5-1-1979

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D. N. Brown

F. E. Hutchinson

THE EFFECT OF ACIDITY, ORGANIC MATTER, AND SESQUIOXIDE POLYMERS ON THE PERMANENT CHARGE AND pH-DEPENDENT CATION EXCHANGE CAPACITY OF THE CARIBOU LOAM SOIL

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THE EFFECT OF ACIDITY, ORGANIC MATTER AND SESQUIOXIDE POLYMERS ON THE PERMANENT CHARGE AND pH-DEPENDENT CATION EXCHANGE CAPACITY OF THE CARIBOU LOAM SOIL[1]

D.N. Brown and F.E. Hutchinson[2]

INTRODUCTION

It has become apparent in recent years that the cation exchange capacity (CEC) of a soil consists of two components, i.e., the permanent charge and the pH-dependent charge. The so-called "permanent charge" component typically measured by 1N KCl (4, 14, 16, 19) has been shown not to be equated with isomorphous substitution. It represents the exchange sites which have not been de-activated by complexed Al and most of this component of CEC is present in the organic fraction. Numerous investigators (4, 16, 19) have defined the pH-dependent CEC as the acidity not displaced by neutral salt but that which is displaced by BaCl₂-TEA buffered at pH 8.2.

The important contribution of organic matter to the pH-dependent CEC of soils has been reported by several workers (2, 3, 18, 19, 20). McLean, et al. (16) correlated pH-dependent CEC before and after organic matter removal from soils in a lime experiment and obtained a highly significant relationship (r=0.942).

Polymers of Al and Fe can lower CEC values for soils by fixation on or within clays, as reported by Sawhney (22).

Interlayers of Al and Fe are formed by "chloritization" in the three-layer expanding silicate clays; this process can materially lower the CEC of some soils. Experimental evidence suggests that this component is present in many soils (7, 8, 9, 12, 13, 21, 22, 24, 25, 26) and that it may reduce the CEC of some surface soils by as much as 30 percent.

[2]Former Graduate Assistant, and Professor of Soil Science, respectively, Plant and Soil Sciences, University of Maine at Orono.

It is apparent from the previous notations that the CEC in a soil available for retention or exchange of cations commonly applied to soils by fertilization (Ca, K, Mg, NH₄) varies tremendously, depending upon acidity, organic matter content, and presence of coatings and interlayer deposits of Al and Fe polymers. In this study the components of CEC were measured in an acid spodosol to determine 1) the increase in KCl-CEC in the whole soil when limed, 2) the contribution of the organic fraction to CEC and 3) the magnitude of CEC reduction by Al and Fe polymers.

MATERIALS AND METHODS

The soil used in this study was the surface horizon (Ap) of the Caribou loam. Three lime levels of 0, 3629, and 7257 kg/0.405 ha of calcitic limestone were applied in the field and allowed to equilibrate for three years. At the end of the three year period, final soil pH values for the three treatments were 4.92, 5.86 and 6.50 respectively.

Analyses were made on the soil samples before and after organic matter removal as well as after removal of interlayer material and hydroxy-Al and/or -Fe coatings. Parameters measured were pH, exchangeable aluminum, KCl-CEC, and pH-dependent CEC.

Soil pH was determined in a 1:1 ratio of soil to water.

Exchangeable aluminum was determined by the aluminon method (15) and the color development was measured with a Bausch and Lomb type 33-29-40 electrophotometer.

KCl-CEC was determined by displacing exchangeable bases plus exchangeable acidity with 1N neutral KCl solution. Potassium was then displaced from the soil sample with 1N NH₄OAc − pH 7.0 and measured by flame emission with a Jarrell-Ash Model 82-500 atomic absorption-flame emission spectrometer. The pH-dependent CEC was determined on all samples by removal and measurement of the acidity which remained after extraction with the KCl solution. The remaining acidity was extracted with BaCl₂-TEA-pH 8.2 and measured with 0.04 N HCl as outlined by Mehlich (17). Total CEC was considered to be the sum of KCl-CEC and pH-dependent CEC.

