore water collected from the consolidation test. The pH ranged from 7.5 to 9.0, and tended to increase with temperature. Although the cause of the pH increase was not identified in this experiment, the pH increase was more pronounced in the specimens cured for 28 days after water addition at 35°C and 50°C than in those cured for shorter periods, suggesting that not only does temperature change accelerate the dissolution

of substances from solid to liquid, but also that microbial activity has an effect. The concentrations of arsenic, fluorine, and boron in the pore water also tended to increase with temperature. Arsenic concentrations increased exponentially, with little difference between 10°C and 20°C, with concentrations ranging from 0.002 to 0.003 mg/L under both conditions, but the increase became more pronounced at 35°C and above. A comparison of arsenic concentrations between 3-day and 28-day curing shows that arsenic concentrations are about 3 times higher at 35°C and about 9 times higher at 50°C, indicating that under high temperature conditions, the longer the curing period, the higher the arsenic concentration in the pore water. The large increase in arsenic concentration in the case of 28-day curing at 50°C must take into account the effect of pH. Previous studies have shown that the amount of arsenic leached from soil increases significantly when pH is greater than 8.5 and peaks at pH 10, for example: (Takahashi et al, 2011). On the other hand, the concentration of boron and fluorine increased linearly with temperature, and the effect of different curing periods was small. Comparing concentrations at 10°C and 50°C, the increases for fluorine and boron were about 2 and 2.3 times higher, respectively, and the effects of temperature differences on both substances were quite similar.

3.2 Effect of temperature on diffusion coefficient

To calculate the diffusion coefficient using the pore water concentration obtained from the consolidation test, we first calculated the mass transfer from soil particles to pore water per unit time and unit area (leaching flux) using Equations (1) and (2).

$$q = \frac{c_i V}{m S_w} \tag{1}$$

$$J = \frac{q}{t_i} \tag{2}$$

where q: leaching volume per unit area (mg/m^2), c_i: pore water concentration (mg/L), V: initial pore water volume (L), m: dry weight of soil (kg), S_w: specific surface area of soil particles (m^2/kg), J: leaching flux (mg/m^2/s), t: elapsed time after water addition (s).

