Breakthrough Curves and Elution Patterns of Heavy Metals in Sandy Clay Loam and Clay Soils.

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We investigated the mobilization of Cd, Pb, and Cr in two different soils in response to sorption capacities and competition for available sorption site while they moved under saturated water conditions. Two soil samples that were clay and sandy clay loam were collected within 20 cm from the upland surface. To do this, we used three different systems of heavy metal combinations such as single, binary, and ternary as solution phase. And then we observed the breakthrough curve (BTC) and elution as a function of pore volume by applying heavy metal solution and displacing K solution until these curves reached to maximum and minimum. The results showed that BTC and elution curves were not symmetric and it required more pore volumes with increasing species of heavy metals in solution phase, as well as longer tailings. Compared the areas over and under BTC and elution curve, relatively small amount of heavy metal was displaced by K even though there were differences in electronegativity among heavy metals. Conclusively, we assumed that heavy metals transport in soil could be influenced by soil physical nonequilibrium and chemical equilibrium in solution as far as there were more than two species of heavy metals existed.

Introduction

Soil has an important role in heavy metal accumulation, retarding the migration processes, which influences further ground- and surface water contamination processes. In most cases, several contaminants are simultaneously released into the environment. A systematic comparison between mobile pools of heavy metals determined by single or sequential batch extractions and the amounts of metals released in column leaching experiments is currently lacking (Andreas et al., 2003). Therefore, it is of crucial importance to study in detail the mutual influence and retention processes of the various heavy metals simultaneously present in soils.

Compounds can be leached out of one region and enter into the adjacent region in soil only through the combination of percolation and dissolution of various water-soluble compounds. Also the leaching process is limited by the rate of chemical reactions under chemical nonequilibrium conditions. The amount of heavy metal existed in soil system can be governed by the soil physical heterogeneity and the water input pattern

dependent on the flux (Nkedi-Kizza et al., 1983, Brusseau and Rao, 1990, Sun et al., 2001). Water flow and convective transport between the regions, driven by the hydraulic gradient, strive to reestablish hydraulic and chemical equilibrium for ions in soils (Reedy et al., 1996; Hutson and Wagenet, 1995).

The chemical behavior of heavy metals in soils is controlled by a number of processes, including metal cation release from contamination source materials, cation exchange and specific adsorption onto surfaces of minerals and soil organic matter, and precipitation of secondary minerals (Manceau et al., 2000; McBride et al., 1997; McBride, 1999; Morin et al., 1999). And the movement of heavy metals through soil is strongly affected by the processes of adsorption and exchange. These mechanisms depend on soil properties such as the cation exchange capacity (CEC), and exchange selectivity coefficients for the various cations and heavy-metal cations in the soil system.

Sorption phenomena significantly affect the transport and ultimate fate of heavy metals in soil and subsurface system, while the rate at which adsorption of heavy metal becomes significant varies with the particular metals and the solid involved, the solid vs. solution ratio, the specific

surface area of the solid, the total metal concentration, and the concentration of interacting species. The reversibility of adsorption under different given conditions warrants serious scrutiny as the fate of adsorbed ions seem to depend heavily on this process. Also, multicomponent transport processes can play an important role in heavy metal migration in soils.

Column leaching experiments provide information about heavy metal release and transport in soil, and are also useful for testing possible soil remediation or stabilization treatments, to assess heavy metal binding and desorption kinetics, or to study processes such as colloid-facilitated metal transport at the laboratory scale (Grolimund et al., 1996; Kedziorek et al., 1998; Kretzschmar and Sticher, 1997; Temminghoff et al., 1997).

Therefore, our objectives were to (i) investigate the mobilization of different contaminations of Cd, Pb, and Cr from two different soils in response to sorption capacities and competition for available sorption site while they move under saturated water flow, and (ii) compare the amounts of heavy metals eluted from column leaching experiments. Column effluent concentration patterns were discussed in the light of competitive adsorption characteristics among the heavy metals investigated in this experiment.

