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Instructions for use

1    **Strontium adsorption and penetration in kaolinite at low $Sr^{2+}$ concentration**

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6    **Abstract**

7        Behavior of radioactive $Sr^{2+}$ in contaminated soils is an important issue in relation to nuclear

8    power plant accidents. The $Sr^{2+}$ adsorption on kaolinite and its migration in a kaolinite soil were

9    investigated because toxic effects of radioactive $Sr^{2+}$ have been found very severe for living

10    organisms at low $Sr^{2+}$ concentrations. Adsorption isotherms of $Sr^{2+}$ on kaolinite at different NaCl

11    concentration and pH were obtained by the batch method. The calculated distribution coefficients

12    ($K_D$) ranged between 600 and 40000 L $kg^{-1}$, which showed a strong preference for the adsorbed

13    phase. The results were used to evaluate the ratio (r) of penetration length of $Sr^{2+}$ relative to that of

14    water in a model kaolinite soil. When the $Sr^{2+}$ solution was percolated constantly into the kaolinite

15    soil, the penetration of $Sr^{2+}$ was delayed strongly at low $Sr^{2+}$ concentration due to adsorption. The

16    $Sr^{2+}$ penetration length was only 0.001 - 0.056 % of the water penetration length at pH 6.5 (0.1-10

17    mmol $L^{-1}$ NaCl). At pH 4.1 (1 mmol $L^{-1}$ NaCl) the effect was about 17 times less than at pH 6.5 (1

18    mmol $L^{-1}$ NaCl). Under all conditions, the $Sr^{2+}$ penetration increased when the $Sr^{2+}$ concentration

19    increased due to the $K_D$ decrease. The $Sr^{2+}$ isotherms could be fitted well to the Langmuir

20    adsorption equation, which indicates that only one site type is involved in the $Sr^{2+}$ adsorption.

21    **Key Words:** Adsorption, distribution coefficient, kaolinite, penetration length ratio, strontium

22    **1. Introduction**

23    Strontium (Sr)-90 is one product of the radioactive elements of nuclear fuel materials emitting

24    beta particles. Its half-life is 29 years, and it is harmful to the human health (Fetter *et al.* 1988).

25    During the nuclear power plant accidents in Fukushima, Japan and Chernobyl, Ukraine, large

26    amounts of radioactive species, including Sr-90, were released into the environment (Bondarkov

27    *et al.* 2011; Steinhauser et al. 2013). As the radioactive damage is serious, the environmental fate

28    of $Sr^{2+}$ must be considered, especially at low $Sr^{2+}$ concentrations.

29        Kaolinite is one of the most ubiquitous phyllosilicates in soils and it has next to the basal

30    plane surface a relatively large edge surface (White and Dixon 2002); therefore, $Sr^{2+}$ adsorption

31    and transport in kaolinite soils deserve specific attention. Some reports consider $Sr^{2+}$ adsorption

32    on kaolinite on the basis of ion exchange (Wahlberg *et al*. 1965; Parkman et al. 1998; Bascetin and

33    Atun 2006; Keçeli 2015). $Sr^{2+}$ transport in quartz sand (Rod *et al*. 2010), sediment (Wallace *et al.*

34    2012) and porous material (Prigiobbe *et al*. 2012) were reported. The fact that the distribution

35    coefficient provides information on both adsorption and migration of a component, which is the

36    simplest parameter to characterize the influence of environmental factors on the fate of a pollutant.

37    Thus, the distribution coefficient has been used to characterize Sr-kaolinite systems (Meyer 1979;

38    Rafferty et al. 1981; Erten et al. 1988; Bunde et al. 1997; Bascetin and Atun 2006; Rani and

39    Sasidhar 2012). The distribution coefficient of the Sr - kaolinite system under the given conditions

40    can be determined when the adsorption isotherm of $Sr^{2+}$ on kaolinite is known. Migration of $Sr^{2+}$

41    in kaolinite soil can be conveniently characterized by using the ratio of average penetration length

42    of $Sr^{2+}$ relative to that of water. This ratio is determined by the distribution coefficient and the

43    solid and solution content in the kaolinite soil (Bolt 1978). The migration of $Sr^{2+}$ is retarded

44    because of the adsorption of $Sr^{2+}$ to kaolinite soil. This retardation can be quantified by using the

45    parameter of distribution coefficient. However, there has been little research of $Sr^{2+}$ penetration

46    length at low $Sr^{2+}$ concentration compared with water penetration length.