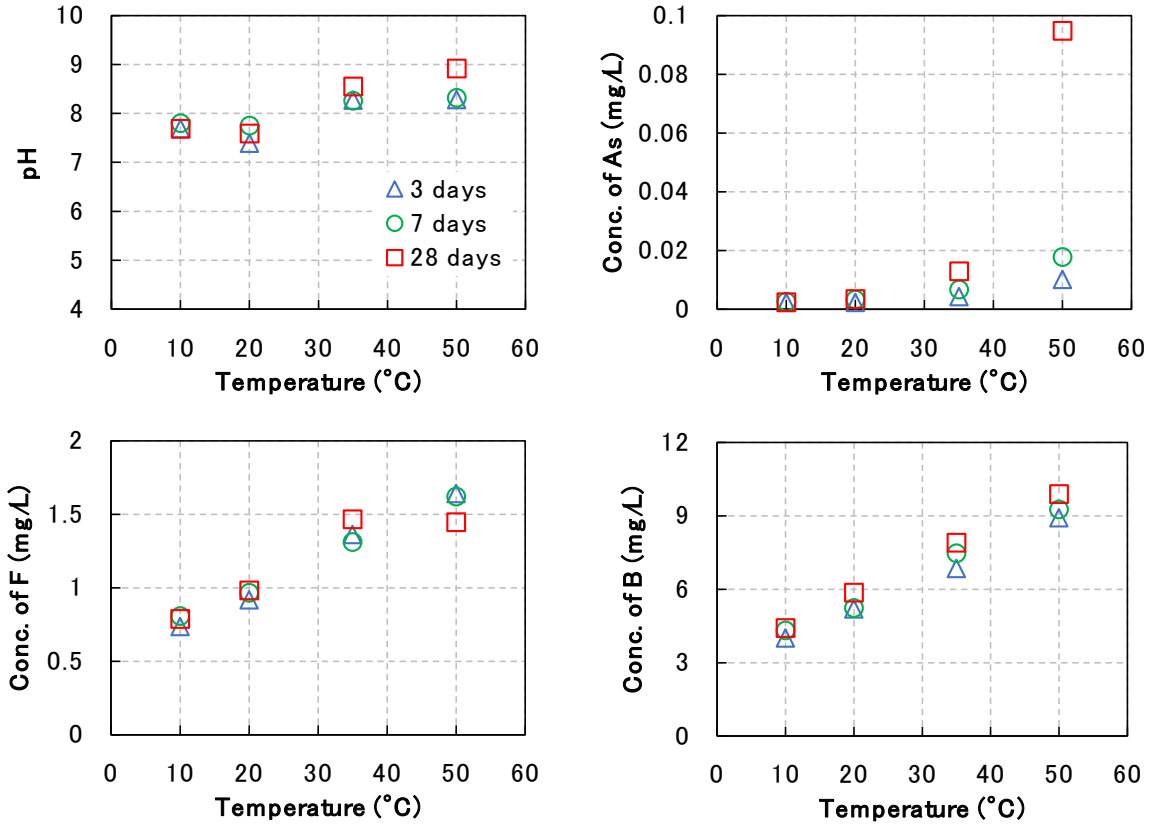


Figure 4. *Relationship between temperature and pH or concentration in pore water*

The relationship between the elapsed time after water addition and the average leaching flux is shown in Figure 5. Table 3 next to the graph shows the approximate equation for each case, its slope, and the coefficient of determination. The average elution flux of arsenic decreases with elapsed time, and its slope varies significantly with temperature conditions. At 10°C, the slope of the approximate line was almost -1.0, and the mean elution flux decreased in proportion to time. This indicates that arsenic elution was completed by day 3 and that little mass transfer from soil particles to pore water occurred after that time. At 50°C, on the other hand, the average leaching fluxes at 3, 7, and 28 days were almost the same, indicating that the amount of arsenic transferred from soil particles to pore water remained constant throughout the 28-day period. This indicates that arsenic leaching continues over a longer period of time under higher temperature conditions. The trends of fluorine and boron were in good agreement, with the slope of the approximate equation for both being approximately -1.0 at all temperature conditions, and the coefficient of determination was also very high. These results confirm that the mass transfer of fluorine and boron from soil particles to pore water converged during up to 3 days of water addition. Nakamura et al. (2014) reported that the peak concentration of fluorine was reached at a cumulative liquid-solid ratio of 1 to 4, while the concentration of arsenic increased even after a cumulative liquid-solid ratio of 10, indicating that the time until the convergence of leaching varied