Interlayer material and hydroxy-Al and/or -Fe coatings were removed from the soil by sodium citrate extraction as originally proposed by Tamura (25). The procedure employed for removal of this material was a modification of methods used by Sawhney (22) and by Weed and Nelson (26). The pH of the sodium citrate solution was adjusted to 5.5 with HCl and a 10 percent ratio of soil to solution was maintained. The total extraction time was 9 hours and the solution was renewed twice during the extraction period. After the extraction procedure, excess sodium citrate was washed out of the soil by a series of three washings

with 0.5N $(NH_4)_2CO_3$ solution. The samples were then transferred to evaporating dishes and placed in an oven heated to 110C to drive off excess $(NH_4)_2CO_3$.

Simple linear regression equations were developed on all data with pH as the independent variable and exchangeable aluminum, KCl-CEC, pH-dependent CEC, and total CEC as dependent variables. Equations were developed before organic matter destruction, after organic matter destruction, and after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

Variables selected to have correlation coefficients determined were as follow:

X_1 Original pH

X_2 Exchangeable aluminum before organic matter destruction

X_3 Exchangeable iron before organic matter destruction

X_4 Organic matter

X_5 KCl-CEC before organic matter destruction

X_6 pH-dependent CEC before organic matter destruction

X_7 Total CEC before organic matter destruction

X_8 pH after organic matter destruction

X_9 Exchangeable aluminum after organic matter destruction

X_{10} KCl-CEC after organic matter destruction

X_{11} pH dependent CEC after organic matter destruction

X_{12} Total CEC after organic matter destruction

X_{13} pH after removal of interlayer material and hydroxy-A1 and/or -Fe coatings.

X_{14} Exchangeable aluminum after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

X_{15} KCl-CEC after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

X_{16} pH-dependent CEC after removal of interlayer material and hydroxy-Al and/or -Fe interlayers.

X_{17} Total CEC after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

RESULTS AND DISCUSSION

Relationship of Organic Matter to Cation Exchange Capacity

The mean value for concentration of organic matter in the soil samples used for this study was 3.51 percent. Since all original test plots were located in a general area on one soil series, small correlation coefficients were observed between organic matter and the various components of cation exchange capacity (Table 1).

Cation exchange capacity (CEC) was influenced by the presence of organic matter. This relationship is clearly shown in Table 2. In observ-

TABLE 1. — Correlation coefficients for the relationship between various chemical factors in the Caribou soil.[1]

Variable a	Variable b	r
X_1	X_2	− .8493**
X_1	X_3	− .6668**
X_1	X_5	.7290**
X_1	X_6	− .7320**
X_1	X_7	− .0749
X_2	X_3	.6979**
X_2	X_5	− .6171**
X_2	X_6	.6210**
X_4	X_5	.0162
X_4	X_6	.1771*
X_4	X_7	.1630
X_8	X_9	− .4322**
X_8	X_{10}	− .488
X_8	X_{11}	− .2095*
X_8	X_{12}	− .2650**
X_{13}	X_{14}	− .1002
X_{13}	X_{15}	.0530
X_{13}	X_{16}	− .1644
X_{13}	X_{17}	− .0454

[1]See page 3 for key to variables.
*P= .05
**P= .01

TABLE 2. — Mean values for pH, KCl-CEC, pH-dependent CEC, and total CEC, before and after organic matter destruction and removal of interlayer material and hydroxy-Al and/or -Fe coatings in the Caribou soil.

Phase of Investigation	pH	Cation exchange capacity (me/100g)		
		KCl	pH-dependent	Total
Before organic matter destruction	5.26[1]	6.19	9.66	15.77
After organic matter destruction	5.60	4.73	6.26	11.11
After removal of interlayer material and hydroxy-Al and/or -Fe coatings	6.33	4.19	3.57	7.75

[1]Statistical analyses were not possible because of variable replication between the final phase of investigation and the preceding phases.

ing the total CEC which is taken as the sum of the KCl-CEC and pH-dependent CEC, it is seen that a decrease occurred after organic matter destruction in the magnitude of 4.66 me/100g of soil, or 29 percent of the original value. These data are similar to those of other investigators (1, 10, 16, 20). Pratt and Blair (20) found that at pH 5.0, the contribution of organic matter to cation exchange capacity was 37 percent.

