

# CLAY DISPERSION AND HYDRAULIC CONDUCTIVITY OF CLAY-SAND MIXTURES AS AFFECTED BY THE ADDITION OF VARIOUS ANIONS<sup>1</sup>

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**Abstract**—The effect of anions on clay dispersion and the hydraulic conductivity (HC) of clay-sand mixtures has received little attention. This study investigates the effect that adding small amounts of anions has on the dispersivity and HC of reference clays. Mixtures of 3 and 6 g 100 g<sup>-1</sup> kaolinite, smectite, and illite with quartz sand were packed in columns. The columns were saturated with Ca and then leached with 1 mol<sub>c</sub> m<sup>-3</sup> of one of the following organic and inorganic Na salts: chloride, hydroxide, EDTA, silicate, citrate, formate, oxalate, hexametaphosphate, orthophosphate, tartrate, or humate. Changes in HC and clay concentration in the effluent were measured and clay dispersion was evaluated as a function of the various anions added. In the kaolinite clay-sand mixtures, a significant amount of clay was observed in the effluent for all anions tested, and the HC increased above its original value. The HC of smectite clay-sand mixtures decreased following the addition of the various anions. Dispersed clay appeared in the effluent only upon addition of citrate or hexametaphosphate. In the latter two cases, the HC started to increase once maximum clay concentration appeared in the effluent. Clay concentration in the effluent of smectite was one order of magnitude lower than that of kaolinite. Illite clay-sand mixtures showed dispersion behavior intermediate between smectite and kaolinite but behaved in the same way as kaolinite with respect to HC changes.

**Key Words**—Illite, Kaolinite, Organic anions, Smectite.

## INTRODUCTION

Many studies have indicated that soil hydraulic conductivity (HC) is related to the composition and concentration of exchangeable cations and soluble electrolytes. Quirk and Schofield (1955) showed that the HC of a soil decreased with increasing exchangeable sodium percentage (ESP) and decreasing electrolyte concentration in the soil solution. Swelling and dispersion of clay have been proposed to explain the decrease in soil HC due to the presence of monovalent exchangeable cations. Quirk and Schofield (1955), McNeal *et al.* (1966), and Russo and Bresler (1977) suggested that the swelling of clay particles which accompanies an increase in ESP could result in total or partial blockage of the conducting pores. McNeal *et al.* (1966) found a linear relationship between reduction of HC and the degree of macroscopic swelling of the extracted soil clay. Deflocculation and movement of clay into the conducting pores was proposed as the second mechanism for explaining the reduction in HC. It appears that clay dispersion is a major factor affecting the HC of soils irrigated with sodic water (Shainberg and Letey, 1984 and references cited therein). Conversely, clay

dispersion and HC reduction does not generally occur at ESP < 10 provided the electrolyte concentration of the water used is greater than 5–10 mol<sub>c</sub> m<sup>-3</sup>. The different consequences of swelling and dispersion are important to recognize: whereas a change in soil HC due to swelling is essentially a reversible process, changes due to dispersion and particle movement are irreversible (Frenkel *et al.*, 1978).

Clay swelling is not greatly affected by low ESP values (< 10–15) but it increases markedly as ESP increases above 15 (McNeal *et al.*, 1968; Oster *et al.*, 1980). Clay dispersion, on the other hand, is highly sensitive to low levels of exchangeable Na and increases sharply with a very small increase in ESP (Oster *et al.*, 1980). The intensity of this effect depends often on the type of soil and the trends observed are difficult to predict (Ali *et al.*, 1987; Yousaf *et al.*, 1987).

The effect of anions on clay swelling, dispersion, and the HC has received little attention. Shanmuganathan and Oades (1983) reported that several organic and inorganic anions increased clay dispersion in a kaolinitic soil. These investigators further noted that the extent of dispersion increased with an increase in anion sorption. Frenkel *et al.* (1992) showed that addition of small amounts of the anions to suspensions containing reference clays increased the critical flocculation concentration (CFC) of Na-montmorillonite five to six times, and that of Na-kaolinite by an order of mag-

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Table 1. Source and properties of the clays studied.