Material and Methods

Soil Sampling and Physico-chemical properties Soil samples were collected within 20 cm from the surface of upland located in Nonsan, Chungnam and Yoosung, Daejeon. The upland soil in Nonsan was reclaimed from a moderate highland two years ago while the soil in Yoosung has been cultivated to grow vegetables more than ten years. These soils were air-dried and ground to pass 2 mm sieve using Wiley Mill (AARON Model 3370, USA). The chemical and physical properties were analyzed by Soil analysis method (NIAST, 2000). The analytical methods were as follows: Soil texture analysis by hydrometer method, soil pH (Soil : Distilled water=1:5), EC_e (saturated water extract), CEC (1N ammonium acetate), OM (Walkley-Black method). For water-soluble cations and heavy metals in soil samples, 150 g of soil packed into acryl column were washed with 750 mls of distilled water twice and the leachate was collected for analysis of cations and heavy metals. The soils washed with the

distilled water were oven-dried for 48 hours at $105℃$. And 100 g of the oven-dried soil samples were collected to be used for exchangeable cations and heavy metals. These soils were extracted with 500 ml of 0.1 M HCl twice. The extract was analyzed with Atomic Absorption Spectrophotometer (AAS, Shimazu, AA-680A, Japan).

Soil column experiment for BTC and elution The BTCs and the corresponding elution patterns for three heavy metals were measured in water saturated, isotropic homogeneous soil columns. The acryl column was 15 cm in length and 6.4 cm in diameter. A stainless screen filter attached with 200 nylon mesh was used as bed support on the bottom of the column. The columns were packed with prepared dry soils into 5.0 cm from the bottom of the column under saturated water conditions in a water bath by successive addition of soil to establish uniform bulk density and water content. The pore volumes (PV) were calculated as the product of the column volumes and $θ_v$ at 84.93 and 82.76 cm^3 for sandy clay loam and clay soil, respectively. The measured saturated hydraulic conductivities (K_{sat}) were of 50.11 and 40.08 cm d^{-1} for sandy clay loam and clay soil, respectively.

The treatments were single, binary, and ternary systems with different combinations of each heavy metals. The treated concentrations of the individual heavy metal which was prepared with Cd^{2+} as $CdCl_2$, Cr^{3+} as $CrCl_3$, Pb^{2+} as $PbCl_2$ were 10 mmol L^{-1}. These solution were added to the top of soil column as of pore volume basis until the relative concentration reached to 1 after analysis of heavy metals collected as effluent from the bottom of the soil column. At regular time intervals, aliquot samples of the leachate were collected, weighed, and stored at $4℃$ until chemical analysis. After the relative concentration reached to 1, the solution was immediately switched to 30 mmol L^{-1} of KCl until the relative concentration of the heavy metals reached to minimum during investigation period. The relative concentration was calculated by C_i/C_0, where C_i was concentration in the effluent and C_0 was initial concentration of each heavy metal. The concentration of the heavy metals in the effluent were measured by Atomic Absorption Spectrophotometer.

Results and Discussion The physical and chemical properties of soils used in this experiment were as follows: soil textures were sandy clay loam (pH 4.5) and clay (pH 5.5), while CEC of sandy clay loam was 4.5 cmol kg^{-1} which was higher by about 1.0 cmol$_c$ kg^{-1} than

that of clay soils although clay soil had a little bit higher organic matter (Table 1). The amounts of exchangeable cations extracted by 0.1 M HCl were higher than those extracted by distilled water for both soils. Among the heavy metals the concentration of Cr was only recovered in extract (Table 2). The sums of soluble and exchangeable heavy metals were 5.37 and 13.04 mmol l^{-1} which were equivalent to about 11 % and 30 % of CEC for sandy clay loam and clay soils.

Carefully interpreted column leaching experiments can provide valuable information on the coupling of simultaneous processes such as cation exchange and adsorption /desorption, as well as on the importance of slow reaction kinetics in metal leaching (Andreas et al., 2003). They also suggested that the lability of metals in soil, and the kinetics of metal desorption/dissolution were the main factors controlling the behaviour of metal leaching in soil.

Fig. 1 showed the curves of BTC and elution measured with single species of Cr, Cd, and Pb in sandy clay loam and clay soil columns. These curves were plotted as relative concentration of the heavy metal in the effluent (C_i) and influent (C_o) as a function of pore volume. The curves of BTC and elution were not symmetric. This indicated that adsorption mechanism and exchange by K were not same. A comparatively retarded BTC behavior was observed in the order of Cr, Pb, and Cd on both soil columns. The heavy metals entered into soil column could be adsorbed onto the available sorption sites on soil particle surface and could exchange with other cations already adsorbed on the particle surfaces.