47        The objectives of the present study are therefore to investigate both the characteristics of $Sr^{2+}$

48    adsorption on kaolinite at different NaCl concentration and pH values with emphasis to low $Sr^{2+}$

49    concentrations and the migration of $Sr^{2+}$ in a kaolinite soil under these conditions. The $Sr^{2+}$

50    distribution coefficients were derived and used to describe the $Sr^{2+}$ migration. To gain further

51    insight in the mechanism of the adsorption, the $Sr^{2+}$ adsorption results were analyzed with the

52    Langmuir model. The Langmuir model assumes ideal adsorption behavior in a monolayer and

53    considers the adsorption capacity as independent parameter and neglects valence effects

54    (Langmuir 1918; Rani and Sasidhar 2012).

55    **2. Materials and Experiments**

56        The kaolinite from Iriki, Kagoshima, was suspended in deionized water and the fraction with a

57    diameter less than 2μm was obtained by centrifugation. The obtained fraction was freeze-dried

58    and stored in a closed container. The sample was characterized by using the X-ray diffraction

59    apparatus (RINT1200, Rigaku Group of companies, Japan); the kaolinite content was 98.1% and

60    the alunite content was 1.9%.

61        Three g of kaolinite powder was put in a 400 mL centrifuge bottle, mixed well with 300 mL

62  of 1 mol L$^{-1}$ NaCl solution and repeated for three times to reach a complete exchange. The clay

63  was equilibrated with the prescribed NaCl solution at 0.1, 1 or 10 mmol L$^{-1}$. This process was

64  repeated until the electrical conductance of the suspension equaled that of the prescribed NaCl

65  solution at 0.1, 1 or 10 mmol L$^{-1}$. A suitable volume of 0.01 mol L$^{-1}$ HCl or NaOH solution was

66  added to adjust the pH of the Na-Kaolinite suspension to 6.5 or 4.1. The final 10 g L$^{-1}$

67  Na-kaolinite suspension was kept for further use.

68      The Sr$^{2+}$ adsorption experiments were conducted by the batch method using a series of

69  polypropylene centrifuge tubes. Ten mL of the 10 g L$^{-1}$ Na-kaolinite suspension was pipetted into

70  the centrifuge tube. Subsequently, 20 mL of SrCl$_2$ solution containing the prescribed NaCl

71  concentration (0.1, 1 or 10 mmol L$^{-1}$) at prescribed pH (4.1 or 6.5) was added to reach an initial

72  Sr$^{2+}$ concentration in the range $2 \times 10^{-3}$ mmol L$^{-1}$ to $6 \times 10^{-2}$ mmol L$^{-1}$. The tubes were shaken for 24

73  hours at room temperature. The pH of the equilibrium suspension was measured and recorded.

74  Triplicate runs were performed for all batch experiments.

75      The equilibrium suspensions were centrifuged and the supernatants were gathered to

76  determine the Sr$^{2+}$ concentrations by inductively coupled plasma mass spectrometry (ICP-MS:

77  PerkinElmer SCIEX-ELAN DRC-e). The adsorbed amount of Sr$^{2+}$ on the Na-kaolinite was

78  calculated by subtracting the amount of Sr$^{2+}$ in equilibrium solution from the total amount of Sr$^{2+}$

79  added initially.

80  **3. Modeling**

81  **3.1. Distribution coefficient**

82      The distribution coefficient, $K_D$ (L kg$^{-1}$), describes the distribution of the adsorbate over the

83  solid phase and the solution phase that can be defined as

84  $$K_D = \frac{Q_{Sr}}{[Sr]} \qquad (1)$$

85  where $Q_{Sr}$ is the adsorbed amount of Sr$^{2+}$ on kaolinite (mmol kg$^{-1}$) and [Sr] is the equilibrium Sr$^{2+}$

86  concentration in the solution (mmol L$^{-1}$). The larger $K_D$ is, the stronger is the accumulation in the

87  adsorbed phase.