KCl-extractable cation exchange capacity (KCl-CEC) was decreased after organic matter destruction by 1.46 me/100g of soil, or 24 percent of its original value. Thus, nearly one-fourth of the KCl-CEC is located in the organic fraction of the Caribou soil at the organic matter content quoted above. If the organic matter content were raised, it can be assumed that the relative contribution to CEC would increase. It is also noted that the pH-dependent component of CEC was decreased by 3.40 me/100g of soil, or 34 percent of its original value as a result of organic matter destruction.

Destruction of organic matter resulted in an increase in pH (Table 2). The most plausible explanation for this phenomenon is that undissolved carbonates react with the soil-water system as a result of treatment with hydrogen peroxide.

Relationship of Lime Level to Cation Exchange Capacity

The pH of the soil increased significantly among the three lime levels (Table 3). Lime level was found to be the variable of most importance for predicting the two components of CEC in the Caribou soil. This relationship was most pronounced in the soil before organic matter destruction.

Figure 1 depicts the regression of total CEC on pH and shows a very low negative correlation coefficient between the two variables. The overall effect of pH on total CEC was only 0.25 me/100g for every unit increase in pH.

TABLE 3. — Magnitude of pH, exchangeable Al and Fe before organic matter destruction and pH and exchangeable Al after organic matter destruction in the Caribou soil as a result of three rates of limestone application.

Lime level kg/0.405 ha	Before organic matter destruction			After organic matter destruction	
	pH	Aluminum (me/100g of soil)	Iron (me/100g of soil)	pH	Aluminum (me/100g of soil)
0	4.72a[1]	1.05a	0.10a	5.25a	1.73a
3629	5.30b	0.27b	0.08b	5.63b	1.30a
7257	5.60c	0.06c	0.07c	5.91c	0.97a

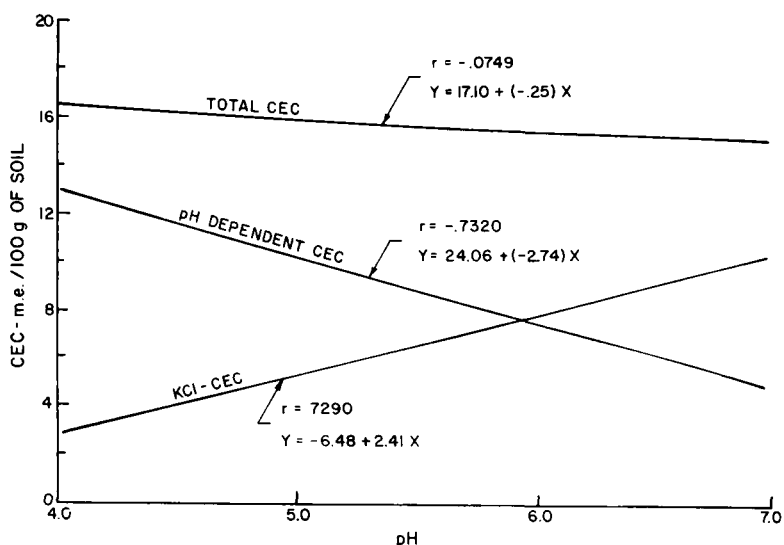[1]Values with a common letter do not differ significantly.

Figure 1. The regression of KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil before organic matter destruction.

Regression lines for KCl-CEC and pH-dependent CEC on pH in the Caribou soil before organic matter destruction are also illustrated in Figure 1. From the regression of KCl-CEC on pH, it can be predicted that for every unit increase in pH there will be an increase in KCl-CEC of approximately 2.41 me/100g and a decrease in pH-dependent CEC of 2.74 me. It can be seen from Table 4 the relationships of pH to KCl-CEC and to pH-dependent CEC were highly significant.

TABLE 4. — Correlation coefficients and equations for the regression of exchangeable Al and Fe, KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil before organic matter destruction.

