

Comparison of the Titration and Ion Adsorption Methods for Surface Charge Measurement in Oxisols

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ABSTRACT

The pH-dependent charge properties of the A and B horizons of two representative oxisols from the Cerrado Region, Brazil, were studied by a comparison of the potentiometric titration method of net charge measurement and direct measurement of anion and cation exchange capacity from K^+ and Cl^- adsorption. Most soil samples bore net negative charge at their native pH, but variability in the magnitude of this charge was attributed to the effect of Al-blocked exchange sites or contributions from strongly acidic organic functional groups, since these soils had similar mineralogies. The soils also bore significant pH-dependent positive charge at their natural acidic pH values, which was more evident in the B horizons than the surface horizons. Estimates of net surface charge by potentiometric titration and ion adsorption did not agree, especially as the soil pH was adjusted away from the point of zero charge. This lack of agreement is attributed to dissolution reactions of minerals and organic matter at high and low pH, which consume acid or base and overestimate surface charge.

THE TWO MOST COMMON methods of studying the charge characteristics of amphoteric surfaces are indirect (potentiometric titration) and direct measurement of anion and cation retention as a function of pH (Parks and de Bruyn, 1962; Schofield, 1949). Potentiometric titration involves the measurement of the net adsorption of H^+ or OH^- at different pH and at different ionic strengths. Because of the characteristics of amphoteric surfaces, there is a pH at which the different curves intersect, i.e., a point at which adsorption of protons is independent of ionic strength. This pH is termed the point of zero salt effect (PZSE), (Parfitt, 1980; Parker et al., 1979).

The second method, ion adsorption, involves the measurement of retention of an indifferent ion (adsorbed onto the surfaces by electrostatic attraction), such as Na^+ , K^+ , NO_3^- , and Cl^- as a function of pH at a fixed ionic strength (Schofield, 1949). This method provides an estimate of the cation exchange capacity (CEC) and anion exchange capacity (AEC), as well as the net charge (AEC minus CEC) at each pH, and is considered to measure point of zero net charge (PZNC).

In general, the A horizons of soils tend to have lower PZNC than their corresponding B horizons.

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This is attributed to the greater negative charge in the upper horizons because of the higher organic matter content (Van Raij and Peech, 1972). Direct comparison of PZNC values from different soils cannot generally be done because of the dependence of the measurement on the ionic strength of the solution in which the AEC and CEC were measured. For example, Gallez et al. (1976) and Morais et al. (1976) showed a decrease in PZNC values of highly weathered soils with increasing ionic strength. The PZNC and PZSE values in soils are very low compared to those of pure oxides, presumably because soils generally contain organic matter, permanently charged materials, and SiO^- groups, all of which increase the negative charge of the soil, lowering apparent points of zero charge (Parfitt, 1980).

Recent investigations of charge development on Al oxide as a function of pH have demonstrated that dissolution can introduce a large error into the estimates of surface charge by the potentiometric titration method (Schulthess and Sparks, 1987). A similar error is likely to arise in soils dominated by oxide mineralogy, although the degree to which this occurs is probably sensitive to the crystallinity of the Al and Fe oxides present.

Indeed, Parker et al. (1979) have discussed the pitfalls inherent in measuring net charge in soils by potentiometric titration. The present investigation was undertaken to compare the potentiometric titration and ion adsorption methods for determining pH-dependent surface charge of Oxisols.

MATERIALS AND METHODS

Soil Characteristics

For this study, samples of the A and Bo horizons of two Oxisols from the Cerrado Region, Brazil, were collected by J. Macedo from two representative soil profiles (Macedo and Bryant, 1987). Both soils are classified as Typic Acrustox in the *Soil Taxonomy* system, and as Dark Red Latosol and Red Yellow Latosol in the *Brazilian Classification* scheme (Macedo and Bryant, 1987). The samples studied correspond to the A and Bo1 horizons of the Dark Red Latosol (site A) and the Ap and Bo1 horizons of the Red Yellow Latosol (site H) in Macedo and Bryant (1987). In this study the soil samples will be referred to as Dark Red Acrustox (DRA and DRB for the A and Bo horizons, respectively) and Red Yellow Acrustox (RYA and RYB).