88  **3.2. Retardation and penetration length ratio**

89  Let a certain volume of solution containing a concentration [Sr] mmol L$^{-1}$ of cation Sr$^{2+}$ at the

90  prescribed NaCl concentration and pH feed to per unit area of kaolinite. The distribution ratio, $R_D$,

91  is a commonly used physical parameter for estimating the retardation of a dissolved pollutant in

92    the case of a solution with pollutant that permeates in a column of soil. Because the retardation is

93    caused by adsorption, $R_D$ is related to the partition of the contaminant between the solution and the

94    solid (adsorbed) phase. As $K_D$ quantifies the partition, $K_D$ and $R_D$ are directly related:

95    $$R_D = K_D \frac{\rho_b}{\theta} \qquad (2)$$

96    where, $\rho_b$ (kg L$^{-1}$) the bulk density of the kaolinite soil (mass of kaolinite in the column per unit

97    volume of kaolinite and void) and $\theta$ the volumetric water content of the column of soil per unit

98    soil volume in saturated condition (L L$^{-1}$ or m$^3$ m$^{-3}$). For the calculations, the soil bulk density of

99    the hypothetical kaolinite soil has to be assumed to be homogeneous and constant. As the bulk

100   density of natural soil usually ranges from 1.0 to 1.5 kg L$^{-1}$, a kaolinite soil bulk density of 1.25 kg

101   L$^{-1}$ is used in this study. From this value, and the density of pure kaolinite of 2.65 kg L$^{-1}$ (Kuroda

102   *et al.* 2003), the volume fraction of kaolinite in the soil can be obtained: 1.25/2.65 = 0.472.

103   Consequently, the volume fraction of solution in the soil equals 0.528 when the kaolinite soil is

104   saturated and this implies that under these conditions $\theta$ has reached its maximum value of 0.528.

105   When only the saturation condition is adopted, $R_D$ is proportional to $K_D$ because the $\theta$ and $\rho_b$ are

106   constant.

107       In the saturated column, the average penetration length, $x_p$, of Sr$^{2+}$ fed into the soil by the Sr$^{2+}$

108   solution is defined by the relation (Bolt 1978):

109   $$x_p = \frac{V_F}{\theta} \frac{1}{1+R_D} \qquad (3)$$

110   where $V_F$ is the feed volume of Sr$^{2+}$ solution per m$^2$ of soil (m$^3$/m$^2$) and $\theta$ is the saturated water

111   content (m$^3$/m$^3$). $V_F/\theta$ is the average penetration length (m) of water in the soil. The ratio, $r$, of the

112   average penetration length of Sr$^{2+}$ to that of water is thus obtained as:

113   $$r = \frac{x_p}{V_F/\theta} = \frac{1}{1+R_D} \qquad (4)$$

114   The inverse of $r$, 1+$R_D$, is the retardation factor which is widely used for analyses of solute

115   transport in soils.

116       As $K_D$ is a positive quantity, $R_D$ is positive and this implies that $r < 1$ and a small $r$ implies a

117   strong Sr$^{2+}$ retardation. For large $R_D$ ($R_D > 100$), i.e., large $K_D$, $r$ is practically speaking inversely

118   proportional to $R_D$. We can also assume that $\theta$ is a constant unsaturated volumetric water content.

119   However, in order to obtain the largest penetration length ratio, the saturated condition is adopted

120   here.

121   **3.3. Langmuir model**

122   The Langmuir model was used for the adsorption of Sr$^{2+}$ on kaolinite as follows:

123
$$Q_{Sr} = \frac{Q_{Sr,m}K_L[Sr]}{1+K_L[Sr]} \quad (5)$$

124     where $Q_{Sr}$ (mmol kg$^{-1}$) is the Sr$^{2+}$ adsorption, $Q_{Sr,m}$ (mmol kg$^{-1}$) is the maximum adsorption, $K_L$ the

125     Langmuir adsorption constant (L mmol$^{-1}$) and [Sr] (mmol L$^{-1}$) is the equilibrium Sr$^{2+}$

126     concentration. As the Langmuir model is the theoretically derived adsorption model, we use it for

127     the theoretical consideration.

128     **4. Results and discussion**

129     **4.1. The Sr$^{2+}$ distribution coefficient**

130     The adsorption isotherms of Sr$^{2+}$on Na-kaolinite at pH 6.5 and pH 4.1 and different NaCl

131     concentrations are depicted in Fig. 1 as a double logarithmic plot. The measured adsorption

132     increases linearly with slope $\approx$ 1 at low Sr$^{2+}$ concentrations, but gradually levels off at higher

133     Sr$^{2+}$concentrations. Slope $\approx$ 1 implies a constant adsorption affinity and $K_D$ value; the low

134     concentration ranges for which the slope $\approx$1 is the so-called 'Henry region'; in this range the