X	Y	r	Y=a+bX
pH	Exchangeable aluminum	−.8493**	Y = 7.02+(−1.25)X
pH	Exchangeable iron	−.6668**	Y = .30+(− .04)X
pH	KCl-CEC	.7290**	Y = − 6.48+ 2.41 X
pH	pH-dependent CEC	−.7320**	Y = 24.06+(−2.74)X
pH	Total CEC	−.0749	Y = 17.10+(− .25)X

⁺P= .05
**P= .01

These results indicate that as the pH of a Caribou soil containing 3.5 percent organic matter is raised from 5.0 to 6.0 by liming, the positive charge of Al and Fe hydroxide ions decreases and the negative charge increases. The "charge blocking" by Al and Fe hydroxide ions is therefore decreased and negative sites become available for normal cation exchange.

From the regression equation (Figure 1) it can be calculated that the KCl-CEC (sometimes referred to as the "effective" CEC) in this soil is increased 40 percent within the pH range 5.0 to 6.0. Therefore, at a higher pH level, more of any cation would be required to maintain the same percentage saturation of the "effective" CEC than would be the case at a lower pH value.

Although the decrease in total CEC with increasing pH was highly significant (Table 5 and Figure 2), the linear correlation was relatively small ($r = -.2650$) and without practical importance. KCl-CEC was not significantly correlated with pH after organic matter destruction, and a statistically significant but very small negative correlation ($r = -.2095$) was found between pH-dependent CEC and pH after organic matter destruction.

TABLE 5. — Correlation coefficients and equations for the regression of exchangeable Al, KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil after organic matter destruction.

X	Y	r	$Y = a + bX$
pH	Exchangeable aluminum	$-.4322^{**}$	$Y = 4.41 + (-.55)X$
pH	KCl-CEC	$-.0488$	$Y = 5.32 + (-.10)X$
pH	pH-dependent CEC	$-.2095^{*}$	$Y = 9.16 + (-.52)X$
pH	Total CEC	$-.2650^{**}$	$Y = 16.15 + (-.90)X$

*$P = .05$
**$P = .01$

These results indicate that the cation exchange sites in the mineral soil which were measured by extraction with $BaCl_2$-TEA were not pH-dependent to any great degree. This is consistent with data reported by other investigators (2, 3, 18, 19, 20) who show that the pH-dependent CEC is primarily associated with the organic fraction of the soil system.

The Relationship of Interlayer Material and Hydroxy-Al and/or -Fe Coatings to Cation Exchange Capacity

Data presented in Table 2 indicate that the mean pH of the Caribou soil increased from 5.60 after organic matter destruction to 6.33 after removal of interlayer material and hydroxy-Al and/or -Fe coatings. This
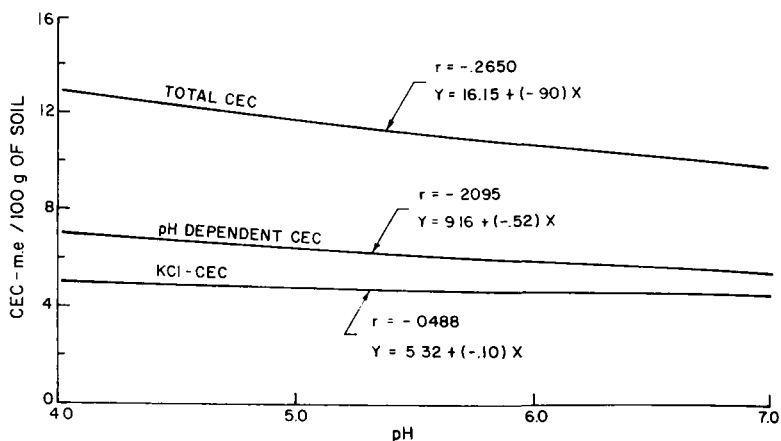
Figure 2. The regressions of KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil after organic matter destruction.

increase in pH may have been a result of the movement of the Al and Fe out of the soil system as a result of treatment with sodium citrate.

The correlation coefficients and regression lines for KCl-CEC, pH-dependent CEC, and total CEC on pH after removal of interlayer material and hydroxy-Al and/or -Fe coatings are found by referring to Table 6 and Figure 3.