Some of the chemical and physical characteristics of the soil samples are listed in Table 1. Soil pH was measured in a 1:10 soil/solution ration (in order to maintain the same ratio as that used for the ion adsorption measurements) us-

ing a combination glass-AgCl electrode. Organic C was determined on a Model 240C Perkin-Elmer elemental analyzer (Perkin-Elmer Corp., Norwalk, CT). Free Fe oxides were extracted by the CBD method (0.3 M sodium citrate, 1 M sodium bicarbonate, and dithionite) of Mehra and Jackson (1960). Organically bound Fe and Al were extracted with a 0.1 M solution of sodium pyrophosphate using a soil/solution ratio of 1:100 (USDA-SCS, 1984). Both elements were determined by atomic absorption spectrophotometry.

All four soil samples belong to the clay textural classification (Macedo and Bryant, 1987). The mineralogical compositions were qualitatively determined for this study by x-ray diffraction (XRD) on a Norelco x-ray spectrophotometer (Phillips Electronic Instruments, Mount Vernon, NY) (Macedo and Bryant, 1987) and by infrared spectroscopy (IR) using a Perkin-Elmer Model 281 infrared spectrophotometer, revealing the same mineralogy for the A and Bo horizons within the same soil profile. The clay fraction, separated by procedures detailed by Macedo and Bryant (1987) consisted mostly of kaolinite and gibbsite in all four samples, based on the intensities of XRD and IR peaks. The Dark Red Acrustox also contained goethite and hematite, while the only Fe oxide in the Red Yellow Acrustox was goethite. The latter soil sample contained relatively more kaolinite, gibbsite, and goethite. The sand and silt fractions contained, in addition to the previous minerals, quartz and traces of maghemite. Surface areas of the soil samples, measured by a three point Brunauer-Emmitt-Teller (B.E.T.) isotherm for N₂ adsorption at -196 °C on a Quantasorb sorptometer (Quantachrome, Syosset, NY), are reported in Table 1.

For this study, the soil samples were air-dried and screened through a 0.7-mm sieve prior to use; the results are expressed on an oven-dry weight basis.

Charge Characterization

Ion Adsorption Method

An estimate of the CEC and AEC as a function of pH was determined by measuring the amount of K⁺ and Cl⁻ retained by the soils at different pH using a modification of Schofield's method (Schofield, 1949). Based upon the known mineralogy of these soils, it was assumed that the clays contained essentially no sites capable of specifically absorbing K⁺, and therefore that KCl could be treated as an indifferent electrolyte. Triplicate 2-g samples of soil were weighed in centrifuge tubes and washed with 0.1 M KCl to minimize soluble Al; after discarding the supernatants, 20 mL of the same solution were then added and the pH adjusted with KOH or HCl to give a pH range between 2 and 8. The samples were equilibrated at room temperature (24 ± 2 °C) by shaking intermittently on a reciprocal shaker for 12 h. Then the samples were centrifuged, the supernatants dis-

carded, and 20 mL 0.01 M KCl added; this 0.01 M KCl wash was repeated two more times. After the final washing, the supernatant pH was measured as well as the Cl⁻, K⁺, and Al concentrations. Next, the adsorbed K⁺ and Cl⁻ ions were displaced by washing the soil with 0.5 M NH₄NO₃. The amounts of K⁺ and Cl⁻ displaced, after correction for the entrained KCl within the soil volume, were used as estimates of the negative and positive charges, respectively. Chloride was measured using an Orion specific ion electrode (Orion Research, Cambridge, MA) with a double-junction reference electrode filled with 100 g kg⁻¹ KNO₃ solution in the outer chamber, and K⁺ by atomic absorption.

Potentiometric Titrations

The methodology of Van Raij and Peech (1972) was used. Soil samples weighing 1.5 g were suspended in 15 mL of 0.1, 0.01, and 0.001 M KCl, and the pH adjusted to a range between 3 and 8 with measured amounts of HCl or KOH. The suspensions were kept at room temperature (24 ± 2 °C) in capped 20-mL plastic vials and shaken twice daily over a 3-d period. After this time the pH values of the supernatants were recorded. The amounts of H⁺ and OH⁻ adsorbed by the soil samples were estimated from the amount of acid or base necessary to bring the samples to the final equilibrium pH, minus the amount necessary to bring 15 mL of a blank solution (KCl solution without soil) to the same pH.