Clay source	CEC <sup>1</sup> mmol.kg <sup>-1</sup>	S <sup>2</sup> m <sup>2</sup> kg <sup>-1</sup> × 10 <sup>-3</sup>
Smectite—Upton, Wyoming (API-25)	850	775
Kaolinite—Georgia	45	41
Illite—Fithian, Illinois (API-35)	160	120

<sup>1</sup> CEC = Cation exchange capacity.

<sup>2</sup> S = Surface area, by ethylene glycol monoethyl ether (EGME) method.

nitide. Levy *et al.* (1991) added a very small amount (10 g m<sup>-3</sup>) of hexametaphosphate to a kaolinite-sand mixture and noted that it caused a sharp decrease in the kaolinite's HC.

It is conceivable, therefore, that many of the organic and inorganic anions which are either naturally present in soil or which are enriched through adding fertilizers may influence clay dispersion and the hydraulic properties of the soil. The objective of this study was to examine such deflocculating effects on the HC of columns packed with different clay-sand mixtures leached with solutions to which small amounts of various anions were added, all with a view to gaining a better understanding of the chemical and mineralogical factors affecting clay dispersion and mobility.

## MATERIALS AND METHODS

The source and properties of the three clay minerals studied are presented in Table 1. Wyoming smectite and Fithian illite were obtained from Ward's Natural Science Establishment, Rochester, New York, and the kaolinite from the Georgia Kaolin Company.

Various clay-sand mixtures (3% or 6% clay by mass) were prepared by mixing dry, powdered clay with acid-washed quartz sand (0.212–0.425 or 1–2 mm diameter). The clay-sand mixtures were wetted with distilled water and mixed again, then dried overnight at 323 K. Wetting of the clay-sand mixture bonds the clay to the sand particles (Alperovitch *et al.*, 1985) and minimizes clay and sand segregation during packing of the column. The mixture (180 g) was packed in a perspex column (52 mm internal diameter) to a bulk density of 1.5 mg m<sup>-3</sup> (60 mm depth). The pore volume of the column was calculated to be 0.045 liter. Basal support of the column consisted of a rubber stopper with a hole to accommodate an outflow tube, a screen covered with glass wool and a 10 mm layer of coarse (1–2 mm) sand.

The individual columns were wetted slowly from the bottom (in order to displace air) with a 0.25 M solution of CaCl<sub>2</sub> to ensure column saturation, and then leached with the CaCl<sub>2</sub> solution using a constant-head device (height = 50 mm). Clay saturation with Ca was accomplished by passing one liter of the CaCl<sub>2</sub> solution through the column over a 24 h period so that the total amount

of Ca applied was at least 250 times that of the clay cation exchange capacity (CEC). The purpose of clay saturation with Ca was to minimize changes in HC and/or initiation of clay dispersion resulting from the exchangeable cation. The columns were then leached with approximately 10 pore volumes of distilled water (~0.5 liter), to obtain effluent with electrical conductivity (EC) less than 0.030 dS m<sup>-1</sup>. Saturated HC was determined from continuous drainage measurements with the aid of a fraction collector. Columns prepared in this manner were thereafter leached with one of the following Na solutions (all 1 mol(Na) m<sup>-3</sup>): chloride, EDTA, hydroxide, hydroxide + chloride, silicate, monohydrogen orthophosphate, hexametaphosphate, formate, tartrate, oxalate, citrate, or humate. The latter was extracted from a Vertisol (Haplustert) near Pietermaritzburg, South Africa, with a 0.5 M NaOH solution, and neutralized with HCl to pH 5 and dialysed until free of excess salts before use.

The volume of leachate passing through the column was monitored continuously with a fraction collector. The EC and pH of each leachate fraction were measured. Clay concentration in each fraction was determined turbidimetrically using a spectrophotometer at a wave length of 530 nm. This method was calibrated using suspensions of known concentration of Na-saturated kaolinite or smectite (Felhendler *et al.*, 1974). Each leaching was performed twice and the results of duplicate runs for volume of, and clay concentration in, the leachate were found to differ by less than three percent.