The adsorption could be influenced by concentration at

**Table 1. Physico-chemical properties of the soils used.**

Soil Sample	pH (1:5)	ECe (dS m^{-1})	CEC (cmol$_c$ kg^{-1})	OM (%)	PSD[†] (%)			Soil Texture
					Sand	Silt	Clay	
Nonsan	4.6	0.03	4.5	ND[†]	54	20	26	SCL
Yoosung	5.5	0.04	3.5	1.1	25	35	40	C

[†] Particle Size Distribution

[†] Not Detected.

Table 2. Water-soluble and exchangeable cation of the soils used.

Soil Sample		Cation				Heavy metal		
		(cmol kg^{-1})						
		K	Na	Ca	Mg	Cr	Cd	Pb
Nonsan	Soluble	0.011	0.012	0.010	0.005	0.011	ND	ND
	Exchangeable	0.035	0.021	0.293	0.133	0.006	ND	ND
Yoosung	Soluble	0.011	0.019	0.015	0.013	0.011	ND	ND
	Exchangeable	0.035	0.028	0.832	0.336	0.003	ND	ND
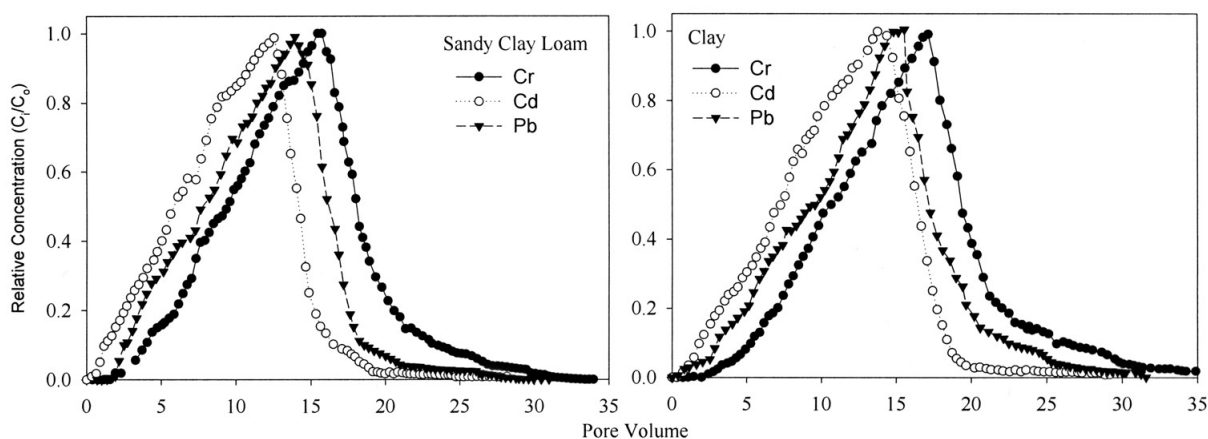
Fig. 1. BTCs and elution of single species of Cr, Cd, and Pb in sandy clay loam and clay column as a function of pore volume. The elution curves were obtained by switching to 30 mmol KCl after the relative concentration of BTC reached to 1.

equilibrium as observed from adsorption isotherm. Also, the amount of heavy metal in solution could decrease with increasing travel distance in soils. Therefore, the available sorption site in the specific region decreased with increasing number of pore volumes passed through the region whereas the amount of heavy metal in solution increased with increasing pore volumes along soil depth. These phenomena could initially occur at the top of the soil column and proceed to the adjacent lower soil region as the reactive solute moved with water. Therefore, adsorption rate among the heavy metals in soil system influenced the shape of the BTC curve. The higher affinity of the heavy metal was observed as the increment of the BTC curve was smaller. From these BTCs by single species of heavy metal we could conclude that the orders of affinity were same regardless of soil texture.

To compare the removal of the heavy metals from solution flowing through the soil columns, we calculated the removal ratio from the BTC curves by multiplying the relative concentration by each pore volume until the BTC curve reached to 1 (Table 3). The areas above and below the BTC curve in Fig. 1 meant the heavy metals adsorbed from solution and recovered in the effluent, respectively. The results showed that the total area for amounts of adsorption were slightly higher in clay than those in sandy clay loam soil. This results were similar to the results from the adsorption isotherms for these heavy metals with the same soils observed by Noh (2005).