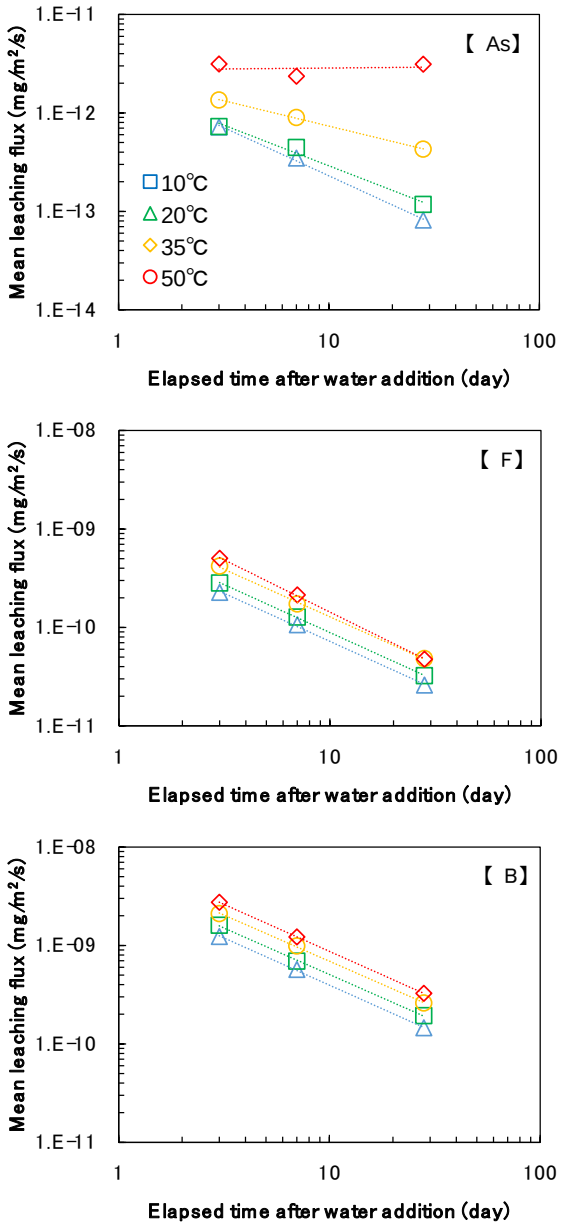


Figure 5. *Relationship between elapsed time and mean leaching flux*

Table 3-1. *Approximation equation and coefficient of determination for As*

Temperature (°C)	Equation	Gradient	R^2
10	$y = 2E\text{-}12x^{-0.982}$	-0.982	0.9974
20	$y = 2E\text{-}12x^{-0.828}$	-0.828	0.9852
35	$y = 2E\text{-}12x^{-0.516}$	-0.516	0.9994
50	$y = 3E\text{-}12x^{0.019}$	0.019	0.0176

Table 3-2. *Approximation equation and coefficient of determination for F*

Temperature (°C)	Equation	Gradient	R^2
10	$y = 7E\text{-}10x^{-0.974}$	-0.974	0.9987
20	$y = 8E\text{-}10x^{-0.971}$	-0.971	0.9998
35	$y = 1E\text{-}09x^{-0.963}$	-0.963	0.9988
50	$y = 2E\text{-}09x^{-1.059}$	-1.059	0.9997

Table 3-3. *Approximation equation and coefficient of determination for B*

Temperature (°C)	Equation	Gradient	R^2
10	$y = 4E\text{-}09x^{-0.959}$	-0.959	0.9996
20	$y = 4E\text{-}09x^{-0.944}$	-0.944	0.9996
35	$y = 6E\text{-}09x^{-0.938}$	-0.938	0.9997
50	$y = 8E\text{-}09x^{-0.954}$	-0.954	1.0000

depending on the substance, which is consistent with the trend in this study.

Diffusion coefficients from the soil particles to the pore water were calculated from the leaching fluxes obtained in the above calculations. First, the mass transfer from soil particles to pore water under saturated conditions as in this experiment is modeled in Figure 6. The leaching flux can be expressed by Equation (3) from Fick's first law.

$$J = -D\frac{dc}{dx} \tag{3}$$

where J : leaching flux (mg/m^2/s), D : diffusion coefficient (m^2/s), c : concentration (mg/L), x : distance from the interior of the soil particle to the surface (m). The concentration c is a function of time t and distance x. Therefore, it can be expressed from Fick's second law as equation (4).

$$\frac{\partial c}{\partial t} = -D\frac{\partial^2 c}{\partial x^2} \tag{4}$$

Assume that the concentration of pore water (c_1) is zero, and the concentration of the deepest soil particle (c_0) is maintained at the initial concentration. Equation (5) is then obtained by solving under the following conditions.

$t = 0,\ \ x > 0;\ \ c = c_0$

$t > 0,\ \ x = 0;\ \ c = 0$

$t > 0,\ \ x = \infty;\ \ c = c_0$

$$J = c_0\sqrt{\frac{D}{\pi t}} \tag{5}$$

The diffusion coefficient obtained from this experiment refers to the diffusion transfer caused by the concentration gradient between soil particles and pore water, i.e., it includes the diffusion transfer within the solid layer and the liquid phase (boundary membrane) that does not contribute to water permeability. As shown in Figure 5, some substances stopped eluting after 3 days, so the diffusion coefficient was calculated from Equation (5) using the leaching fluxes up to 3 days. The relationship between temperature and diffusion coefficients is shown in Figure 7. The diffusion coefficients of all substances increased with increasing temperature, with arsenic, fluorine, and boron increasing approximately 20, 5, and 5 times, respectively, between 10°C and 50°C. This indicates that arsenic, fluorine, and boron in soil particles are more likely to migrate into pore water at higher temperatures. On the other hand, arsenic, fluorine, and boron were about 1.0, 1.5, and 1.7 times higher, respectively, than those at 10°C and 20°C, indicating that the effect of temperature is small under low-temperature conditions. Therefore, the results suggest that when ground temperature is varied at actual sites, the effect on the surrounding environment can be controlled if the temperature can be controlled so that the range of variation is