## RESULTS AND DISCUSSION

The results present the relative hydraulic conductivity (RHC) or the clay concentration in the leachate as a function of the cumulative volume leached. RHC is the ratio of the HC determined during solution application, to the HC determined at the end of the distilled water leaching. Unless stated otherwise, percentage clay in the mixture was 3 g 100 g<sup>-1</sup> and the quartz sand used was 0.212–0.425 mm. It is important to note that because the clay was saturated with Ca, no change in HC in any of the clay sand mixtures was observed when distilled water replaced the 0.25 M CaCl<sub>2</sub> solution.

### A. Kaolinite-sand mixture

Of the anions studied, addition of Na-hexametaphosphate or Na-silicate had the greatest effect on the RHC and the percentage of clay leached out of the column (Figure 1), with maximum clay concentration in the leachate reaching 54 g liter<sup>-1</sup> after 0.085 liter of effluent passed through the column. A change in RHC was noticed only once maximum clay concentration in the leachate was attained. The RHC increased then to 175% and 138% for hexametaphosphate and silicate, respectively. Although maximum clay concentration was the same for the two anions, the total amount of

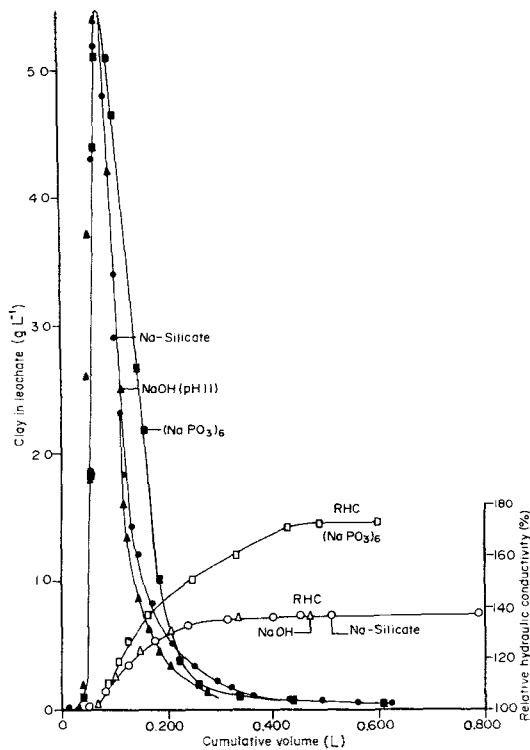


Figure 1. Relative hydraulic conductivity and clay concentration in the leachate of 3 g 100 g<sup>-1</sup> kaolinite-sand mixtures leached with Na-silicate and Na-hexametaphosphate at a concentration of 1 mol<sub>e</sub> m<sup>-3</sup>.

clay removed was larger in the hexametaphosphate treatment, a fact which probably explains why the HC in this case increased to a higher value (Figure 1).

The effect of Na silicate on the CFC of Na kaolinite was studied by Frenkel *et al.* (1992). They found that a 0.022 mol<sub>e</sub> m<sup>-3</sup> solution of Na silicate increased the CFC of kaolinite from 1.8 to 70 mol<sub>e</sub> m<sup>-3</sup> NaCl. Such an effect, however, could be attributed to the high pH associated with Na-silicate solution (the pH of a 1 mol<sub>e</sub> m<sup>-3</sup> solution was measured as 10.4). It is well known that the dispersion of kaolinite is highly pH sensitive (Schofield and Samson, 1954; Goldberg and Glaubig, 1987). Therefore, to evaluate the effect of silicate *per se*, the same experiment was conducted using different concentrations of NaOH to give five different pH values: 7.2, 8.2, 10.0, 10.5, and 11.0. To those solutions with pH below 11, the total Na concentration was adjusted to 1 mol<sub>e</sub> m<sup>-3</sup> with NaCl. From the results shown in Figure 2 it is clear that no dispersed clay was washed out at pH 7.2 and 8.2; only a small amount (<1.0 g liter<sup>-1</sup>) was observed at pH 10.0; but significant amounts were noted at pH 10.5 and 11.0, with respective maximum clay concentration in the leachate of 17 and 54 g liter<sup>-1</sup>. The dispersion at pH 10.5 can be compared with that from Na-silicate (Figure 1) which had a pH of 10.4. The comparison indicates that the presence of

