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Sodium-Calcium-Magnesium Exchange Reactions on a Montmorillonitic Soil:

II. Ternary Exchange Reactions¹

PHILIP FLETCHER, KENNETH M. HOLTZCLAW, CLAIRE JOUANY, GARRISON SPOSITO, and C. S. LEVESQUE²

ABSTRACT

The exchange of magnesium for calcium at 298 K on a montmorillonitic soil separate (principally clay and silt) was investigated in a 50 mol m⁻³ perchlorate background at pH 5, 6, and 7. The charge fraction of sodium in the aqueous solution phase was controlled at 0.70 or 0.85 during the exchange experiments, resulting in average exchangeable sodium percentages ranging from 5 to 25% on the soil. Exchange isotherms for magnesium indicated some preference on the soil for calcium over magnesium. This preference was attributed primarily to the presence of organic matter associated with the soil separate. However, the exchange isotherms showed no effect of pH in the range between 5 and 7, and no effect of exchangeable sodium percentage in the range between 0 and 25%.

Additional Index Words: cation exchange, exchangeable sodium percentage, pH effect on cation exchange, soil salinity.

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THE exchange of Mg²⁺ for Ca²⁺ at 25°C on specimen montmorillonite suspended in a 50 mol m⁻³ perchlorate background at pH 7 was investigated recently by Sposito et al. (1983) in respect to the effect of exchangeable sodium percentage (ESP). In the ESP range between 0 and 36%, the clay was found to exhibit equal affinities for Ca²⁺ and Mg²⁺ within the precision of the experiments (Sposito et al., 1983, Fig. 1). A similar result has been reported by Levy et al. (1983), who showed that the ratio of Mg charge fraction to Ca charge fraction on specimen montmorillonite initially at zero ESP tended not to shift significantly after equilibration of the clay with 200 mol m⁻³ NaCl to produce an ESP of about 27%. Both of these studies lend support to the long-standing hypothesis that, on arid-zone soil clays, Ca²⁺ and Mg²⁺ show essentially identical exchange selectivities (Bresler et al., 1982).

In this paper, the selectivity question addressed by Sposito et al. (1983) for pure montmorillonite is investigated for a montmorillonitic soil separate associated with organic matter. The exchange of Mg for Ca at ESP values between 0 and 25% is studied in a 50 mol m⁻³ perchlorate background maintained at pH 5, 6, or 7. The principal objective of the research was to determine the effect of ESP (in the range of practical importance) on the affinity of the soil separate for Ca and Mg. Fletcher et al. (1984) found that Ca was preferred slightly over Mg at zero ESP using the same soil separate as in the present study. Moreover, the exchange isotherms determined by Fletcher et al. (1984) at pH 6, 6.6, and 7 were congruent, indicating no effect

of proton competition on the selectivity relationship. A similar kind of soil exchange investigation at non-zero ESP does not appear to have been published heretofore, although Robbins et al. (1980) have reported data pertaining to Ca-Mg exchange at pH values between 6 and 7 on two Entisols in the presence of adsorbed Na and K. These two soils, which contained only illite and kaolinite in the clay fraction, showed preference for Ca over Mg at ESP values in the narrow range of 0 to 4% which resulted from the composition of the waters used to irrigate the soils. No attempt was made to study the effect of ESP in a broader range under controlled conditions of pH and ionic background medium as in the present experiments.

MATERIALS AND METHODS

Soil Separate

A sample of Altamont clay loam (fine, montmorillonitic, thermic Typic Chromoxererts) was used in this study. The pH value of the saturated soil paste is 5.7 and the content of organic C in this soil is 2.9 ± 0.1% (w/w). The cation exchange capacity of the soil is 0.38 mol(p⁺) kg⁻¹ at pH 8.2. The procedures used to obtain a soil separate consisting almost entirely of clay and silt and to prepare the separate in the sodium-saturated form have been described in detail by Fletcher et al. (1984). The soil-separate batches had an average organic C content of 2.6 ± 0.7% (w/w).