The pH-dependent component of CEC decreased an additional 2.69 me/100g, or 43 percent of the previous value, after organic matter destruction and 28 percent of the original CEC. The KCl-CEC decreased slightly after removal of interlayer material and coatings from the value after organic matter destruction. It is probable that this component should have actually increased as a result of exchange sites being freed which had originally been occupied by hydroxy-Al and/or -Fe

TABLE 6. — Correlation coefficients and equations for the regression of exchangeable Al, KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

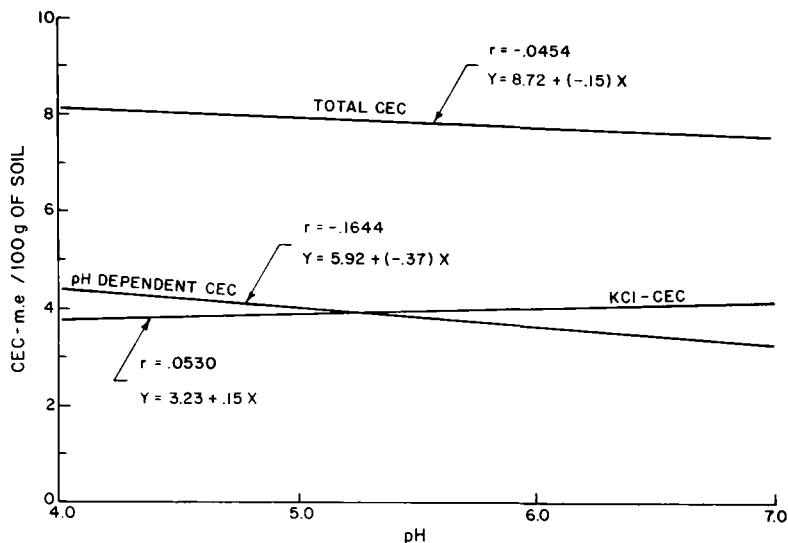| X	Y	r	Y=a+bX
pH	Exchangeable aluminum	− .1002	Y= .58+ (−.07)X
pH	KCl-CEC	.0530	Y=3.23+ .15 X
pH	pH-dependent CEC	− .1644	Y=5.92+ (−.37)X
pH	Total CEC	− .0454	Y=8.72+ (−.15)X

*P= .05
**P= .01

Figure 3. The regressions of KCl-CEC, pH-dependent CEC, and total CEC on pH in the Caribou soil after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

polymers. During the procedure of removing interlayer material and coatings, a drying step was included which could have collapsed the expandable clay minerals, resulting in their being held together so tightly that it would have been impossible for potassium cations to enter the interlayer space. It has been found previously (5, 6, 7, 22, 23, 26) that the removal of interlayer material increased CEC. By using calcium as the saturating cation and sodium as the displacing cation, Sawhney (22) found CEC of surface soil to increase as much as 30-40 percent after removal of interlayer material.

It is reasoned that the reduction in pH-dependent cation exchange capacity was a result of removing Al and Fe polymers.