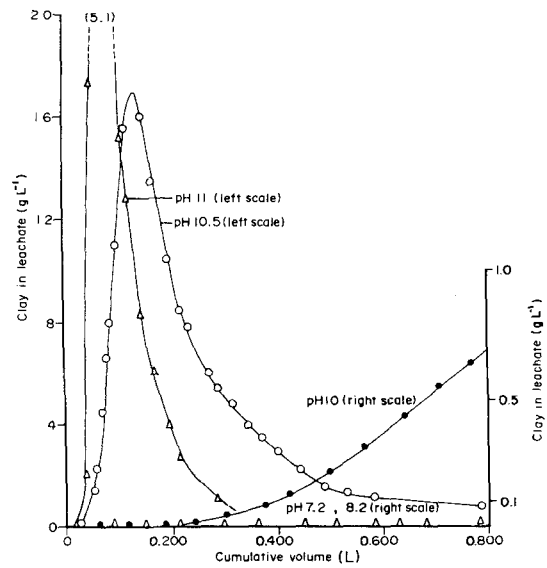


Figure 2. The effect of pH on clay concentration in the leachate of 3 g 100 g<sup>-1</sup> kaolinite-sand mixtures leached with NaOH + NaCl solution with total Na concentration of 1 mol<sub>e</sub> m<sup>-3</sup>.

silicate leads to dispersion over three times that of a NaOH solution adjusted to the same pH and Na concentration.

Addition of Na-citrate resulted also in a significant clay dispersion (Figure 3), with clay appearing in the effluent and reaching a maximum concentration of 15 and 38 g liter<sup>-1</sup> after 0.120 and 0.210 liter of solution has passed through the column for the 3 and 6 g 100 g<sup>-1</sup> clay-sand mixtures, respectively. Similar to the

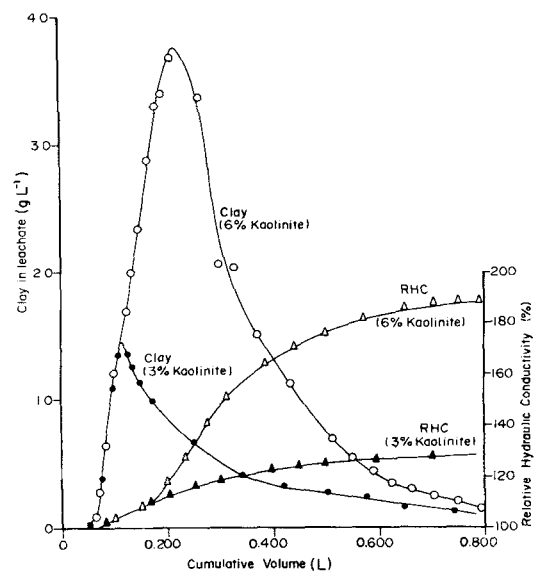


Figure 3. Relative hydraulic conductivity and clay concentration in the leachate during leaching of 3 and 6 g 100 g<sup>-1</sup> kaolinite-sand mixtures with 1 mol<sub>e</sub> m<sup>-3</sup> Na-citrate solution.