According to McBride (1994), if the ability of the metals to chemisorb were based on only electrostatics, the strongest bond should be formed by the metal with the greatest charge-to-radius ratio, which would produce a different order for the same metals, that is, Ni > Cu > Co > Zn > Cd > Pb. Also electronegativity is an important factor in determining which of the trace metals chemisorb with the highest preference and, on this basis, the predicted order of bonding preference would be Cu >

Ni > Co > Pb > Cd > Zn. However, the two adsorption sequences most found were Cr > Pb > Cu >Cd > Zn > Ni and Pb > Cr > Cu > Cd > Ni > Zn. These sequences did not exactly follow the order of the electronegativity of the metal cations, which is, according to Evans (1966), Cu (1.9), Pb (1.8), Ni (1.8), Cd (1.7), Cr (1.6), and Zn (1.6). The presence of Cr as one of the most retained cations, in spite of its lower electronegativity value, seems to be related to the fact that this metal was applied in its trivalent form, which is how it appears, predominantly, in soils (Smith and McGrath, 1990). Therefore, the BTC curves of the heavy metal in soil columns were mainly influenced by electronegativity.

Initially, there was no heavy metals detected in the effluent because the soil columns were saturated with water (C = 0, x ≥ 0, t =0) and these heavy metals were reactive with the soil particles, resulting in adsorption which could be described as Eq. 2 below where the boundary conditions used in this experiment were $C = C_o$ and x = o at the inlet boundary.

$$\frac{\partial C}{\partial x} = 0 \ , \ x \rightarrow \infty \qquad t > 0 \qquad \text{[Eq. 1]}$$

where C is concentration of solute, x is specific depth, and t is time.

The governing mathematical equation obtained by curve fit could be described as sigmoidal type for all three heavy metals of the BTC curves [Eq. 2].

$$Y = Y_o + \frac{a}{1+e^{(-(x-x_o)/b)^c}} \qquad \text{[Eq. 2]}$$

where Y is concentration in the effluent, Y_o is concentration at time equal to 0, x is pore volumes passed through, and a, b, and c are slope and coefficients governing the graph.

Based on the changes in relative concentration and curve fit of the measured BTC for both soils, the slopes

Table 3. Comparison of the removal ratio of heavy metals adsorbed or leached from the soil column experiments.

Category	Soil Texture	Cr	Cd	Pb	Remark
Area above the BTC (A)	Sandy Clay Loam	0.60	0.47	0.57	Adsorption
	Clay	0.78	0.48	0.66	
Area below the BTC (B)	Sandy Clay Loam	0.40	0.53	0.43	Desorption
	Clay	0.22	0.53	0.34	
PV at $C_i/C_o = 1$	Sandy Clay Loam	20.57	15.99	17.86	Desorption
	Clay	19.76	16.66	18.99	
Adsorption	Sandy Clay Loam	12.39	7.46	10.10	
	Clay	15.50	7.98	12.44	

(a) were 6.12, 1.29, and 2.53 for Cr, Cd, and Pb, respectively, showing that the slope and parameter for pore volume (x) were increased with increasing affinity (adsorption), and intercept (Y_o) was 0 because there was not any heavy metal in the effluent due to adsorption and the column length was greater than 0.

To displace the heavy metals in soil columns, 30 mmol L^{-1} KCl was applied just after the relative concentration reached to 1 for each heavy metal. As shown in Fig. 1, similar leaching behavior of Cr, Pb, and Cd as a result of K-exchange was observed for the two soils (clay and sandy clay loam). The elution curves rapidly approached to 0 within 8 pore volumes of effluent collected while the displacement of the heavy metals were relatively smaller than the amount of removed by both soils. However, release of Cr, Cd, and Pb continued with long tailings over the entire duration of the experiment. The tailings in sandy clay loam were relatively longer than those in clay soil, which was probably due slow and exchange of Cr, Pb, and Cd by K.