Exchange Experiments

Sodium-soil samples were reacted at 25 ± 0.3°C with ternary mixed salt solutions of NaClO₄/Ca(ClO₄)₂/Mg(ClO₄)₂ following the general procedure described by Fletcher et al. (1984). The total perchlorate concentration was maintained within 10% of 50 mol m⁻³ in all experiments. In one series of exchange experiments, the charge fraction of Na in the aqueous solution phase ($\bar{E}_{Na}$) was 0.70 and the pH value was maintained at 6.0 ± 0.2 or 7.02 ± 0.06. In another series of experiments, $\bar{E}_{Na}$ was 0.85 and the pH value was 4.7 ± 0.3, 5.8 ± 0.1, or 6.9 ± 0.2.

Chemical Analysis

Soil Concentration

The concentration of Na-soil in the stock suspension was determined as described by Sposito et al. (1981). The concentration of the Na-soil suspension was 44.0 ± 0.0 g soil kg⁻¹ suspension.

Supernatant and Soil Slurry Composition

Each of the three replicate supernatant solutions in an exchange experiment was analyzed for Na by flame emission and for Ca and Mg by flame absorption on a Perkin-Elmer Model 5000 atomic absorption spectrophotometer. The three soil slurries recovered in each experiment were analyzed for their content of Na, Ca, Mg, and water as described by Sposito et al. (1983) and Fletcher et al. (1984).

Data Analysis

The surface excesses and moles of adsorbed charge of Na, Ca, and Mg were calculated as described by Sposito et al. (1981). The total adsorbed metal charge in a perchlorate

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Table 1—Experimental data on Na-Ca-Mg exchange at $\bar{E}_{Na} = 0.70$ on Altamont soil.

c_{Na}	c_{Ca}	c_{Mg}	q_{Na}	q_{Ca}	q_{Mg}	Q_0
mmol kg ⁻¹			mol (±) kg ⁻¹			
pH 6.0 ± 0.2						
33.0 ± 0.1	0.129 ± 0.000	7.80 ± 0.03	0.067 ± 0.002	0.019 ± 0.002	0.582 ± 0.001	0.67
32.9 ± 0.1	0.984 ± 0.003	7.03 ± 0.05	0.066 ± 0.004	0.101 ± 0.001	0.502 ± 0.001	0.67
33.14 ± 0.04	1.867 ± 0.006	6.25 ± 0.03	0.01 ± 0.01	0.169 ± 0.002	0.437 ± 0.006	0.62
33.2 ± 0.2	2.633 ± 0.000	5.48 ± 0.01	0.03 ± 0.01	0.241 ± 0.002	0.372 ± 0.002	0.64
32.95 ± 0.05	3.51 ± 0.03	4.73 ± 0.01	0.03 ± 0.01	0.300 ± 0.005	0.319 ± 0.007	0.65
33.0 ± 0.1	4.384 ± 0.000	3.95 ± 0.02	0.01 ± 0.01	0.351 ± 0.002	0.254 ± 0.004	0.62
33.05 ± 0.09	5.26 ± 0.03	3.17 ± 0.01	0.02 ± 0.02	0.401 ± 0.001	0.194 ± 0.001	0.61
33.1 ± 0.2	6.10 ± 0.00	2.39 ± 0.09	0.026 ± 0.003	0.463 ± 0.002	0.145 ± 0.002	0.63
33.05 ± 0.05	6.98 ± 0.03	1.455 ± 0.002	0.037 ± 0.007	0.513 ± 0.006	0.105 ± 0.002	0.65
33.24 ± 0.09	7.86 ± 0.03	0.80 ± 0.01	0.044 ± 0.002	0.567 ± 0.009	0.049 ± 0.001	0.66
33.2 ± 0.1	8.77 ± 0.03	0.019 ± 0.001	0.042 ± 0.005	0.60 ± 0.01	0.000	0.65
pH 7.02 ± 0.06						
33.17 ± 0.06	0.130 ± 0.004	7.73 ± 0.05	0.08 ± 0.03	0.022 ± 0.001	0.55 ± 0.05	0.66
33.0 ± 0.2	1.00 ± 0.02	7.00 ± 0.03	0.04 ± 0.03	0.110 ± 0.002	0.52 ± 0.05	0.67
33.0 ± 0.1	1.87 ± 0.02	6.29 ± 0.02	0.066 ± 0.002	0.187 ± 0.003	0.414 ± 0.006	0.67
33.19 ± 0.09	2.78 ± 0.00	5.51 ± 0.04	0.067 ± 0.002	0.255 ± 0.001	0.350 ± 0.004	0.67
35.9 ± 0.3	3.73 ± 0.00	4.72 ± 0.03	0.071 ± 0.005	0.309 ± 0.001	0.308 ± 0.003	0.69
36.3 ± 0.2	4.51 ± 0.04	3.94 ± 0.02	0.062 ± 0.005	0.365 ± 0.001	0.249 ± 0.001	0.68
36.2 ± 0.2	5.43 ± 0.04	3.13 ± 0.02	0.069 ± 0.004	0.418 ± 0.003	0.197 ± 0.001	0.68
36 ± 1	6.5 ± 0.2	2.39 ± 0.07	0.068 ± 0.007	0.473 ± 0.001	0.146 ± 0.001	0.69
36.03 ± 0.09	7.32 ± 0.04	1.78 ± 0.02	0.07 ± 0.01	0.522 ± 0.006	0.094 ± 0.001	0.68
36.0 ± 0.4	8.24 ± 0.04	0.80 ± 0.01	0.066 ± 0.001	0.567 ± 0.002	0.049 ± 0.001	0.68
35.4 ± 0.3	9.1 ± 0.1	0.0180 ± 0.004	0.072 ± 0.004	0.616 ± 0.002	0.0029 ± 0.0008	0.69