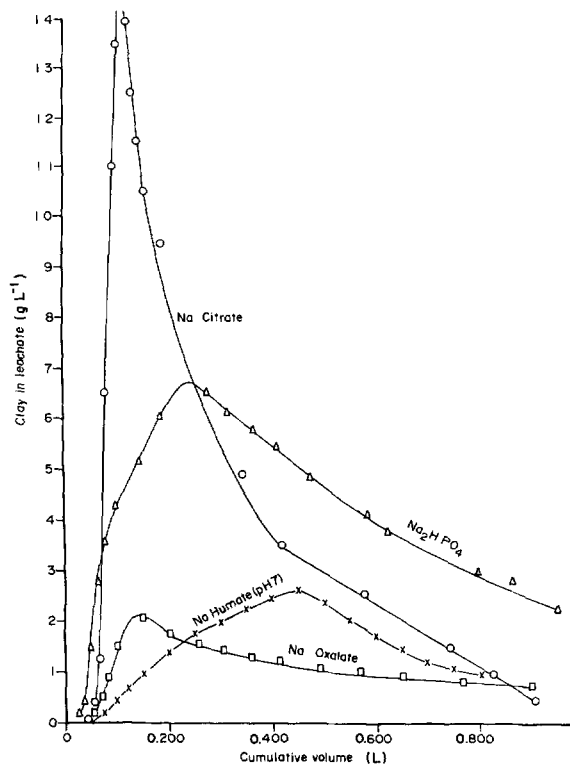


Figure 4. Clay concentration in the leachate as a function of cumulative volume of leaching solution from 3 g 100 g<sup>-1</sup> kaolinite-sand mixtures leached with 1 mol<sub>c</sub> m<sup>-3</sup> Na-citrate, orthophosphate, oxalate, and humate solutions.

hexametaphosphate and silicate treatments, no change in HC was observed until substantial amounts of clay began to appear in the leachate. Thereafter, RHC increased to 120% and 175% in the 3 and 6 g 100 g<sup>-1</sup> mixtures, respectively. The more than two times higher clay concentration in the effluent of the 6 g 100 g<sup>-1</sup> clay in the mixture compared with that of 3 g 100 g<sup>-1</sup> clay indicates that the small amount of anion added is also effective in fully dispersing a mixture rich in clay. Some dispersed clay in the leachate was also observed when the kaolinite-sand mixtures were treated with some other anions (Figure 4). Their effect, however, was much smaller than that of hexametaphosphate and silicate (Figure 1) and citrate (shown again in Figure 4).

The results indicate that kaolinite disperses easily in the presence of small concentrations of anions. Adding anions to a kaolinitic clay satisfies the positive charges on the edges of kaolinite crystals and provides them with excess negative charge (Van Olphen, 1977). Consequently, the positive edge-to-negative face mode of attraction and flocculation in kaolinite (Schofield and Samson, 1954) is eliminated. The interaction between the edges and the planar surfaces of the kaolinite particles is now controlled by the diffuse double layer forc-

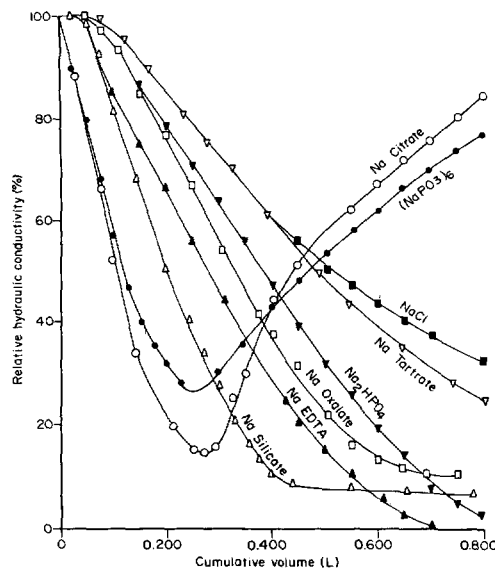


Figure 5. Relative hydraulic conductivity of Wyoming smectite-sand mixtures (3 g 100 g<sup>-1</sup> clay) as a function of cumulative leaching with 1 mol<sub>c</sub> m<sup>-3</sup> Na salt solutions.

es and depends on the type of exchangeable cation and electrolyte concentration. In our study the prevailing conditions (i.e., 1 mol<sub>c</sub> m<sup>-3</sup> of Na-anion solution) were favourable for dispersion of kaolinite. The observed difference in the dispersion of kaolinite in the presence of the various anions could be explained by the findings of Shanmuganathan and Oades (1983) who noted that the degree of dispersion increased with increasing proportion of anions adsorbed on the edges of the mineral. Anion adsorption in turn, involves electrostatic interaction as well as some chemical interaction between the surface and the ion which could be further complicated by the presence of cations like Al and Fe (Pratt, 1978).

