

APPARENT ECEC IN SOME BRAZILIAN SOILS WITH VARIABLE CHARGES DETERMINED USING THREE DIFFERENT EXTRACTORS

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Abstract

This is a study of the relationships among three different extractors for the determination of apparent ECEC values in 40 soil samples with dominant variable charges selected from 25 soil pedons. In the first method (Serviço Nacional de Levantamento e Conservação de Solos [SNLCS]), N KCl was used to extract calcium, magnesium, and extractable acidity, and North Carolina extractor was used for sodium and potassium. The second method used NH₄OAc for calcium, magnesium, sodium, and potassium and the same values of N KCl for extractable acidity. The third one used unbuffered dilute silver thiourea (AgTU) solution (0.01M Ag⁺) for measuring exchangeable bases and extractable acidity. The apparent ECEC was obtained by the sum of all cations expressed in meq/100 g soil or meq/100 g clay. Potassium and sodium were measured by flame photometry, calcium and magnesium by atomic absorption spectro photometry, and extractable acidity by volumetric titration with dilute NaOH using phenolphthalein as indicator. All soil samples used satisfied the criteria for low activity clay: apparent ECEC \leq 12 meq/100 g clay. The apparent ECEC values are highly correlated when referred to 100 g of soil or 100 g of clay. According to statistical analysis and tests of paired observations, AgTU extractor is significantly different from the others.

Determination of exchangeable cations and apparent ECEC in soils with variable charge is widely used in characterizing weathered soils of tropical regions.

The usual methods for determination of CEC extract exchangeable bases with neutral ammonium acetate pH = 7 and extractable acidity with unbuffered potassium chloride. The sum of all cations determined in such a manner is called apparent ECEC (Kamprath 1970 cited in Pleysier and Juo 1980).

Methods using NH₄OAc (Chapman 1965a, b) and BaCl₂TEA (Peech 1965) often overestimate values of CEC of highly weathered soils with reference to their field conditions (Juo, Ayanlaja, and Ogunwale 1976; Gillmann and Bell 1978).

Pleysier and Cremers (1973, 1975) have shown that Ag-thiourea cation has a much stronger affinity for soil colloids than cations commonly occurring in soils so AgTU is a highly effective cation in displacing exchangeable cations in soils and clays at relative low ionic strength. Chhabra, Pleysier, and Cremers (1975) described a method using Ag thiourea for determination of CEC. Pleysier and Juo (1980) developed a single-extraction procedure using an unbuffered dilute Ag-thiourea solution to determine the exchangeable cations and effective CEC in soils with dominant variable charges.

The present paper compares data of exchangeable bases, extractable acidity, and apparent ECEC using three different extractors: Normal ammonium acetate pH = 7, normal potassium chloride, and Ag thiourea comprising 40 samples of high by weathered soils from Brazil.

Materials and Methods

Forty soil samples of B horizons comprising low activity clay soils from Brazil were selected (Table 1).

Methods used in the determination of exchangeable bases, extractable acidity, and apparent ECEC are described in *Manual de Métodos de Análise de Solo* (Empresa Brasileira de Pesquisa Agropecuária [EMBRAPA] 1979); Chapman (1965a, b); Yuan (1959); and Pleysier and Juo (1980). The apparent ECEC was considered as the sum of cations Ca, Mg, Na, K, and extractable acidity. The procedure described in the *Manual de Métodos* (EMBRAPA 1979) uses normal CaOAc pH = 7 in the determination of extractable acidity but, in the present study, normal KCl is employed for this extraction (Yuan 1959).

Soil extractor ratio is the same for all three methods: 5 g soil/50 ml extractor. Time of shaking is 5 min for SNLCS and NH₄OAc and 120 min for AgTU. In all procedures soil samples and extractors remained in contact overnight.

Analytical data relative to chemical and physical properties of the soil samples used are described in Congresso Brasileiro de Ciência do Solo (1979); Reunião de Classificação, Correlação de Solos e Interpretação de Aptidão Agrícola (1984); and VIIth International Soil Classification Workshop (1986).