Statistical Analysis

The statistical analysis was done with MINITAB™ on an IBM Personal Computer. Each experiment was treated as a completely randomized design. Because the experiments were performed individually on each soil, comparisons of surface charge of the soils as a function of pH were accomplished by the use of linear and nonlinear regression analysis. The confidence intervals of the regression lines were used to determine statistical significance of any differences in the surface charge measurements.

RESULTS AND DISCUSSION

Charge Characteristics

Determination of Surface Charge by Ion Adsorption

The ion adsorption method allows the determination of positive and negative electric charge, as well as the net charge, as a function of pH. Figure 1 and 2 show the surface charge as a function of pH determined by the adsorption of K⁺ and Cl⁻ at an ionic strength of 0.01 M for the four soil samples. Anion exchange capacity values ranged from around 25 mmol_c/kg at pH 3.3 in both B horizon samples to -0.2 mmol_c/kg in the RYA at pH 6.6. Cation exchange capacity values ranged from around 2 mmol_c/kg in the RYB at pH 3.7 up to around 40 mmol_c/kg at a pH near 7 in DRA, DRB, and RYA. The values reported here are higher than those reported by Van Raij and Peech (1972) for an Acrorthox B2 at 0.01 M ionic strength, but are within the same range of values reported by Gallez et al. (1976) for two Ultisols and an Alfisol at the same ionic strength. Other similar studies in the literature utilized ionic strength of 0.05 M or higher and the reported AEC and CEC are usually greater in magnitude than the ones shown here.

Cation exchange capacity tends to be higher in A horizons than in B horizons of highly weathered soils,

Table 1. Chemical and physical characteristics of the soils.

Soil	pH (H ₂ O)	pH (0.01 M KCl)	Organic C†	CBD Fe	
				g/kg	
DRA	4.68	4.32	28.2	78.2	
DRB	4.98	4.78	09.6	53.7	
RYA	4.61	4.20	23.9	40.0	
RYB	4.43	4.66	08.6	37.3	
Pyrophosphate extraction					
	Fe		Clay content†	Surface area	
	Al				
	mmol/kg		g/kg	m ² /g	
DRA	69.64	91.57	594	43.5	
DRB	2.49	16.49	649	45.4	
RYA	19.15	41.52	703	43.9	
RYB	1.67	16.49	649	48.9	

† Data from Macedo and Bryant (1987).

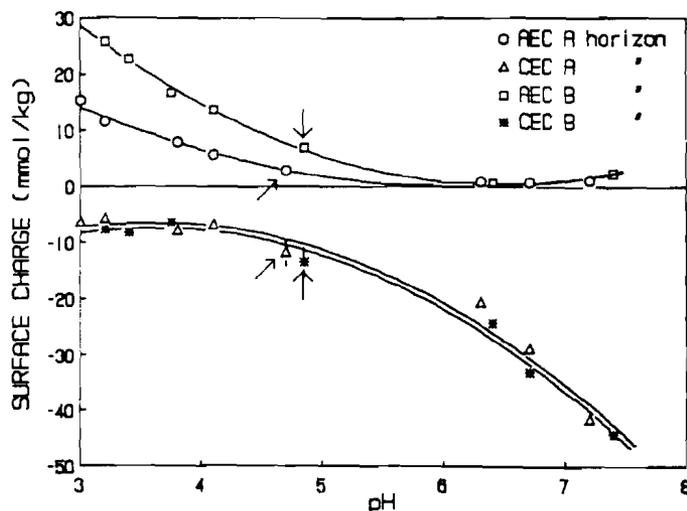


Fig. 1. Variation of the surface charge with pH in the Dark Red Acrustox. Arrows refer to the CEC and AEC of the soils at their natural pHs. Confidence intervals (95%) are occluded by the symbols in both AEC lines.