#### B. Smectite-sand mixture

Two different patterns of changes in RHC can be distinguished for the Wyoming smectite following the addition of the anions (Figure 5). Addition of citrate or hexametaphosphate to the leaching solution resulted in a sharp reduction in RHC to 14% and 27%, respectively, after about 0.25 liter of leachate had been collected. Thereafter, RHC increased to more than 75% after 0.8 liter of leachate had passed through the column. The initial reduction in RHC in the case of the two anions was accompanied by dispersion and removal of clay in the leachate (Figure 6), with the maximum reduction in RHC coinciding with the maximum clay concentration in the leachate (3.8 g liter<sup>-1</sup> for both hexametaphosphate and citrate). There was a slight difference in the respective clay breakthrough curves, with the maximum appearing after 0.25 liter in the case of hexametaphosphate and after 0.30 liter for ci-

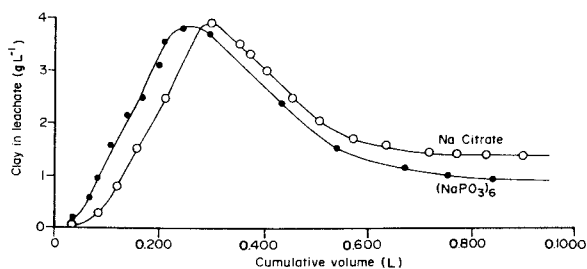


Figure 6. Clay concentration in the leachate from 3 g 100 g<sup>-1</sup> Wyoming smectite-sand mixtures as a function of cumulative volume of leachate.

trate. This difference may be related to a difference in the rate at which these two ligands are adsorbed onto the clay surfaces or to the intensity of the repulsive forces which are induced by their adsorption.

A mixture of the same clay with coarser sand (1–2 mm) treated with citrate showed a similar RHC curve to those of the kaolinite-sand mixtures treated with hexametaphosphate, silicate, and citrate: no change in HC occurred until dispersed clay appeared in the effluent, and thereafter the RHC increased to more than five-fold (Figure 7). However, unlike in the kaolinite-sand mixtures and the smectite with fine sand mixture, clay concentration in the effluent reached a maximum of 0.11% which was then maintained for the rest of the leaching. The results for both sand size ranges used appear to confirm the explanation given by Pupisky and Shainberg (1979), who suggested that changes in the mode of flow are responsible for the decrease and consequent increase in RHC. Initially (before the onset of clay dispersion), the leachate solution passes through a medium of clay-coated sand grains. As clay disperses, the flow of the solution is restricted by blockage of the pores between clay-coated sand grains by the dispersed clay particles (provided that the sand particles are sufficiently fine). Once the coating of clay itself is removed, the mode of flow changes to one of clay suspension through naked sand grains. The increased pore radii of the latter medium (i.e., naked sand grains) enhances hydraulic conductivity and allows a rapid discharge of the remaining dispersed clay. Consequently, a sharp increase in RHC, as clay purges from the mixture, is observed (Figures 5 and 7).

All the remaining ligands caused a gradual decrease in RHC to levels below 40% (Figure 5). The effectiveness of these anions in reducing HC decreased in the order: silicate > EDTA > oxalate > orthophosphate > tartrate > chloride. The decrease in RHC upon addition of NaCl is suggested to be due to the exchange of Na for Ca on the clay surfaces and the resulting contribution of the adsorbed Na to clay dispersion. Thus, the net effect of the other anions added is the difference between their RHC curves and that of NaCl. In none of these treatments however, did clay appear

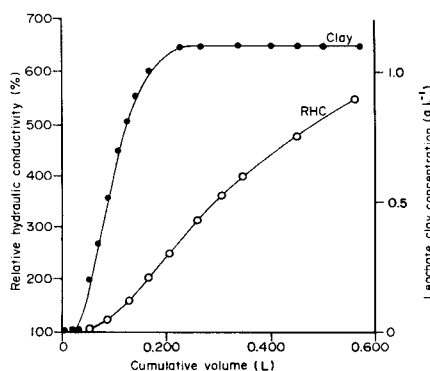


Figure 7. Relative hydraulic conductivity and clay concentration in the leachate of 3 g 100 g<sup>-1</sup> Wyoming smectite-sand mixture, in which sand particles were 1–2 mm in diameter, as a function of leaching with 1 mol<sub>c</sub> m<sup>-3</sup> Na-citrate.