background medium, Q_0 , was calculated with the equation:

$$Q_0 = \Gamma_{Na} + 2(\Gamma_{Ca} + \Gamma_{Mg}) \equiv q_{Na} + q_{Ca} + q_{Mg} \quad [1]$$

where Γ_M is a surface excess of metal M (M = Na, Ca, or Mg), in moles per kilogram of soil, and q_M is the corresponding number of moles of adsorbed metal charge per kilogram

of soil. The charge fraction of each metal in the exchanger and aqueous solution phases were calculated with Eq. [2] in Fletcher et al. (1984). The fraction of adsorbed bivalent metal cation charge accounted for by Mg (E'_{Mg}) and the similar charge fraction for the aqueous solution phase ($\bar{E}'_{Mg}$) were calculated with the equation (Sposito et al., 1983):

Table 2—Experimental data on Na-Ca-Mg exchange at $\bar{E}_{Na} = 0.85$ on Altamont soil.

c_{Na}	c_{Ca}	c_{Mg}	q_{Na}	q_{Ca}	q_{Mg}	Q_0
mmol kg ⁻¹			mol (±) kg ⁻¹			
pH 4.7 ± 0.3						
46.0 ± 0.2	0.000	3.72 ± 0.03	0.16 ± 0.03	0.000	0.46 ± 0.02	0.62
46.00 ± 0.06	0.548 ± 0.003	3.43 ± 0.04	0.16 ± 0.04	0.0897 ± 0.0007	0.391 ± 0.006	0.64
46.0 ± 0.2	0.972 ± 0.008	3.03 ± 0.01	0.16 ± 0.03	0.16 ± 0.01	0.342 ± 0.009	0.66
46.08 ± 0.09	1.42 ± 0.02	2.62 ± 0.02	0.17 ± 0.03	0.206 ± 0.004	0.304 ± 0.007	0.68
46.21 ± 0.05	1.897 ± 0.007	2.213 ± 0.008	0.15 ± 0.06	0.251 ± 0.007	0.255 ± 0.009	0.66
46.3 ± 0.1	2.348 ± 0.002	1.880 ± 0.007	0.15 ± 0.04	0.298 ± 0.004	0.202 ± 0.005	0.65
46.18 ± 0.05	2.9 ± 0.1	1.54 ± 0.03	0.17 ± 0.03	0.34 ± 0.01	0.148 ± 0.006	0.66
46.2 ± 0.2	3.274 ± 0.006	1.123 ± 0.007	0.14 ± 0.02	0.386 ± 0.007	0.116 ± 0.004	0.65
46.3 ± 0.2	3.76 ± 0.02	0.76 ± 0.01	0.13 ± 0.04	0.393 ± 0.008	0.074 ± 0.004	0.59
46.4 ± 0.05	4.198 ± 0.009	0.392 ± 0.002	0.15 ± 0.03	0.43 ± 0.01	0.042 ± 0.002	0.63
46.1 ± 0.2	4.69 ± 0.02	0.000	0.13 ± 0.04	0.470 ± 0.002	0.000	0.60
pH 5.8 ± 0.1						