Fig. 2 shows the BTC and the elution of binary systems of Cr, Cd, and Pb from sandy clay loam and
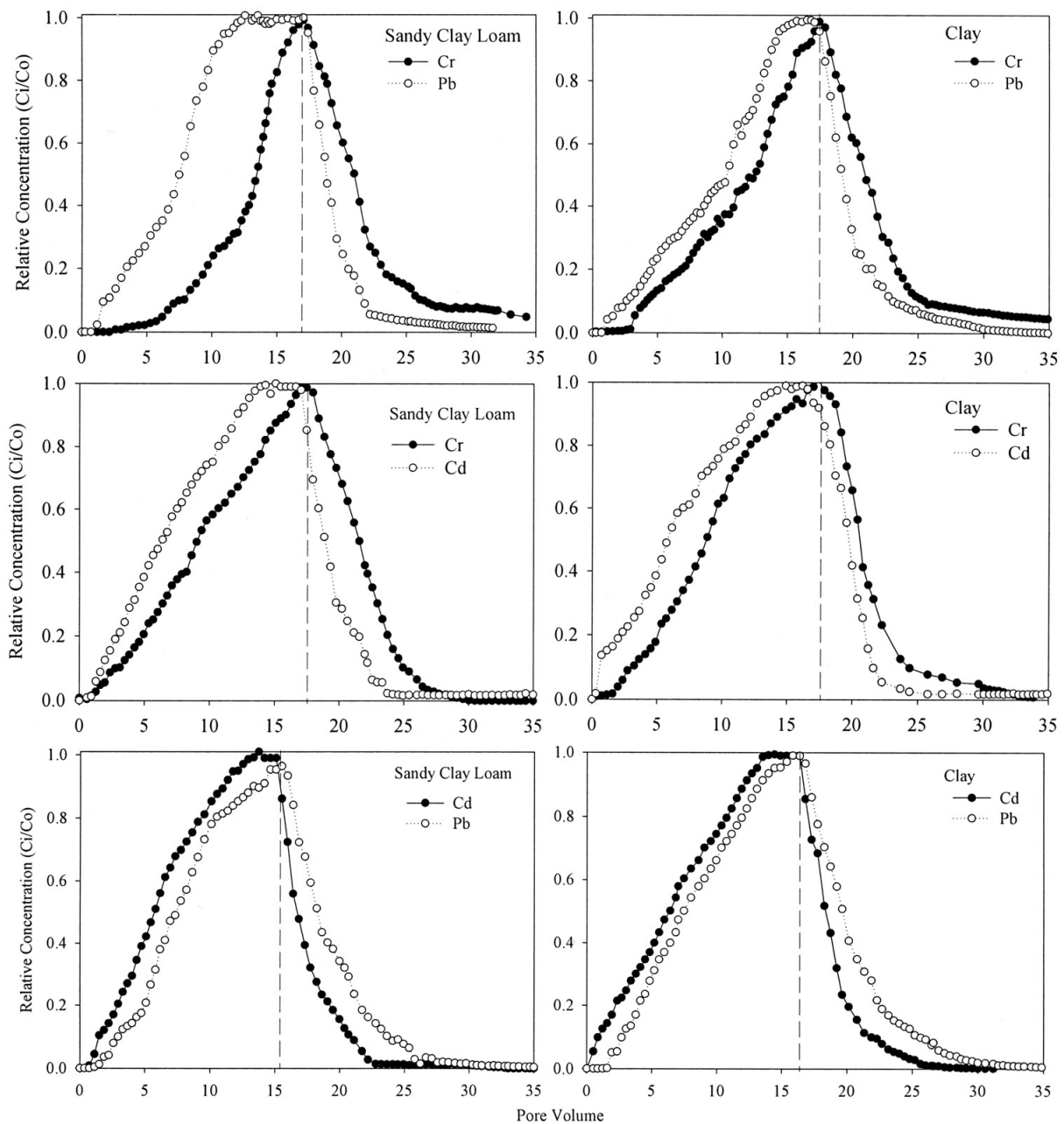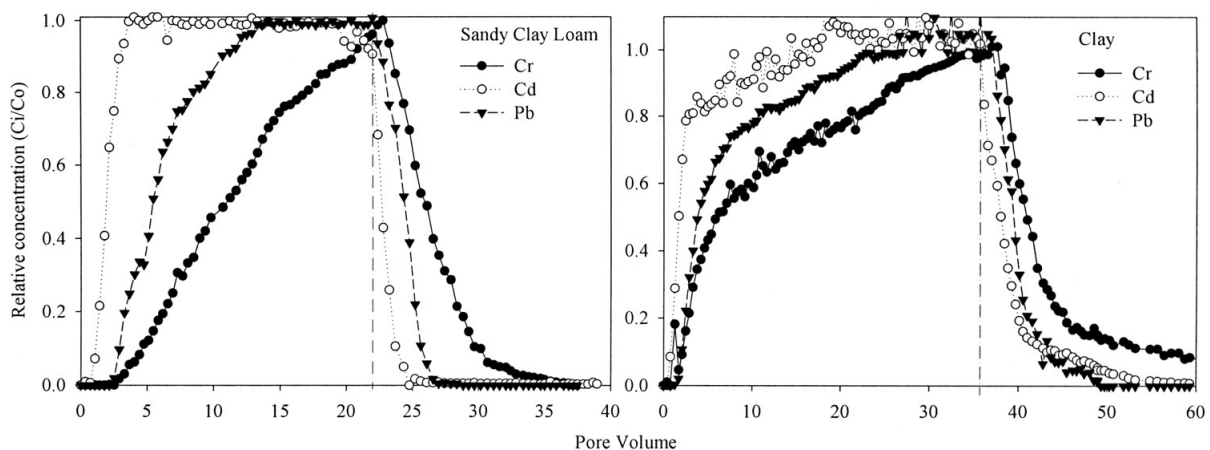