in the leachate. It is suggested that short distance migration of dispersed clay particles leading to partial blockage of the pores is responsible for the observed decrease in RHC. The differences in the RHC among the various anions can be attributed partly to differences in the surface charge modification through specific adsorption and partly to the extent of complex formation with Ca, promoting the exchange of Na for Ca on the clay surface. Specific adsorption may be the less important of the two mechanisms in this case because of its restriction to edge sites on the smectite surface.

The anions studied were less effective in dispersing smectite than kaolinite. In the smectite-sand mixture, dispersed clay in the leachate was observed only upon addition of citrate or hexametaphosphate (Figure 6). On the other hand, in the case of kaolinite, dispersed clay was observed practically in all the treatments. Furthermore, maximum concentration of leached clay in the smectite was up to an order of magnitude smaller than that found for kaolinite-sand mixtures, 4.0 and 54 g liter<sup>-1</sup>, respectively.

The observed difference in the effect of citrate on smectite and kaolinite in the current study is consistent with its effect on the CFC of these clays (Frenkel *et al.*, 1992). In the latter study, kaolinite was found to be more sensitive than smectite to dispersion in the presence of organic ligands, probably due to the higher ligand sorption capacity per unit surface of the kaolinite. Thus 1 mol<sub>c</sub> m<sup>-3</sup> of Na citrate in the solution increased the CFC of Na-montmorillonite from 16 to 56 mol<sub>c</sub> m<sup>-3</sup> NaCl and the CFC of Na-kaolinite from 1.8 to 23 mol<sub>c</sub> m<sup>-3</sup>. The results in Figure 2 indicate that even a small addition of Na-citrate to Ca-kaolinite causes significant dispersion of the kaolinite.

The contrast in trends in HC of smectite- and kaolinite-sand mixtures in the treatments where clay was leached out of the columns could be attributed to swelling of smectite. Swelling of smectite in the column may

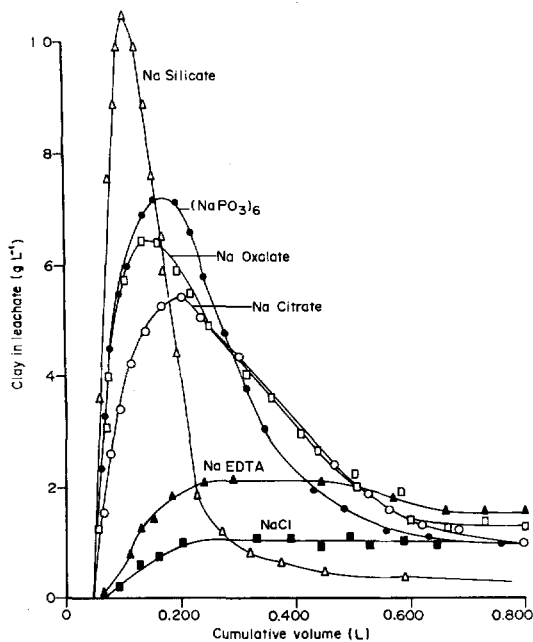


Figure 8. Clay concentration in the leachate as a function of cumulative volume of leaching solution from 3 g 100 g<sup>-1</sup> illite-sand mixtures leached with 1 mol<sub>c</sub> m<sup>-3</sup> Na-silicate, hexametaphosphate, citrate, EDTA, and chloride solutions.