47.3 ± 0.1	0.000	3.71 ± 0.02	0.183 ± 0.006	0.000	0.481 ± 0.003	0.66
47.2 ± 0.1	0.52 ± 0.01	3.35 ± 0.01	0.187 ± 0.007	0.100 ± 0.001	0.412 ± 0.003	0.70
47.2 ± 0.1	0.93 ± 0.01	3.01 ± 0.02	0.165 ± 0.002	0.159 ± 0.001	0.352 ± 0.002	0.68
47.4 ± 0.1	1.348 ± 0.008	2.61 ± 0.01	0.160 ± 0.005	0.2148 ± 0.0002	0.303 ± 0.0003	0.68
47.4 ± 0.1	1.777 ± 0.007	2.24 ± 0.01	0.184 ± 0.003	0.266 ± 0.001	0.256 ± 0.001	0.71
47.2 ± 0.2	2.19 ± 0.02	1.88 ± 0.01	0.176 ± 0.005	0.315 ± 0.001	0.210 ± 0.001	0.70
47.2 ± 0.2	2.63 ± 0.01	1.522 ± 0.005	0.156 ± 0.006	0.352 ± 0.003	0.161 ± 0.003	0.67
47.1 ± 0.1	3.08 ± 0.01	1.122 ± 0.003	0.153 ± 0.007	0.400 ± 0.001	0.120 ± 0.001	0.67
47.19 ± 0.03	3.50 ± 0.01	0.764 ± 0.009	0.16 ± 0.01	0.439 ± 0.003	0.0789 ± 0.0003	0.68
47.09 ± 0.09	3.96 ± 0.01	0.387 ± 0.000	0.16 ± 0.02	0.480 ± 0.001	0.044 ± 0.003	0.69
46.83 ± 0.08	4.370 ± 0.003	0.000	0.166 ± 0.004	0.522 ± 0.005	0.000	0.69
pH 6.9 ± 0.2						
39.7 ± 0.2	3.68 ± 0.06	0.000	0.144 ± 0.004	0.552 ± 0.003	0.000	0.70
39.8 ± 0.3	3.38 ± 0.03	0.388 ± 0.002	0.146 ± 0.003	0.500 ± 0.005	0.0501 ± 0.0006	0.70
39.7 ± 0.2	2.98 ± 0.03	0.766 ± 0.003	0.146 ± 0.006	0.459 ± 0.006	0.098 ± 0.001	0.70
39.8 ± 0.2	2.55 ± 0.03	1.07 ± 0.02	0.150 ± 0.003	0.409 ± 0.003	0.144 ± 0.002	0.70
39.2 ± 0.2	2.23 ± 0.02	1.42 ± 0.01	0.155 ± 0.003	0.358 ± 0.004	0.196 ± 0.002	0.71
39.7 ± 0.2	1.90 ± 0.02	1.86 ± 0.03	0.154 ± 0.002	0.317 ± 0.002	0.243 ± 0.002	0.71
39.6 ± 0.3	1.52 ± 0.01	2.26 ± 0.04	0.156 ± 0.004	0.268 ± 0.003	0.299 ± 0.002	0.72
39.6 ± 0.3	1.154 ± 0.006	2.61 ± 0.03	0.156 ± 0.003	0.211 ± 0.002	0.346 ± 0.005	0.71
39.7 ± 0.2	0.794 ± 0.009	2.98 ± 0.05	0.191 ± 0.004	0.154 ± 0.002	0.406 ± 0.005	0.75
39.8 ± 0.2	0.445 ± 0.005	3.36 ± 0.05	0.159 ± 0.003	0.0956 ± 0.0007	0.482 ± 0.006	0.74
39.7 ± 0.3	0.000	3.63 ± 0.03	0.163 ± 0.002	0.000	0.596 ± 0.003	0.76