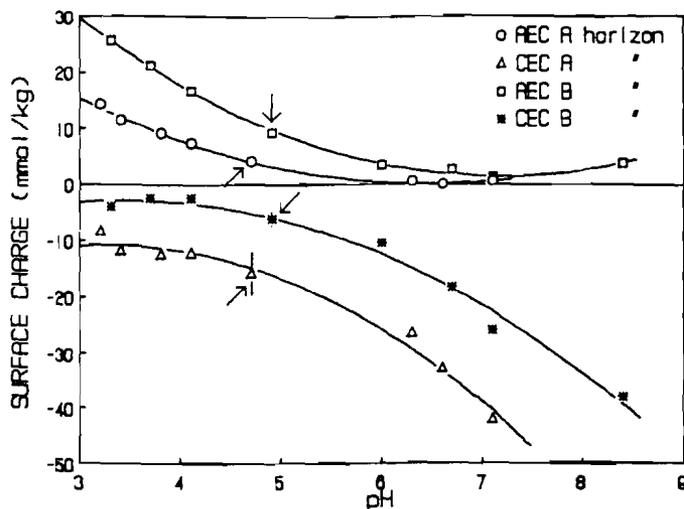


Fig. 2. Variation of the surface charge with pH in the Red Yellow Acrustox. Arrows refer to the CEC and AEC of the soils at their natural pHs. Confidence intervals (95%) are occluded by the symbols in both AEC lines.

presumably an effect of organic matter content (Morais et al., 1976; Van Raij and Peech, 1972). This was observed in the Red Yellow Acrustox where the CEC in the A horizon sample was consistently higher in magnitude than in the B horizon (Fig. 2), but this trend did not occur in the Dark Red Acrustox where the CEC values of the A and B horizon samples were not significantly different ($P < 0.05$) (Fig. 1). Blocking of the exchange sites of organic matter by Al ions could explain this phenomenon. Aluminum complexed with organic matter is not easily removed by ions such as K^+ at low ionic strengths (Bloom et al., 1979). The higher concentration of Al and Fe removed by pyrophosphate (organically bound Al and Fe) in the A horizons might support this hypothesis (Table 1); the level of organically bound Fe + Al in the A horizon sample relative to that in the B was higher in the Dark Red than in the Red Yellow Acrustox.

For both soil profiles, the AEC was higher in the B horizon sample than in the A horizon sample. Because the mineralogical composition is the same for both horizons of the same soil, this can be attributed to the difference in organic matter. Organic groups (e.g., carboxylic, phenolic) are known to displace water ligands at positive sites on the oxide surfaces (Parfitt, 1978; McBride and Wesseling, 1988), which may reduce the AEC.

At pH 6.5, the positive charge in all soil samples except the RYB was virtually zero. The lowest AEC value in the RYB was 1.4 ± 0.7 mmol/kg at pH 7.1, although this could represent a systematic error in the determination of Cl^- adsorption by this soil. Even if this Cl^- adsorption near pH 7 is real, it does not in our opinion prove the existence of permanent positive charge in the soil, as suggested by others (Tessens and Zauyah, 1982; Sposito, 1983). It seems more reasonable to suggest that exposed $M-OH_2$ groups (not neutralized by anion adsorption) can exist at pH 7, keeping in mind that the ZPC of "clean" Fe oxide surfaces can be well above 8.

In the lower pH range (<4), the CEC in all four soil samples tended to approach constant values. Estimates of this pH-independent charge are 6.8 ± 2.1 and 7.3 ± 2.6 mmol/kg for DRA and DRB, respectively, and 11.0 ± 3.8 and 2.9 ± 1.2 mmol/kg for RYA and RYB, respectively. The charge could originate in the kaolinite; this is the only mineral present that is known to generate surface charge by isomorphous substitution. Kaolinite has been shown to bear a small permanent charge from about 14 to 36 mmol/kg clay (Bolland et al., 1976) and the estimated 30% kaolinite content of this soil (Macedo and Bryant, 1987) could then account for 4 to 11 mmol/kg of CEC. The large difference in low-pH CEC between the RYA and RYB, however, suggests that exchange sites on kaolinite may be blocked by Al in the B horizon, or that organic matter in the A horizon may contain some strongly acidic groups that contribute to this low-pH charge. Adsorbed Al is known to block exchange sites on clays, reducing apparent CEC (Hodges and Zelazny, 1983; McBride, 1978). The much higher pyrophosphate extractable (Fe + Al) in the DRA than in the RYA suggests that most of the organic charge sites are blocked by complexation in the DRA, accounting for the similar CEC values in the DRA and DRB.

The PZNC values were higher in both B horizon samples than in the A horizon samples as shown in Table 2. This has been attributed to the negative charge arising from the greater organic matter content

Table 2. Points of zero net charge (PZNC) and net charge as determined by ion adsorption method. Point of zero salt effect (PZSE) as determined by potentiometric titration method.