Fig. 2. BTCs and elution of binary combinations of Cr, Cd, and Pb in sandy clay loam and clay columns as a function of pore volume. The elution curves were obtained by switching to 30 mmol L^{-1} KCl after the relative concentration of BTC reached to 1.

clay columns as a function of pore volume. Competition between chemical species in solution for adsorption sites on soil surfaces can be of major significance in determining the effective mobility of any potentially adsorbing species. For all binary combination of heavy metals for the BTC curves, we found that one of the two heavy metals showed retarded occurrence to reach the relative concentration equal to 1. This retardation of BTCs could be explained by two general processes such as physical nonequilibrium and sorption-related nonequilibrium suggested by Brusseau et al., (1989). Physical nonequilibrium results from the existence of mobile and immobile domains within the porous medium and affects both sorbing and nonsorbing solutes. Sorption-related nonequilibrium involves only sorbing solutes and arises from chemical nonequilibrium and intrasorbent diffusion. Slight changes in BTC-elution of the binary system of the heavy metal indicated that some physical nonequilibrium transport occurred between sandy clay loam and clay. Also it was noteworthy to observe distinct retardation of the BTC from clay soil which had smaller flux during the experimental period. Cr required approximately 3 pore volumes more than that of Pb for both sandy clay loam and clay, respectively. For Cr and Cd, Cr required approximately 6 and 1.5 pore volumes more for both sandy clay loam and clay, respectively. Also Pb needed approximately 3 and 4 pore volumes more than that of Cd for sandy clay loam and clay, respectively. Comparison of the BTC for the heavy metal combination, the heavy metal having the stronger electronegativity was more retarded. Therefore, the preferences for sorption of the competing heavy metals

were as follows: Cr > Pb, Cr > Cd, and Pb > Cd. This also indicated that the retarded BTC meant stronger affinity and higher adsorption onto soil particle surfaces in soils. Thus, we found that the pore volumes of the BTC in a given combination of the heavy metals were influenced by its affinity for sorption sites due to different electronegativity carried by the heavy metals.

For elution of corresponding BTC for the heavy metals displaced by K, the heavy metal having stronger electronegativity showed longer tailing from both soils. But the tailing was smaller and stiff from the heavy metal having stronger electronegativity. Tailing is typical for BTC obtained under nonequilibrium conditions (Brusseau et al., 1989) when the adsorption-desorption precess is reversible. The elution of the heavy metals measured from effluent which was displaced by K influenced by concentration between solution and solid. Generally the displaced ions can be readsorbed into the soil particle surfaces as far as the sorption sites are available during downward transport. However, readsorption can be determined by equilibrium concentration and affinity of ions. Based on the area above and below the BTC and elution, the total amount of the heavy metal eluted were much smaller in elution than those on BTC. Therefore, elution of multispecies in soils may show that the heavy metal having lower affinity can be leached out ahead of other competing ones.