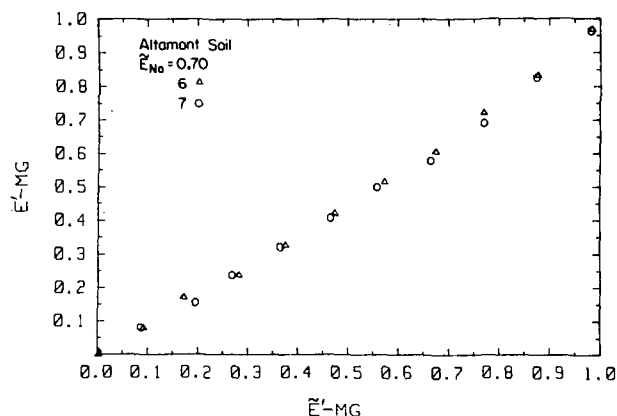


Fig. 1—Effect of pH on the E'_Mg - $\tilde{E}_Mg$ relationship at $\tilde{E}_{Na} = 0.70$ for Altamont soil.

$$E'_{Mg} = q_{Mg}/(q_{Ca} + q_{Mg}) \quad \tilde{E}'_{Mg} = c_{Mg}/(c_{Ca} + c_{Mg}) \quad [2]$$

where c_M is in mmol/kg solution.

RESULTS AND DISCUSSION

The primary laboratory data from the ternary exchange experiments are listed in Tables 1 and 2. In these tables, c is an equilibrium millimolality (millimoles per kilogram of solution) and q is an adsorbed cation charge. The last column in each table gives values of the total adsorbed metal cation charge.

Figures 1 and 2 show plots of E'_Mg against $\tilde{E}_Mg$ for varying pH values at $\tilde{E}_{Na} = 0.70$ and 0.85 , respectively. For $Ca \rightarrow Mg$ exchange at $\tilde{E}_{Na} = 0.0$, this kind of plot is an exchange isotherm. The figures demonstrate clearly that, within the precision of the data, there is no effect of pH on the relationship between the Mg-fraction of the total bivalent cation charge on the exchanger and that in aqueous solution. This same result was found by Fletcher et al. (1984, Fig. 3) for $Ca \rightarrow Mg$ exchange at $\tilde{E}_{Na} = 0.0$ on the Altamont soil. The experiments at $\tilde{E}_{Na} = 0.70$ (Fig. 1) correspond to $E_{Na} = 0.10 \pm 0.01$ and 0.05 ± 0.03 at pH 7 and 6, respectively, whereas those at $\tilde{E}_{Na} = 0.85$ (Fig. 2) correspond to $E_{Na} = 0.22 \pm 0.01$, 0.25 ± 0.02 , and 0.24 ± 0.02 at pH 7, 6, and 5, respectively. Thus the pH-independence of the $\tilde{E}'_{Mg}$ - $\tilde{E}_Mg$ relation on the Altamont soil has been shown for ESP between 0 and 25%. The sharp reduction in ESP from 10 to 5% at $\tilde{E}_{Na} = 0.70$ as the pH value decreased from 7 to 6 also suggests a