Soil	PZNC	PZSE	Net charge at natural soil pH, mmol/kg
DRA	3.86	4.34	-9.14 ± 2.16
DRB	4.45	5.25	-6.56 ± 2.53
RYA	3.59	4.25	-11.5 ± 3.7
RYB	5.26	5.18	3.17 ± 1.45

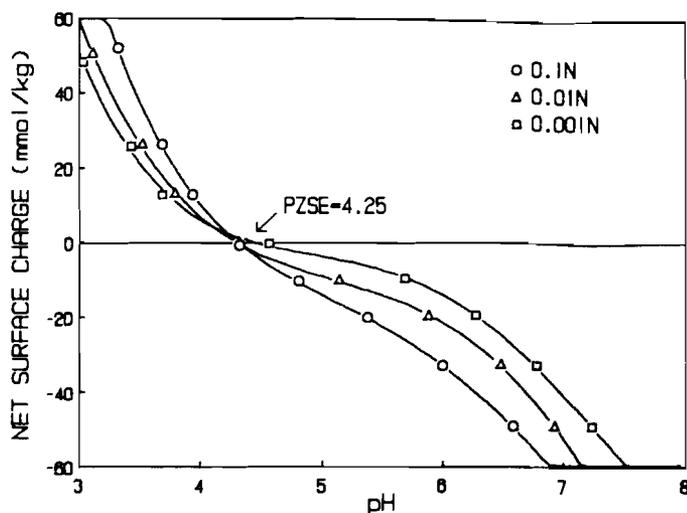


Fig. 3. Estimation of surface charge in RYA by potentiometric titrations at three electrolyte concentrations.

in the upper horizons (Morais et al., 1976; Parfitt, 1980; Van Raij and Peech, 1972). Again, the fact that the PZNC values of the A and B horizon samples were more different for the Red Yellow than the Dark Red soil suggest the presence of dissociated (highly acidic) organic functional groups or greater "unblocked" kaolinite exchange sites in the Red Yellow A horizon soil.

The net charge of the soil is estimated by subtracting the CEC value at a given pH from the corresponding AEC value; these values are shown in Table 2 at the soils' natural pH along with the calculated PZNC values.

Potentiometric Titrations

Potentiometric titration curves describe net adsorption and desorption of H^+ as a function of pH and ionic strength. If surface charge were due only to the dissociation of hydroxylated (variable charge) surfaces, and if all proton consumption/release reactions altered the surface charge, these titrations would be estimates of the variation of net charge with pH and electrolyte concentration. The potentiometric titrations for RYA and RYB are shown in Fig. 3 and 4 [DRA and DRB curves (not shown) were similar to RYA and RYB, respectively].

The titration curves at different ionic strengths intersect at a common point that is the PZSE; at this pH the net charge is independent of ionic strength. The PZSEs are listed in Table 2 along with the PZNC determined for the same soil samples. For both A horizon samples, the PZSEs occurred very close to the native pH, while for the B horizons the PZSEs occurred on the alkaline side of the native pH (Fig. 5 and 6).

The lower PZSE values in the A horizon samples reflect the effect of higher organic matter (Gallez et al., 1976; Keng and Uehara, 1974; Van Raij and Peech, 1972). Samples from corresponding horizons of the Dark Red and Red Yellow Acrustox showed very similar PZSE values (Table 2), suggesting a lower sensi-

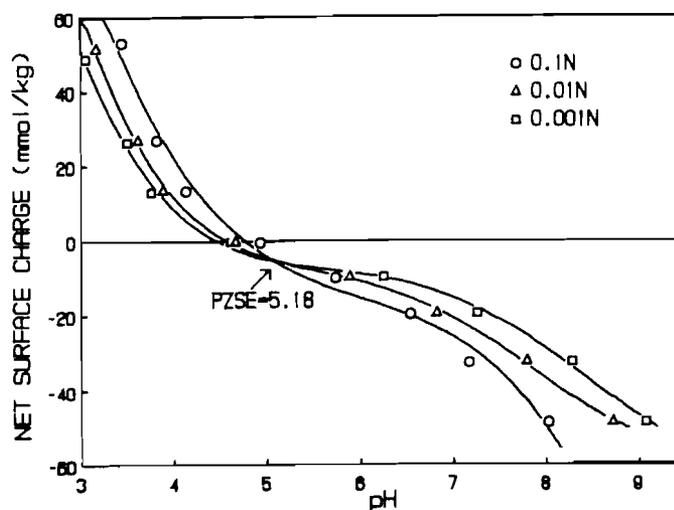


Fig. 4. Estimation of surface charge in RYB by potentiometric titrations at three electrolyte concentrations.

tivity of PZSE (compared to PZNC) to significant differences in surface chemistry.