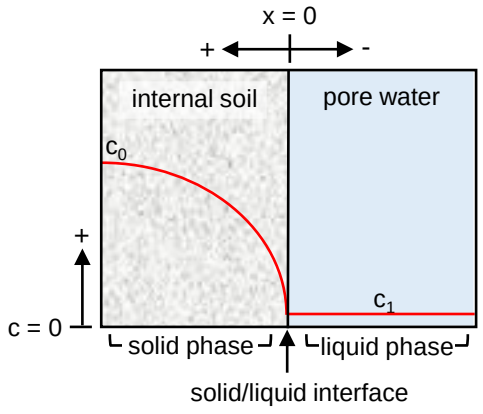


Figure 6. *Diffusion model*

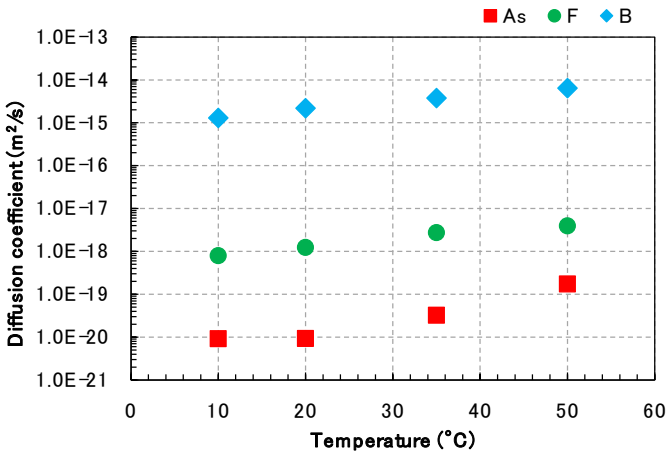


Figure 7. *Relationship between temperature and diffusion coefficient*

small. In this experiment, it was confirmed that the mass of transfer of substances from soil particles to pore water varies with temperature, which is attributed to the increase or decrease in diffusion coefficient caused by temperature change. Since activation of Brownian motion in the solid layer and decrease in viscosity of pore water are possible causes of the change in diffusion coefficient with temperature, further research should be conducted to elucidate the contribution of these factors.

3.3 Effect of temperature on the mass of substance involved in adsorption/desorption reaction

Figure 8 shows the results of leaching tests conducted by changing the liquid-solid ratio. Generally, the larger the liquid-solid ratio, the smaller the concentration in the liquid phase, and the same tendency was confirmed in this study. For the same liquid-solid ratio, the higher the temperature, the more arsenic, fluorine, and boron were eluted. For arsenic, the effect of temperature was observed in the range of liquid-solid ratio from 5 to 100, while for fluorine and boron, the effect of temperature became smaller as the liquid-solid ratio increased, and there was almost no difference in the amount of arsenic eluted at liquid-solid ratios of 50 or higher. The amount of arsenic eluted decreased linearly from a liquid-solid ratio of 5 to 50, but for fluorine and boron, there was a sharp decrease from a liquid-solid ratio of 5 to 20, and the change became smaller for larger liquid-solid ratios. Figure 9 shows the relationship between the solid-liquid contact area and the mass of substances in the solvent. Here, the solid-liquid contact area is the surface area of soil particles in the solvent, and was calculated by multiplying the specific surface area shown in Table 1 by the dry weight of the soil. For fluorine and boron, the relationship between the solid-liquid contact area and the mass of substance in the solvent is generally proportional if the solid-liquid contact area is 200 m^2 or less. As the solid-liquid contact area increases above 200 m^2, the deviation from the tangent line through the origin increases. On the other hand, for arsenic, the relationship between the solid-liquid contact area and the mass of arsenic in the solvent is proportional when the solid-liquid contact area is 70 m^2 or less, but the increase in the amount of arsenic in the solvent becomes slower above that level, and almost converges at 350 m^2. Thus, in this test, it was confirmed that the mass transfer from soil particles to solvent is not simply proportional to the solid-liquid contact area under small liquid-solid ratio conditions. This could be due to adsorption on the soil particle surface or co-precipitation with other substances. The results of the liquid-solid ratio batch tests