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Table 1. Soil Selected Pedons

| | Soil classification |
|-----------|--|
| ISCW 1 | Humic Akrudox; fine, kaolinitic, isothermic |
| ISCW 2 | Humic Akrudox; very fine, allic, isothermic |
| ISCW 3 | Rhodic Hapludox; fine-loamy, kaolinitic, isohyperthermic |
| ISCW 4 | Rhodic Akrudox; very fine, kaolinitic, isohyperthermic |
| ISCW 5 | Typic Akrudox; very fine, kaolinitic, isohyperthermic |
| ISCW 6 | Typic Eutrudox; very fine, kaolinitic, isohyperthermic |
| ISCW 7 | Humic Rhodic Akrudox; very fine, ferritic-gibbsitic, isohyperthermic |
| ISCW 8 | Rhodic Eutrudox; very fine, ferritic, isohyperthermic |
| ISCW 9 | Humic Akrudox; fine, kaolinitic, isohyperthermic |
| ISCW 10 | Typic Akrudox; fine-loamy, kaolinitic, isohyperthermic |
| ISCW 11 | Typic Akrudox; very fine, ferritic-gibbsitic, isothermic |
| ISCW 12 | Plinthic Akraquox; very fine, gibbsitic, isohyperthermic |
| ISCW 13 | Typic Haplustox; coarse-loamy, kaolinitic, isohyperthermic |
| ISCW 14 | Humic Rhodic Eutrustox; very fine, ferritic, isohyperthermic |
| ISCW 15 | Typic Eutrustox; fine, kaolinitic, isohyperthermic |
| ISCW 16 | Ustic Kandihumult; clayey, kaolinitic, isohyperthermic |
| ISCW 17 | Humic Rhodic Akrustox; fine, kaolinitic, isohyperthermic |
| ISCW 18 | Typic Akrustox; very fine, gibbsitic, isohyperthermic |
| ISCW 19 | Humic Akrustox; clayey-skeletal, ferritic, isohyperthermic |
| ISCW 20 | Typic Akrustox; very fine, gibbsitic, isohyperthermic |
| ISCW 22 | Rhodic Haplustox; fine, kaolinitic, isohyperthermic |
| ISCW 23 | Aeric Ochraquox; fine, kaolinitic, isohyperthermic |
| IIRCC-4RJ | Typic Haplorthox; clayey, kaolinitic, hyperthermic |
| SBCS M-3 | Aplic Acorthox; very fine, kaolinitic, isohyperthermic |

Results and Discussion

The ECEC values and exchangeable cations determined using three different extractors are shown in Tables 2, 3, and 4. The results are in good agreement for exchangeable bases and ECEC, but for most samples they seem a little higher for extractable acidity when AgTU is used. According to Gallez et al. (1976), cited by Pleysier and Juo (1980): "Errors in exchangeable acidity may arise from the adsorption or release of protons from the oxide surface during the extraction with normal KCl solution. Nevertheless, such errors are generally small for most well-weathered soils because the soil pH value in N KCl suspensions is often near the point zero charge (PZC) of the soil." In measuring exchangeable acidity, the dilute AgTU solution minimizes the adsorption or desorption of protons at the oxide surface that may occur during extraction with N KCl (Pleysier and Juo 1980).

The apparent ECEC values are highly correlated when referred to 100 g of soil or 100 g of clay (Table 5), although in some samples the ECEC seems higher when AgTU is used due to extractable acidity data.

The statistical analysis points out that the mean values of ECEC using AgTU differ statistically from the others (Table 6). All soil samples fit in the criteria for low activity clay (ECEC ≤ 12 meq/100 g clay) for all extractors used.

According to the results obtained we can conclude that for strongly weathered soils with a predominance of variable charge colloids, the apparent ECEC may be determined by any of these three extractors which are highly correlated.

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Table 2. Exchangeable Cations and Apparent ECEC Using Three Different Extractors