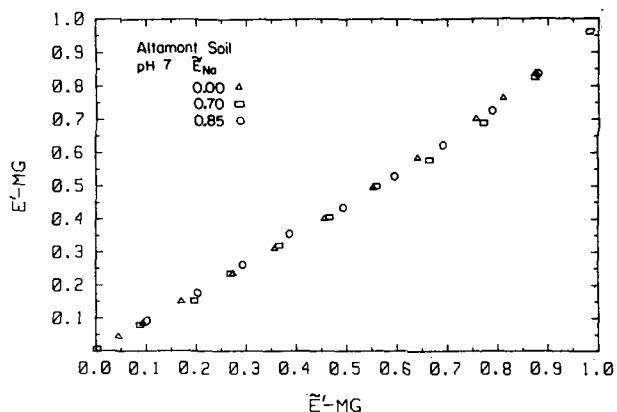


Fig. 3—Effect of $\tilde{E}_{Na}$ on the E'_Mg - $\tilde{E}_Mg$ relationship at pH 7 for Altamont soil.

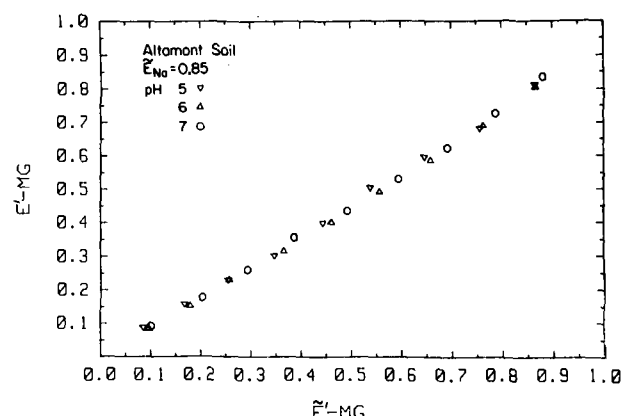


Fig. 2—Effect of pH on the E'_Mg - $\tilde{E}_Mg$ relationship at $\tilde{E}_{Na} = 0.85$ for Altamont soil.

relatively low affinity of the Altamont soil separate for Na^+ in the face of competition by protons.

Figures 3 and 4 show plots of E'_Mg against $\tilde{E}_Mg$ at fixed pH values with varying $\tilde{E}_{Na}$. At pH 7 there is, within the precision of the experimental data, congruence of the three curves determined at $E_{Na} = 0.00$, 0.10 , and 0.22 . Thus, for ESP values up to 22%, the E'_Mg - $\tilde{E}_Mg$ relationship at pH 7 on the Altamont soil in a perchlorate background is invariant. The same conclusion can be drawn from Fig. 4 at pH 6 for ESP values up to 25%. The ESP-independence of the E'_Mg - $\tilde{E}_Mg$ relation was also found by Sposito et al. (1983, Fig. 1) on specimen montmorillonite at pH 7 in 50 mol m^{-3} perchlorate. However, in that study, the isotherm was a C-curve (Sposito, 1981, Chapt. 5) indicating no preference for Ca over Mg. In the present study, all of the isotherms lie somewhat below a C-curve and, therefore, Ca is slightly preferred over Mg on the Altamont soil. This preference, as discussed by Fletcher et al. (1984), is consistent with the presence of organic matter in the soil.