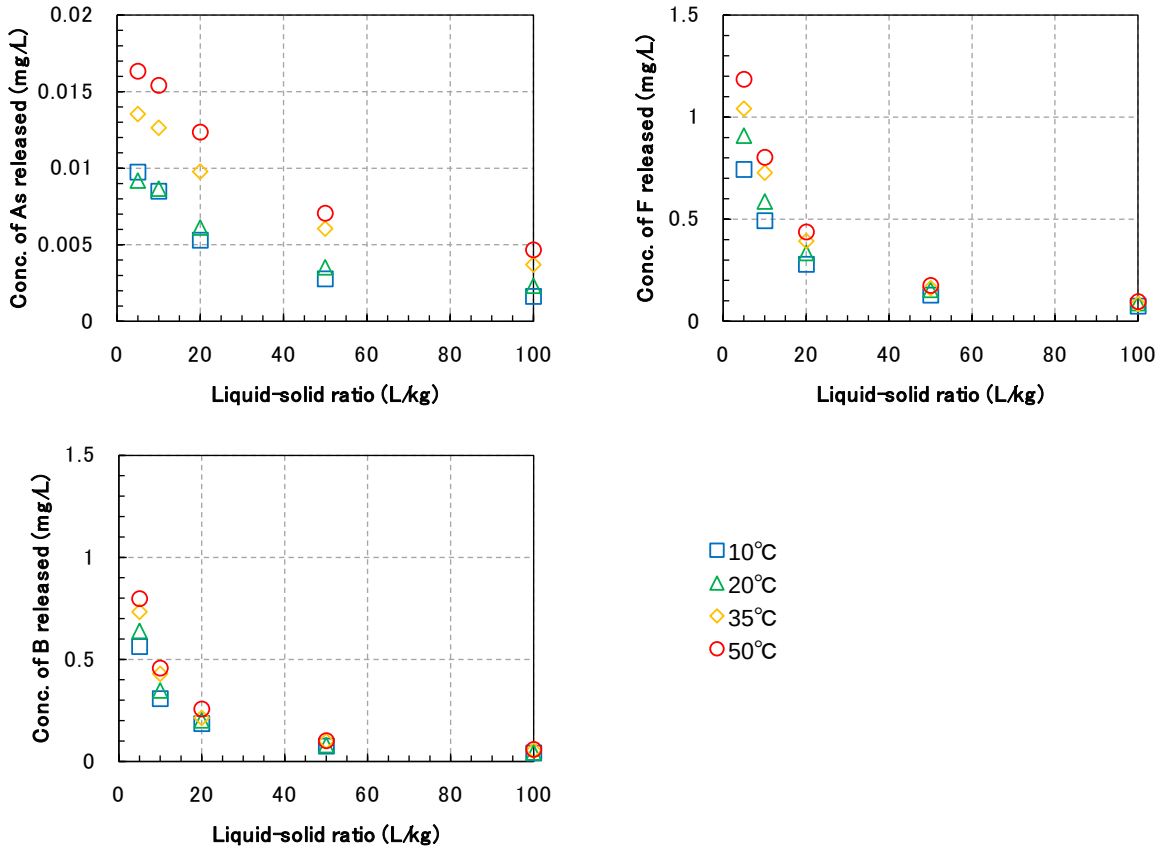


Figure 8. *Relationship between liquid-solid ratio and concentration*

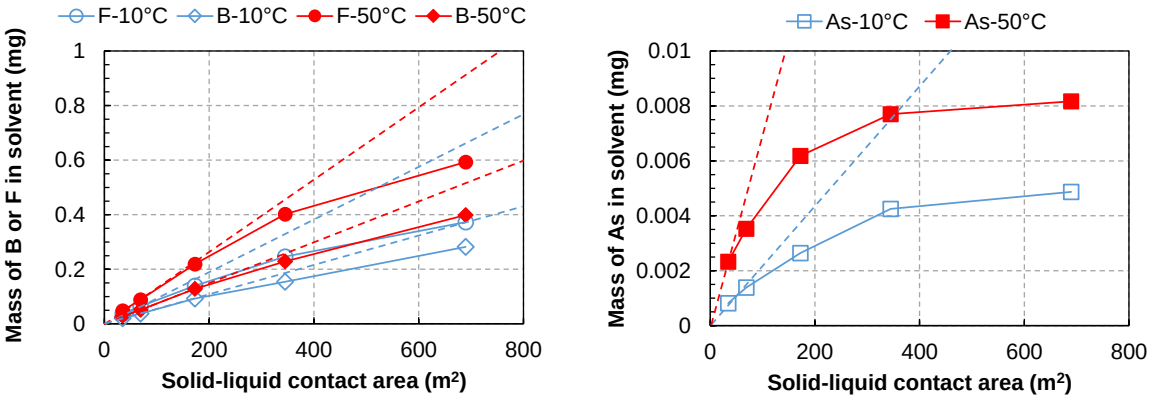


Figure 9. *Relationship between solid-liquid contact area and mass of substances in solvent*

were organized following the method of Sakanakura et al. (2021). When mass transfer from the interior of soil particles to the liquid phase is viewed microscopically, internal diffusion, boundary film diffusion, and interparticle diffusion vary with time. On the other hand, adsorption and desorption reactions are assumed to occur instantaneously between the substance adsorbed on the particle surface and the substance present in the nearby liquid phase. The amount of