For ternary heavy metal system in soils, the BTC showed distinct retardation to reach the relative concentration equal to 1 in the order of Cd, Pb and Cr for both soils (Fig. 3). The required pore volumes to reach the relative concentration of 1 increased in clay soils than those in sandy clay loam for the same heavy metal such

Fig. 3. BTC and Elution of ternary species of Cr, Cd, and Pb in sandy clay loam and clay columns as a function of pore volume. The elution curves were obtained by switching to 30 mmol L^{-1} KCl after the relative concentration of BTC was reached to 1.

as approximately 4.2 pore volumes and 20 pore volumes for Cd in sandy clay loam and clay soil, respectively. Similar trends were observed for Pb and Cr. Compared to the pore volumes of single and binary systems of the heavy metal to reach to the relative concentration equal to 1. The ternary system required more pore volumes. As mentioned above, these phenomena caused by physical nonequilibrium in addition to chemical reaction between the heavy metals and soil particles during transport through the column. Thus, we found that the number of pore volumes to reach the maximum relative concentration in BTC increased in the presence of increasing number of competing heavy metals.

Corresponding to BTCs, the elution of each heavy metal displaced by 30 mmol L^{-1} of K, the heavy metal having stronger electronegativity showed longer tailing from both soils. Also, the tailing in clay soil showed was longer than that in sandy clay loam. We could assumed that the tailing was influenced by soil texture which had different adsorption capacity according to permanent negative charge on the soil particle surfaces, as well as electronegativity. Heavy metal with comparatively weak adsorption affinity could relatively easily displaced by K and another displaced heavy metal in a given equilibrium concentration. The displacement of adsorbed heavy metal by another heavy metal in solution could be determined by concentration at equilibrium. Therefore, these elution patterns probably occurred because the heavy metals are initially adsorbed from the relatively similar concentration as solution phase although the amount of adsorption were different due to their adsorption affinity and then as solution was displaced downward and diluted the soil was unable to hold the adsorbed heavy metals against the decreasing concentration gradient. This leads to the conclusion that when heavy metal attraction occurred, the distribution coefficient was not a constant, and allowed the explanation that the tail of the different elution curves represent heavy metal strength held by soil with greater. Summation of the areas under the elution curves revealed that essentially all of the heavy matals adsorbed were not recovered with the concentration of K. From these results, we presumed that the displacement and transport of muilt-heavy metal in these soil system could be strongly influenced not only by concentration of competing heavy metal for sorption sites that had different electronegativity and potential, but also soil physical nonequilibrium in a given conditions.

Conclusion

BTC and elution which could be interpreted as adsorption and desorption in multispecies system in soil influenced by its respective strength of electronegativity and potential (charge to ionic radius). The pore volumes required to reach maximum of BTC and minimum of elution increased with increasing number of competing heavy metals for sorption sites. This indicated that there was competition for available sorption sites depending on the concentration at equilibrium. And the displaced heavy metals which could react with soil particles during downward movement could influenced the transport of heavy metals to some extent as far as they changed into immobilized form. However, the displacement and transport strongly affected by electronegativity help by heavy metal itself.

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사질식양토와 식토토양에서의 중금속의 용탈과 파쇄곡선

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본 실험은 포화수분상태에서 두개의 서로 다른 토양의 흡착가능장소에 대한 흡착능과 경쟁에 따른 Cd, Pb, 그리고 Cr 이온의 이동성을 조사하였다. 이 조사를 위하여 수용성상태로 단일, 이중, 삼중의 중금속 조합을 이용하였다. 두개의 토양시료는 밭토양의 지표면으로부터 20 cm 이내에서 채취한 토양을 사용하였다. 그리고 공극수량에 따른 출현과 용출곡선을 중금속용액과 치환용 K 이온용액을 가하여 각각의 곡선이 최대와 최소치에 이은 시점까지 조사하였다. 조사 결과 출현과 용출곡선은 대칭을 이루지 않았으며 용액상태로 존재하는 중금속이온의 종류가 증가됨에 따라 용출곡선의 미행이 증가되었을 뿐만 아니라 공극수량도 증가하는 경향을 보였다. 그리고 출현과 용출곡선을 기준하여 곡선의 위와 아래의 면적을 비교하여 본 결과 출현 후 K에 의한 용출면적은 상대적으로 작아 K에 의한 중금속 탈착은 작은 것으로 조사되었는데 이는 중금속이온이 가지는 전기음성도 차이에 기인한 것으로 추정되었다. 결론적으로 토양내에서 중금속이온의 이동은 토양내에서 존재하는 중금속이온의 종류가 2개 이상 존재하는 한 토양의 물리적 비평형과 용액상태의 화학적평형이 중금속이온 이동에 영향을 미치는 것으로 추정하였다.