have initially induced reduction in the radii of pores, decreasing HC until such time as dispersion became sufficient to purge the clay, unblock pores, and regenerate high HC (Figure 5). However, swelling is very limited when the ESP of smectite is below 15 (McNeal and Coleman, 1966). In our study the clay was saturated with Ca before it was leached with a 1 mol<sub>c</sub> m<sup>-3</sup> Na salt. In order to obtain a clay with ESP 10 then the 3 g 100 g<sup>-1</sup> smectite-sand mixture should be leached with at least 0.460 liter (assuming a complete 1:1 exchange ratio between Na and adsorbed Ca, and the cation exchange capacity of the smectite to be 0.85 mol<sub>c</sub> kg<sup>-1</sup>). For both hexametaphosphate and citrate treatments, the decrease in RHC occurred during leaching with about 0.250 liter of solution (Figure 5), at which stage the ESP would be <5. It is suggested that the different trends in HC in the two clays result from the different sizes of the dispersed particles. Kaolinitic tactoids are bigger than smectitic ones. Thus, removal of dispersed kaolinitic tactoids from the clay-coated sand grains will have a significant effect on pore radii and allow quick purging of clay from the column with a resulting increase in the HC. Conversely, in smectite, the removal of dispersed tactoids from the clay-coated sand will not have initially a large effect on pore radii and the dispersed particles will migrate for a short distance and partially clog the pores leading to a decreased HC. Only once enough clay was leached out of the column will the pore radii increase sufficiently to allow an increase

in HC. No swelling occurred in the kaolinitic sand, thus allowing the effect of dispersion and purging of the dispersed clay particles to appear immediately as a change in the HC.

### C. Illite-sand mixture

Illite-sand mixtures respond to the addition of the anions in much the same way as kaolinite-sand mixtures. Significant amounts of dispersed clay particles have generally been observed after 0.15–0.20 liter passed through the columns (Figure 8). The HC increased immediately (data not presented) without the initial decrease exhibited by smectitic sand. The effect of most of the anions on clay dispersion in illite was intermediate between that found for kaolinite and smectite. This may result from the fact that the ratio of edge surface area to planar surface area in illite is smaller than in kaolinite, but larger than in smectite. A particular feature of illite behaviour was that even NaCl and Na-EDTA caused some dispersion. This result conforms, however, with the reportedly very high sensitivity of illite to even small amounts of exchangeable Na (Oster *et al.*, 1980). These authors found that, for a given ESP, the CFC of illite was much larger than for smectite, and hypothesized that the explanation lies in the irregularities of illite particle shapes, which prevent good contact between edges and planar surfaces, reducing the potential for inter-particle attraction. An alternative explanation might be that illite contains organic matter. This was first suggested by the dark colour of the Fithian illite, which was found upon subsequent analysis to have 0.93 g 100 g<sup>-1</sup> total C and 0.33 g 100 g<sup>-1</sup> total N. Assuming that this organic matter occurs as humic substances, it may well have enhanced the dispersibility of the illite. The deleterious effect of adsorbed humate on clay dispersion has been demonstrated in this paper (Figure 4) and elsewhere (Shanmuganathan and Oades, 1983; Frenkel *et al.*, 1992).

## CONCLUSIONS

Of the clays studied, kaolinite is the most sensitive to dispersion in the presence of small amounts of anions, probably because its highest ratio of positively charged edge surface to negatively charged planar surface results in a high anion adsorption capacity. The significant amounts of dispersed kaolinite that are purged out of the columns result in an immediate and rapid increase in HC. In the case of smectite, HC decreases through partial blocking of pores by short distance migration of dispersed particles (provided the sand particle size is sufficiently fine to retain these particles). Measurable clay dispersion took place only in the presence of the most powerful of the dispersants, enabling clay to be purged from the column so that hydraulic conductivity is regenerated. Illite generally shows dispersion behaviour intermediate between that

of kaolinite and smectite, but changes in HC are similar to those observed for kaolinite. The high degree of exchangeable Na-related dispersion shown by the illite (i.e., not caused by anion adsorption) may be related to an organic contaminant associated with the mineral which diminishes the potential for edge-to-face attraction.

Some of the anionic dispersants studied have special relevance to soil environments either naturally or through the likelihood of their addition to soil with chemical amendments. The strength of their dispersive effect can sometimes be greater than the differences in behaviour between groups of clay minerals. Such information could be helpful in understanding mineralogical anomalies which are often apparent in the infiltration rates and hydraulic conductivities observed for different soils. It should also find application in the study of soil genesis for modifying theories of clay migration in the soil profile.

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