substance involved in adsorption/desorption per unit mass of solid (mg/kg), MT, is defined as the sum of substance in liquid phase per unit mass of solid (mg/kg), ML, and that adsorbed on the particle surface (mg/kg), q, by the following equation.

$$M_T = M_L + q \quad (6)$$

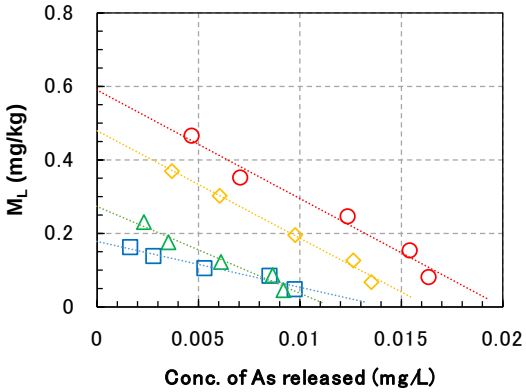
In adsorption/desorption reactions, the Henry-type equation (7) is a representative expression for the relationship between the liquid phase concentration c (mg/L) and the mass of adsorption q on the solid phase surface (mg/kg).

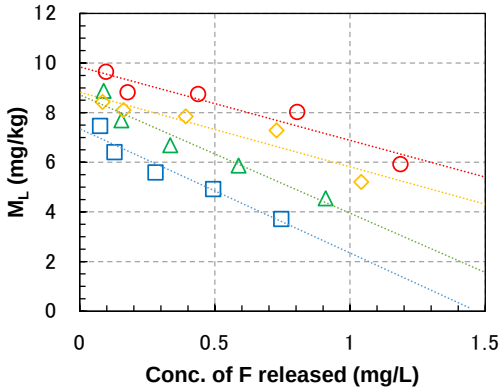
$$q = K_d c \quad (7)$$

Where K_d (L/kg) is the distribution coefficient. Substituting equation (7) into equation (6) yields equation (8).