135     adsorption is directly proportional to the solution concentration, which can be well explained by

136     Langmuir model as written in the later part. The dotted lines (slope =1) are lines with different $K_D$

137     (L kg$^{-1}$) as indicated (see Eq.(1)). The constant values of $K_D$ at low Sr$^{2+}$ concentration are observed

138     for each isotherm in Fig. 1. They ranged between 600 and 40000 L kg$^{-1}$. The magnitude of $K_D$

139     clearly depends on the solution conditions: $K_D$ strongly decreases with increasing salt

140     concentration and decreasing pH. The $K_D$ value at low Sr$^{2+}$ concentration at 0.1 mmol L$^{-1}$ NaCl is

141     about 53 times larger than that at 10 mmol L$^{-1}$ NaCl. The effect of pH is only investigated at 1

142     mmol L$^{-1}$ NaCl. $K_D$ at pH 6.5 is about 17 times larger than $K_D$ at pH 4.1. Similar effects for the

143     kaolinite – Sr$^{2+}$ system have been observed by a few researchers (Meyer 1979; Rani and Sasidhar

144     2012). For all solution conditions $K_D$ decreases with increasing Sr$^{2+}$ concentrations at Sr$^{2+}$

145     concentrations beyond the Henry region, but the Sr$^{2+}$ concentration where $K_D$ starts to decrease

146     depends on the solution conditions. Erten *et al.* (1988) and Bascetin and Atun (2006) also

147     observed a decrease of $K_D$ with the increasing Sr$^{2+}$ concentration for kaolinite. Concluding this

148     section, it is clear that pH and Na$^+$ concentration influence the adsorption of Sr$^{2+}$ on kaolinite and

149     the Sr$^{2+}$ distribution coefficient strongly.

150       The fittings of the Sr$^{2+}$ adsorption data to the Langmuir equation are presented in Fig.1 as

151     double logarithmic plot (solid lines). The measured values of Sr$^{2+}$ adsorbed on kaolinite are quite

152     well fitted to the Langmuir model. The adsorption affinity or Langmuir constant, $K_L$, and the

153     adsorption capacity, $Q_{Sr,m}$, are collected in Table 1. The values of $K_L$ are strongly influenced by

154 NaCl concentration because the influence of competition between $Sr^{2+}$ and $Na^+$ on $K_L$ differs with

155 different $Na^+$ concentration. Thus, $K_L$ obtained in this study should be interpreted as a *conditional*

156 *constant*; $Q_{Sr,m}$ are almost identical under different NaCl concentration.
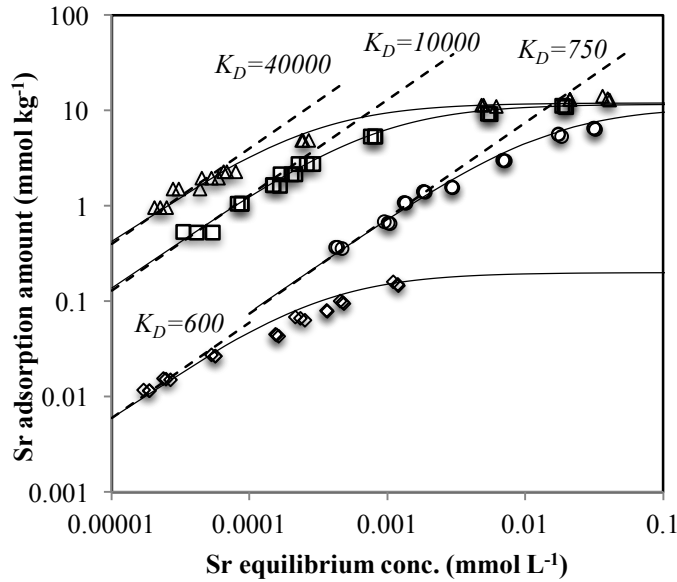


157

158 **Fig. 1** $Sr^{2+}$ adsorption isotherms on kaolinite at two pH values and three salt concentrations. The dotted

159 lines are lines of equal distribution coefficient ($K_D$) and the solid lines are fitting curves of Langmuir

160 model. The symbols are measured values. At pH 6.5: △0.1 mmol $L^{-1}$ NaCl, □1 mmol $L^{-1}$ NaCl, ○