Comparison of Titration and Ion Adsorption Methods

In the DRA, DRB, and RYA the PZNC values (determined by adsorption of ions) were lower than the PZSE (determined by potentiometric titration) as seen in Table 2. The PZNC is usually lower than PZSE (Magalhaes and Page, 1984; Parfitt, 1980; Van Raij and Peech, 1972), a fact that can be attributed to the presence of net permanent negative charge in the soil (Sposito, 1983). In Fig. 5 and 6, net charge determined by the adsorption and titration methods are compared. Potentiometric titrations appear to have overestimated the magnitude of net charge at low and high pH values, especially in both A horizons. One likely source of this error is the dissolution of oxide minerals at low pH, consuming protons without creating surface positive charge, i.e., overestimating the adsorption of protons (Schulthess and Sparks, 1986, 1987). Similarly, high pH dissolution of minerals and organic matter consumes OH^- without creating surface negative charge. Nevertheless, between the pH of the PZNC and pH 6.5, there was fair agreement between the two methods of charge measurement in all soils. Laverdiere and Weaver (1977) have reported good agreement between the methods for an Haplorthod, but this is not commonly the case (Parfitt, 1980). Since the adsorption method measures both permanent and variable charge, while the titration method is purported to measure only variable charge, agreement between the two methods would require low permanent charge.

According to Sposito (1983) and Uehara and Gillman (1980), an estimate of the permanent charge can be obtained from the difference between the AEC and CEC at the PZSE determined by titration. At this pH, the variable charge, σ_v , is zero and any residual net charge must be due to the permanent component σ_p . By this method, the DRA, DRB, and RYA soil sam-

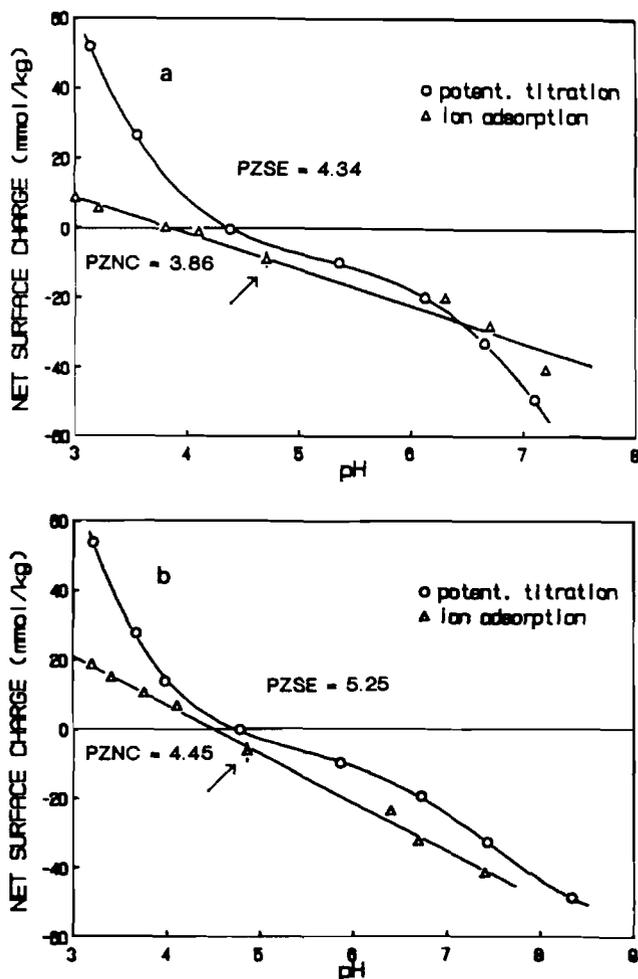


Fig. 5. Comparison of the estimation of the net surface charge by the ion adsorption and potentiometric titration methods. (a) net charge in DRA; (b) net charge in DRB. Arrows indicate the net charge of the soils at their natural pHs. In some points 95% confidence limits are smaller than the symbols.

ples had 4.4, 11.3, and 7.3 mmol/kg of permanent negative charge, respectively, while the RYB had 0.7 mmol/kg of permanent positive charge. Although the accuracy of this method is likely to be compromised by the presence of adsorbed Al, the low permanent charge of these soils is demonstrated.