The Relationship of Exchangeable Aluminum and Iron to Cation Exchange Capacity

There existed a relatively high positive correlation (r=.6979) between exchangeable Al and Fe as shown in Table 1. Concentrations of these ions were significantly reduced with increase in pH according to data presented in Table 3. The regression lines for exchangeable Al and Fe on pH before organic matter destruction are found in Figures 4 and 5, respectively. The Al data are consistent with those reported earlier for
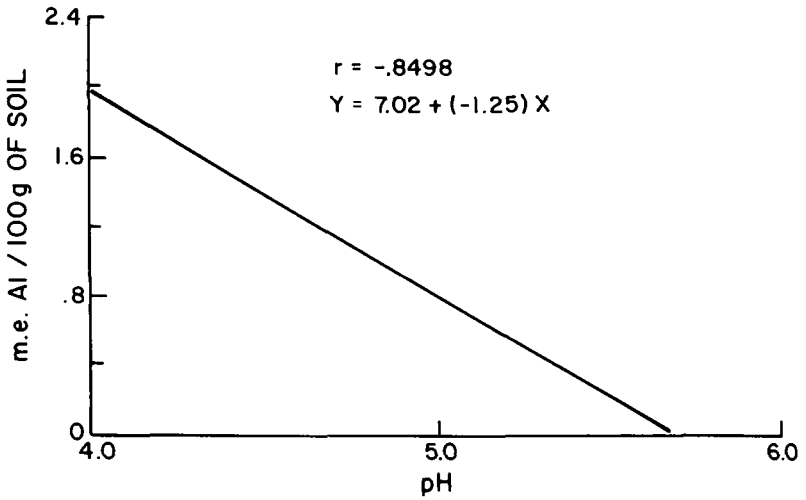
Figure 4. The regression of exchangeable Al on pH in the Caribou soil
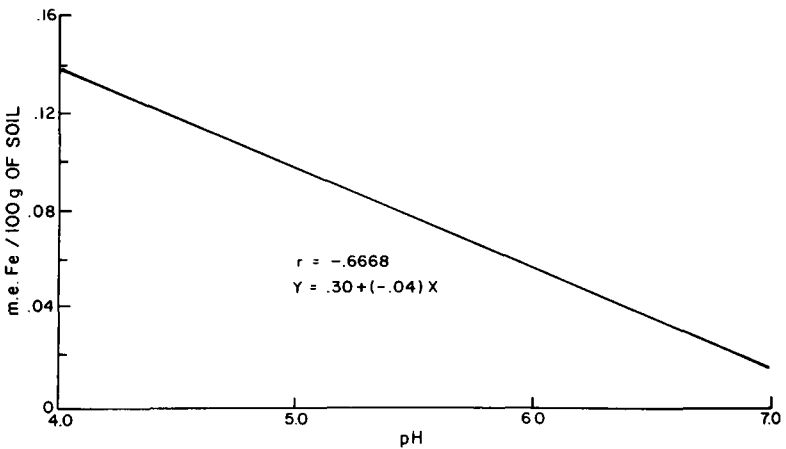before organic matter destruction.

Figure 5. The regression of exchangeable Fe on pH in Caribou soil
before organic matter destruction.

the Caribou soil (13). Although the values for exchangeable Fe are very
low, a high level of significance did occur and is further verified by a

relatively high negative correlation coefficient (r= − .6668) between exchangeable Fe and pH (Table 4). Because of the much higher Al values and the corresponding high correlation coefficient (r= .8493) with pH, it is assumed that Al is the more important of the two variables in this soil.

Data from Table 3 indicate that concentration of exchangeable Al increased greatly after organic matter destruction. The regression line for exchangeable Al on pH after organic matter destruction can be found by referring to Figure 6. An increase of 0.68, 1.03, and 0.91 me/100g was noted for the control treatment, 3629 kg and 7257 kg of limestone per 0.405 ha, respectively.
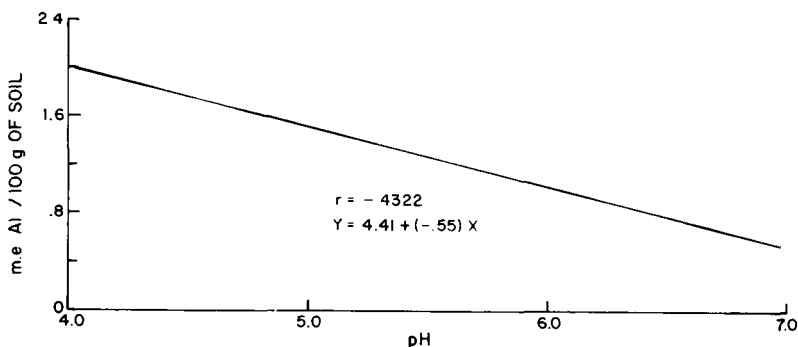

Figure 6. The regression of exchangeable Al on pH in the Caribou soil after organic matter destruction.

Clark (2, 3) suggests that fixation of Al and Fe may occur with the weakly acidic organic exchange sites in soil and that the removal of these fixed forms is a pH-dependent process like the dissociation of hydrogen from weakly acidic functional groups. Much of this complexed Al was not exchangeable with 1N KCl and therefore, it is assumed to make up a considerable portion of the pH-dependent component of CEC in the Caribou soil. There was a highly significant correlation (r= .6210) between exchangeable Al and pH-dependent CEC (Table 1). This compares almost exactly with the negative correlation (r= − .6171) between exchangeable Al and KCl-CEC.