161 10 mmol $L^{-1}$ NaCl. At pH 4.1: ◇1 mmol $L^{-1}$ NaCl

162 **Table 1** Langmuir isotherm parameters

| pH | Na conc. | $Q_{Sr,m}$ | $K_L$ | $R^2$ |
|----|----------|------------|-------|-------|
|     | 10       | 11.0       | 60    | 0.990 |
| 6.5 | 1        | 11.6       | 1100  | 0.995 |
|     | 0.1      | 12.0       | 3300  | 0.987 |
| 4.1 | 1        | 0.2        | 2500  | 0.934 |

163      By comparing $K_D$ and $K_L$ at given solution conditions, it should be noted that $K_L$ is a constant

164 for all $Sr^{2+}$ concentrations, but that $K_D$ is only constant in the Henry region. The difference is due

165 to the fact that Eq.(5) takes into account the adsorption capacity, $Q_{Sr,m}$. At relatively high $Sr^{2+}$

166 concentration the probability of adsorption is decreased by the decrease of the vacant sites for $Sr^{2+}$

167 adsorption. Eq.(5) takes this decreased probability into account, while Eq.(1) does not take the

168 adsorption capacity into account. At sufficiently low $Sr^{2+}$ concentration ($K_L[Sr]<<1$) Eq.(5)

169 reduces to the Henry equation, $Q_{Sr} = Q_{Sr,m}K_L[Sr]$, and this relation equals Eq.(1) with $Q_{Sr,m}K_L = K_D$,

170    indicating that in the Henry region $K_L$ and $K_D$ are closely related.

171        The Sr$^{2+}$ isotherms could be fitted well to the Langmuir adsorption equation, which indicates

172    that only one site type is involved in the Sr$^{2+}$ adsorption. The values of $K_D$ keeping constant at low

173    Sr$^{2+}$ concentration were observed. Therefore, even if the Sr$^{2+}$ concentration is extremely lower

174    than those in this experiment, the values of $K_D$ in the henry region can be valid from the

175    theoretical viewpoint. The value can be applicable for the Sr-90 contaminated sites where the Sr$^{2+}$

176    concentration is supposed to be very low.

177    **4.2. The Sr$^{2+}$ penetration length ratio**

178    The average penetration length ratio $r$, representing the average penetration length of Sr$^{2+}$ relative

179    to that of water is depicted in Fig. 2 as a function of the equilibrium Sr$^{2+}$ concentration of the

180    solution fed in the kaolinite soil. The four depicted data sets correspond with the four-adsorption

181    isotherms depicted in Fig. 1. Since the ratio $r$ is inversely proportional to $(1+R_D)$ and $R_D$ is

182    proportional to $K_D$, the large $K_D$ values corresponding with the isotherms at low Sr$^{2+}$ concentration

183    induce small $r$ values. As $K_D$ increased with decreasing NaCl concentration and increasing pH, the

184    reverse trend should be observed for $r$ and this explains the decrease of $r$ with decreasing NaCl

185    concentration and increasing pH observed in Fig. 2. The small values of $r$ indicate considerable

186    retardation of Sr$^{2+}$ upon solution penetration in the kaolinite soil. The increase of the penetration

187    length ratio of Sr$^{2+}$ in kaolinite with increasing Sr$^{2+}$ concentration is due to the decrease of $K_D$. By

188    considering the above trends quantitatively, the following observations can be made. The

189    dependence of the penetration length ratio of Sr$^{2+}$ in kaolinite on pH is considerable: at 1 mmol L$^{-1}$

190    NaCl and low Sr$^{2+}$ concentration, the $r$ value at pH 4.1 ($7.04 \times 10^{-4}$) is 16.7 times larger than $r$ at pH

191    6.5($4.22 \times 10^{-5}$). It is favorable for Sr$^{2+}$ adsorption at high pH, which results in a strong retardation

192    of Sr$^{2+}$ in sediments (Wallace *et al.* 2012) and iron oxides (Prigiobbe *et al.* 2012). The effect of the

193    NaCl concentration at pH 6.5 and low Sr$^{2+}$ concentration on the penetration length of Sr$^{2+}$ is also

194    large: at 0.1 mmol L$^{-1}$ NaCl r equals $1 \times 10^{-5}$ which is 53.3 times smaller than $r$ at 10 mmol L$^{-1}$

195    NaCl ($5.63 \times 10^{-4}$). The trend of the present results corresponds well with literature results. Wallace

196    *et al.* (2012) have reported that for sediments the presence of Na$^+$ resulted in the much lower

197    sorption of Sr$^{2+}$ which caused a significant migration of Sr$^{2+}$ in the sediments. Hull *et al.* (2008)

198    have reported that Sr$^{2+}$ transported rapidly in the vadose zone sediment at high Na$^+$ concentration

199    and the Sr$^{2+}$ mobility decreased significantly in the absence of competing cations.