If it is correct to assume that the surface-reactive portion of the Altamont soil suspension in the present experiments ($0 \leq ESP \leq 25\%$) contained well-developed quasicrystals, as would exist in pure montmorillonite suspensions under the same conditions (Shainberg and Letey, 1984), then $Ca \rightarrow Mg$ exchange should have involved principally the interlayer sites

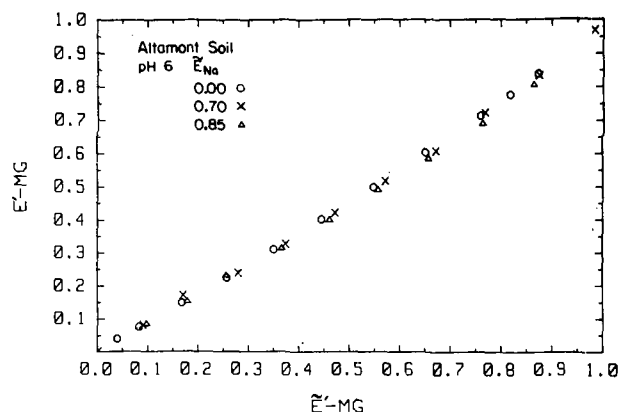


Fig. 4—Effect of $\tilde{E}_{Na}$ on the E'_Mg - $\tilde{E}_Mg$ relationship at pH 6 for Altamont soil.

Table 3—Effects of Mg saturation and pH on Q_0 , the total adsorbed metal cation charge.

E_{Na^+} : 0.00†		0.22		0.00†		0.05	
pH: 7.0		7.0		6.0		6.0	
Q_0	E_{Mg}	Q_0	E_{Mg}	Q_0	E_{Mg}	Q_0	E_{Mg}
mol (±)		mol (±)		mol (±)		mol (±)	
kg ⁻¹		kg ⁻¹		kg ⁻¹		kg ⁻¹	
0.64	0.04	0.70	0.00	0.48	0.04	0.65	0.00
0.65	0.08	0.70	0.07	0.50	0.07	0.66	0.08
0.68	0.15	0.70	0.14	0.53	0.15	0.65	0.16
0.70	0.24	0.70	0.21	0.56	0.22	0.63	0.23
0.69	0.31	0.71	0.28	0.56	0.31	0.61	0.32
0.69	0.40	0.71	0.34	0.58	0.40	0.62	0.42
0.69	0.49	0.72	0.41	0.58	0.50	0.65	0.49
0.68	0.59	0.71	0.49	0.58	0.60	0.64	0.58
0.70	0.70	0.75	0.54	0.59	0.71	0.62	0.71
0.73	0.76	0.74	0.65	0.60	0.78	0.67	0.75
0.75	0.84	0.76	0.79	0.61	0.84	0.67	0.87

† Data from Table 5 of Fletcher et al. (1984).

with little or no competition from Na^+ residing primarily on external surfaces. This hypothesis, based on a high bivalent cation selectivity on the internal surfaces of quasicrystals, is in agreement with the data in Fig. 3 and 4. On the other hand, the data also support a selectivity of organic matter for bivalent cations relative to Na^+ . Both sources of bivalent cation exchange selectivity likely contribute to the ESP independence indicated in Fig. 3 and 4.

To the extent that stable quasicrystals (which require $ESP < 15\%$) existed in the surface-reactive portion of the soil suspension at $\bar{E}_{Na} = 0.70$ and induced exchangeable Na^+ to reside principally on external surfaces, the pH effect on ESP, noted above, can be interpreted as evidence for organic matter with its pH-dependent charge also being bound to external surfaces. If external surfaces are primary sites for bound organic matter, then the Ca-selectivity apparent in Fig. 1 to 4 could be attributed to Na-Ca-Mg exchange com-

petition on these surfaces. The interlayer surfaces would be largely free of organic matter, as might be supposed for optimal quasicrystal formation, and no selectivity for Ca would derive from them, as shown by Sposito et al. (1983) for specimen montmorillonite. This hypothesis certainly will require further experimentation to test its correctness. It is consistent, however, with the bivalent cation effects on Q_0 observed by Fletcher et al. (1984, Fig. 4) in $Na \rightarrow Ca$ and $Na \rightarrow Mg$ exchanges on the Altamont soil, in that Q_0 remained constant once E_M ($M = Ca$ or Mg) reached the threshold value of 0.6 for stable quasicrystal formation.

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