CONCLUSIONS

The net charge of these oxisols (with the exception of RYB) estimated by the ion adsorption method, was negative at the natural soil pH. The soil samples possessed between 3 and 11 mmol/kg of negative charge at acidic pH (<4), probably originating from kaolinite and highly acidic organic functional groups. The samples of both oxisols also possessed significant anion exchange capacity (positive charge) at natural pH; this was particularly evident in the B horizons, suggesting that positive charge sites in the surface horizons were chemically blocked by organic anions. The natural positive charge has important implications for NO_3^- retention in these soils.

The estimates of the soil ZPC varied with the method used. The PZSE was usually higher than the PZNC, a likely consequence of permanent negative

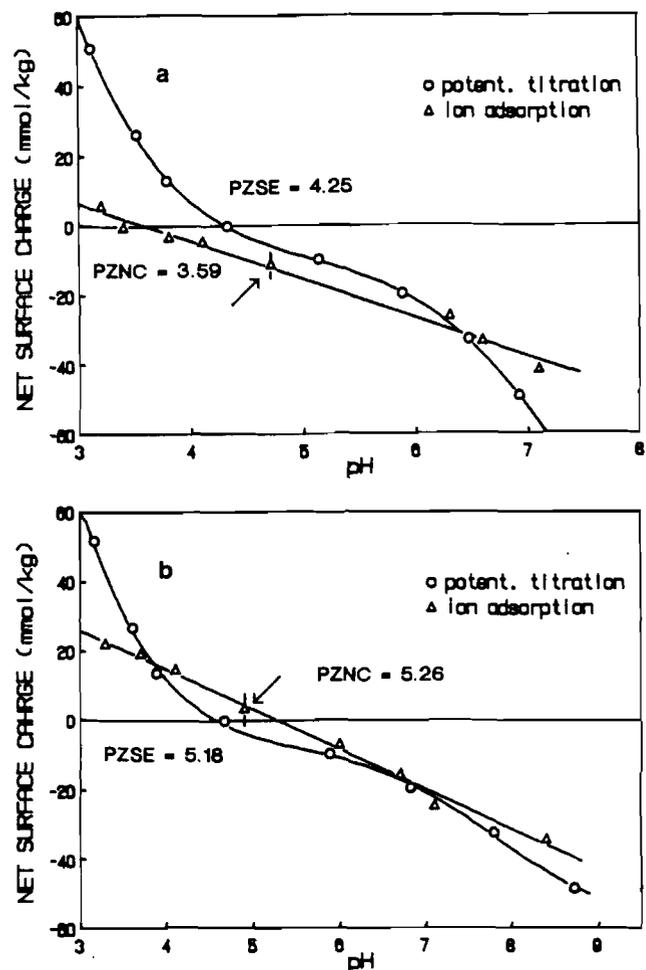


Fig. 6. Comparison of the estimation of the net surface charge by the ion adsorption and potentiometric titration methods. (a) net charge in RYA; (b) net charge in RYB. Arrows indicate the net charge of the soils at their natural pHs. In some points 95% confidence limits are smaller than the symbols.

charge in the soil. The fact that $\text{PZNC} > \text{PZSE}$ in the RYB suggests low available permanent charge sites in this soil. Possibly, Al blocked kaolinite exchange sites in this soil, as the A and B horizons were mineralogically indistinguishable. In the A horizon, Al may be complexed by organic matter to a greater extent, suppressing adsorption on kaolinite.

The ion adsorption method showed net charge to be a nearly linear function of pH, a result inconsistent with prediction of oxide surface behavior from diffuse double layer theory. The presence of numerous types of surface, each with its own characteristic ZPC, could possibly generate a more continuous effect of pH on charge than is predicted by models for single surfaces.

This study supports the view that estimation of charge by the ion adsorption method is more reliable than potentiometric titration because it avoids the error of overestimating variable charge resulting from dissolution reactions. This may be a severe problem only at high and low pH values (below 4, above 7). In the intermediate pH range, the two methods agreed fairly closely, with the difference in charge estimated by the two methods attributed to permanent charge in the soil.

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