$$M_L = -K_d c + M_T \quad (8)$$

That is, M_L is indicated by a straight line with slope $-K_d$ and intercept M_T. Figure 10 shows the relationship between liquid phase concentration and amount of substance in liquid phase per unit mass of solid. The experimental plots were approximated by a Henry-type equation, which showed good agreement with a coefficient of determination greater than 0.8 in all cases. The distribution coefficients and total amount of substance involved in adsorption/desorption per unit mass of solid obtained from the approximations are shown in Table 4. The distribution coefficients for arsenic and fluorine tended to increase at higher and lower temperatures, respectively, while no effect of temperature was observed for boron. Total amount of substance involved in adsorption/desorption per unit mass of solid was found to be greater at higher temperatures for all target substances. In this experiment, each substance was not supplied externally, so the increase in total amount of substance involved in adsorption/desorption per unit mass of solid means that the target substance in its easily soluble form near the surface layer of the soil particles has increased for some reason. This is presumably due to the fact that the solid phase diffusion coefficients of arsenic, fluorine, and boron increase with temperature, as shown in Figure 7. As the diffusion coefficient increases, the supply of substances from the interior of the solid layer to the surface layer is accelerated, and the effect of diffusion leaching extends deeper into the soil particles. This can be assumed to increase total amount of substance involved in adsorption/desorption per unit mass of solid. In this case, mass transfer from soil particles by internal diffusion takes a shorter period of time to converge under higher temperature conditions, but the initial concentration of leaching is higher. It is also possible that the substances present near the surface of the soil particles has changed to a more soluble form due to temperature changes. This experiment did not allow us to distinguish between the degree of influence of the increase in diffusion coefficient and the change in chemical form. Therefore, further experiments will be conducted to clarify this.





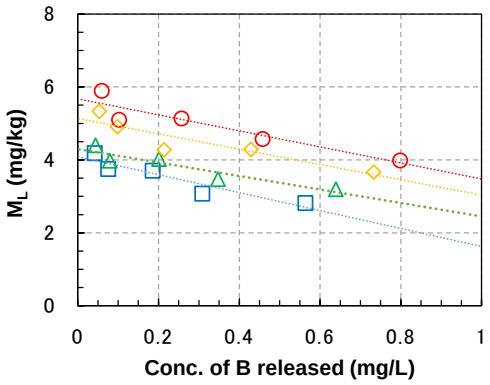


Figure 10. *Relationship between liquid phase concentration and amount present in the liquid*

Table 4-1. *K_d and M_T for As*

Temperature (°C)	Equation	R^2	K_d (L/kg)	M_T (mg/kg)
10	y = -12.5x + 0.178	0.9603	12.5	0.178
20	y = -23.6x + 0.273	0.9621	23.6	0.273
35	y = -29.3x + 0.480	0.9910	29.3	0.480
50	y = -29.5x + 0.590	0.9730	29.5	0.590

Table 4-2. *K_d and M_T for F*

Temperature (°C)	Equation	R^2	K_d (L/kg)	M_T (mg/kg)
10	y = -5.01x + 7.35	0.9430	5.01	7.35
20	y = -4.77x + 8.72	0.9413	4.77	8.72
35	y = -3.01x + 8.83	0.8832	3.01	8.83
50	y = -2.96x + 9.84	0.9018	2.96	9.84

Table 4-3. *K_d and M_T for B*

Temperature (°C)	Equation	R^2	K_d (L/kg)	M_T (mg/kg)
10	y = -2.46x + 4.09	0.8818	2.46	4.09
20	y = -1.85x + 4.30	0.8757	1.85	4.30
35	y = -2.10x + 5.14	0.8349	2.10	5.14
50	y = -2.20x + 5.68	0.8731	2.20	5.68

4 CONCLUSIONS

In this study, leaching tests were conducted on clays containing geogenic hazardous substances by varying temperature conditions and liquid-solid ratios. The results are summarized as follows.

(1) The diffusion coefficients of arsenic, fluorine, and boron increase with higher temperature conditions; at 10°C and 50°C, the diffusion coefficients were 20, 5, and 5 times higher, respectively.
(2) The amount of substance involved in adsorption/desorption of arsenic, fluorine, and boron increases with temperature. Comparing the temperature conditions of 10°C and 50°C, the amounts are 3.3, 1.3, and 1.4 times higher, respectively.
(3) Arsenic is more sensitive to temperature than fluorine and boron, its leaching behavior is easily changed by temperature conditions.

As described above, the leaching of hazardous substances contained in soil particles may be accelerated by temperature increase, and therefore, there is a risk involved in uncontrolled fluctuation of the sub-surface temperature. However, since it was confirmed that the effect is slight when the temperature change is small, it is important to control the temperature change appropriately for the

preservation of the surrounding environment. In order to utilize geothermal heat safely and to make effective use of ground containing geogenic hazardous substances, it is desirable to accumulate further knowledge on the effects of temperature on leaching behavior.

5 ACKNOWLEDGEMENTS

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