Data for the relationship of exchangeable Al to lime level after removal of interlayer material and hydroxy-Al and/or -Fe coatings are presented in Table 7. The corresponding correlation coefficient and equation for the regression of exchangeable aluminum on pH can be found by referring to Figure 7. No significant correlation was found between these variables. This is explained by the fact that sodium citrate

TABLE 7. — The effect of three rates of lime application on the amount of exchangeable Al in the Caribou soil after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

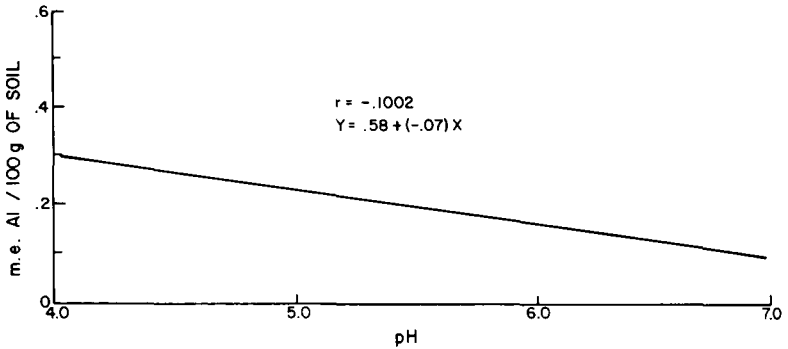| Lime Level kg/0.405 ha	pH	Aluminum (me/100g of soil)
0	6.36a	0.10a
3629	6.20a	0.15a
7257	6.45a	0.21a

Figure 7. The regression of exchangeable Al on pH in the Caribou soil after removal of interlayer material and hydroxy-Al and/or -Fe coatings.

behaves as a chelating agent and removes aluminum ions from the system.

SUMMARY

The components of cation exchange capacity (CEC) were measured in an acid (pH 4.7) spodosol (Caribou) to determine 1) the increase in "effective" CEC (KCl extractable) when limed, 2) relative contribution of the organic fraction to CEC and 3) the magnitude of CEC reduction by Al and Fe polymers and/or interlayer material.

A considerable portion of cation exchange sites in the Caribou soil, both permanent charge and pH-dependent, was found to be located in the organic matter fraction. Permanent charge CEC (KCl-extractable) decreased 24 percent and pH-dependent CEC decreased 34 percent as a result of organic matter destruction.

Lime level exerted a very pronounced effect on both components of CEC. As one of the components varied, the other varied by almost a

reciprocal amount. This was evidenced by the positive correlation coefficient (r= .7290) between pH and KCl-CEC and the negative correlation coefficient (r= − .7320) between pH and the pH-dependent CEC. According to regression curves that were developed for the Caribou soil, KCl-CEC increased approximately 2.41 me/100g of soil for every unit increase in pH between 4.7 and 5.6 and pH-dependent CEC decreased approximately 2.74 me/100g in the same pH range.

Exchangeable Al and Fe increased significantly with a decrease in soil pH. There was a highly significant correlation (r= .6210) between exchangeable Al and pH-dependent CEC and a highly negative correlation (r= − .6171) between aluminum and KCl-CEC, indicating that the Al ion is very active in blocking cation exchange sites in the Caribou soil within the acid range below pH 5.5.

Nearly one-third of the pH-dependent component of CEC in the Caribou soil was accounted for by interlayer material and hydroxy-aluminum and/or -iron coatings. Removal of this material appeared to have no significant effect on permanent charge CEC when the latter was measured using potassium as the saturating cation.

CONCLUSIONS

1. In acid Caribou soils (below pH 5.5) a considerable portion of the cation exchange sites is occupied by hydroxy-Al and -Fe ions, and therefore, the "effective" cation exchange capacity available to hold fertilizer cations (Ca, K, Mg, NH₄) may be significantly reduced.

2. When acid Caribou soils are limed to achieve pH values in the range of 5.5 to 6.0, the effective cation exchange capacity is increased markedly (40 percent increase going from pH 5.0 to 6.0). This means that it may be necessary to apply higher rates of fertilizer nutrients such as potassium when the soil pH rises if crops are to experience the same cation balance as has been found to be most favorable in the past.

3. A significant part of the cation exchange capacity occurs in the organic fraction of a Caribou soil, with the actual value dependent upon the organic matter content (30 percent at an organic content of 3.5 percent).

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