200
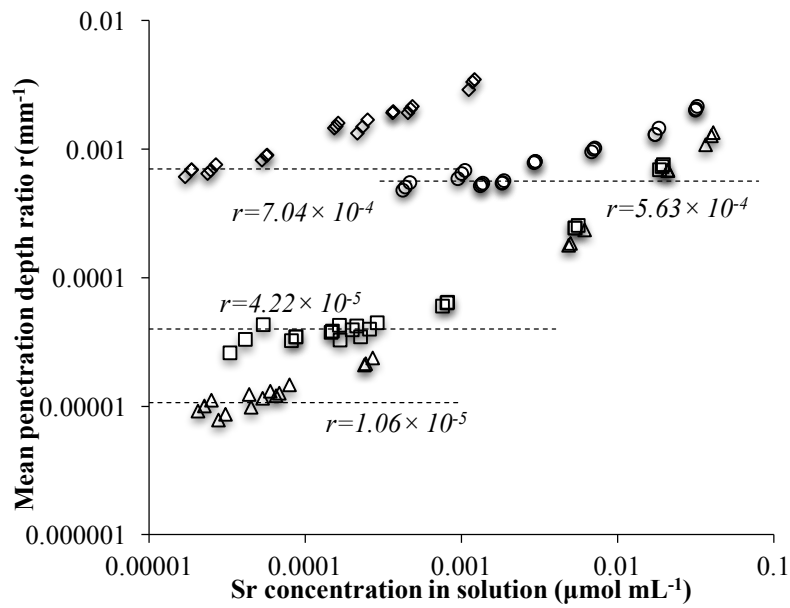
**Fig.2** The penetration length ratio of $Sr^{2+}$ versus the fed solution concentration $Sr^{2+}$. The dotted lines
and indicated r values are the penetration ratios corresponding with the largest $K_D$ values at low $Sr^{2+}$
concentration for each solution condition. At pH 6.5: △0.1 mmol $L^{-1}$ NaCl, □1 mmol $L^{-1}$ NaCl, ○10
mmol $L^{-1}$ NaCl. At pH 4.1: ◇1 mmol $L^{-1}$ NaCl

The evaluated penetration length ratios of the $Sr^{2+}$ at low [Sr] are very small. The $Sr^{2+}$
penetration length is about 0.001% of the water penetration length at the conditions of pH 6.5, 0.1
mmol $L^{-1}$ NaCl and [Sr] < 5x10^{-8} mmol $L^{-1}$. At pH 6.5, 1 mmol $L^{-1}$ NaCl and [Sr]<2x10^{-7} mmol
$L^{-1}$, the value is about 0.004%. At pH 6.5, 10 mmol $L^{-1}$ NaCl and [Sr]<2x10^{-6} mmol $L^{-1}$, the value
is 0.056 %. At pH 4.1, 1 mmol $L^{-1}$ NaCl and [Sr] < 2x10^{-8} mmol $L^{-1}$, the value is about 0.07 %.
These values clearly indicate that $Sr^{2+}$ is most strongly retarded by kaolinite at low NaCl
concentration and relatively high pH.

The values of extremely low penetration length ratio of $Sr^{2+}$ relative to that of water in
kaolinite were calculated theoretically based on the distribution coefficient obtained from
adsorption experiment. Although there are many complicated factors affecting the migration of
$Sr^{2+}$ in soils such as preferential flow, the average penetration length in the soils with uniform soil
structure can be predicted.

In conclusion, because only one site type is involved in the $Sr^{2+}$ adsorption in the kaolinite,
the constant values of $K_D$ are valid to calculate the average penetration length of $Sr^{2+}$ even at
extremely low $Sr^{2+}$ concentration theoretically. The low values of average penetration length ratio
show the strong retardation in kaolinite soils for $Sr^{2+}$ at low $Sr^{2+}$ concentration. Although the
penetration ratio is a simple and useful parameter to estimate the mobility of $Sr^{2+}$, direct

222    observations of $Sr^{2+}$ migration in soil are also necessary to confirm the theoretically calculated

223    retardation.

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