| Pedons ISCW | Horizon | SNLCS | | | Extr. Al ^a | AgTU (meq/100 g soil) | | | Extr. Al ^b | NH ₄ OAc | | | |
|----------------|---------|-------|------|------|--------------------------|--------------------------|------|------|--------------------------|---------------------|------|------|------|
| | | Ca | Mg | Na | | K | Ca | Mg | | Ca | Mg | Na | K |
| 1 | Bo2 | 0.03 | 0.01 | 0.01 | 0.02 | 0.85 | 0.01 | 0.01 | 0.01 | 1.18 | 0.05 | 0.01 | 0.01 |
| 2 | B01 | 0.07 | 0.02 | 0.02 | 0.01 | 0.20 | 0.01 | 0.01 | 0.01 | 0.30 | 0.04 | 0.02 | 0.01 |
| 3 | BA | 0.02 | 0.22 | 0.02 | 0.03 | 1.25 | 0.01 | 0.25 | 0.02 | 0.03 | 1.23 | 0.03 | 0.22 |
| 3 | Bo1 | 0.02 | 0.04 | 0.02 | 0.02 | 1.10 | 0.03 | 0.03 | 0.03 | 1.10 | 0.03 | 0.04 | 0.01 |
| 4 | Bo1 | 0.06 | 0.15 | 0.02 | 0.06 | 1.33 | 0.03 | 0.13 | 0.02 | 0.03 | 1.75 | 0.19 | 0.15 |
| 4 | Bo2 | 0.06 | 0.05 | 0.03 | 0.10 | 0.35 | 0.03 | 0.05 | 0.07 | 0.14 | 0.50 | 0.03 | 0.04 |
| 5 | Bo2 | 0.70 | 0.36 | 0.02 | 0.04 | 1.20 | 0.47 | 0.36 | 0.02 | 0.03 | 1.57 | 0.80 | 0.34 |
| 5 | Bo2 | 0.04 | 0.11 | 0.01 | 0.05 | 0.85 | 0.06 | 0.10 | 0.04 | 0.06 | 1.77 | 0.05 | 0.11 |
| 5 | BC | 0.01 | 0.10 | 0.02 | 0.05 | 2.65 | 0.01 | 0.09 | 0.02 | 0.04 | 3.10 | 0.02 | 0.14 |
| 6 | Bo2 | 4.38 | 1.25 | 0.02 | 0.08 | 0.40 | 4.20 | 1.30 | 0.04 | 0.05 | 0.20 | 4.14 | 1.18 |
| 6 | Bo1 | 0.44 | 0.68 | 0.03 | 0.05 | 1.23 | 0.25 | 0.72 | 0.03 | 0.07 | 1.78 | 0.47 | 0.70 |
| 6 | Bo2 | 1.40 | 2.83 | 0.03 | 0.09 | 0.25 | 1.41 | 2.78 | 0.04 | 0.06 | 0.18 | 1.41 | 2.79 |
| 6 | BC | 0.02 | 0.12 | 0.03 | 0.35 | 1.57 | 0.01 | 0.11 | 0.04 | 0.32 | 2.36 | 0.05 | 0.14 |
| 7 | Bo2 | 0.11 | 0.04 | 0.02 | 0.05 | 0.20 | 0.12 | 0.05 | 0.03 | 0.04 | 0.17 | 0.10 | 0.03 |
| 8 | Bo2 | 2.34 | 0.96 | 0.02 | 0.02 | 0.12 | 2.30 | 1.12 | 0.02 | 0.04 | 0.10 | 2.25 | 0.94 |
| 9 | Bo1 | 0.06 | 0.01 | 0.01 | 0.01 | 0.23 | 0.02 | 0.01 | 0.01 | 0.03 | 0.20 | 0.02 | 0.01 |
| 10 | Bo2 | 0.07 | 0.01 | 0.01 | 0.01 | 0.27 | 0.03 | 0.01 | 0.02 | 0.04 | 0.34 | 0.03 | 0.01 |
| 11 | Bo3 | 0.04 | 0.01 | 0.01 | 0.01 | 0.10 | 0.02 | 0.01 | 0.02 | 0.02 | 0.10 | 0.01 | 0.01 |
| 12 | Bov1 | 0.03 | 0.01 | 0.01 | 0.01 | 0.10 | 0.02 | 0.01 | 0.01 | 0.02 | 0.26 | 0.02 | 0.01 |
| 13 | Bo1 | 0.04 | 0.03 | 0.01 | 0.02 | 0.90 | 0.01 | 0.03 | 0.02 | 0.04 | 1.07 | 0.04 | 0.03 |
| 13 | Bo2 | 0.04 | 0.02 | 0.01 | 0.02 | 0.70 | 0.01 | 0.02 | 0.02 | 0.08 | 1.14 | 0.05 | 0.02 |
| 14 | Bo2 | 2.40 | 0.46 | 0.02 | 0.52 | 0.15 | 2.48 | 0.46 | 0.04 | 0.40 | 0.14 | 2.45 | 0.46 |
| 15 | Bo1 | 1.08 | 0.40 | 0.01 | 0.20 | 0.13 | 1.07 | 0.41 | 0.02 | 0.14 | 0.20 | 1.06 | 0.41 |
| 16 | Bo1 | 1.16 | 0.48 | 0.02 | 0.06 | 0.18 | 1.16 | 0.47 | 0.02 | 0.04 | 0.19 | 1.13 | 0.45 |
| 16 | Bo | 0.53 | 0.63 | 0.02 | 0.18 | 0.15 | 0.48 | 0.66 | 0.02 | 0.12 | 0.12 | 0.45 | 0.70 |
| 17 | Bo3 | 0.80 | 0.13 | 0.02 | 0.04 | 0.07 | 0.78 | 0.14 | 0.02 | 0.03 | 0.06 | 0.79 | 0.15 |
| 18 | Bo | 0.09 | 0.01 | 0.01 | 0.01 | 0.20 | 0.09 | 0.01 | 0.02 | 0.03 | 0.26 | 0.06 | 0.01 |
| 18 | Boc2 | 0.07 | 0.02 | 0.01 | 0.01 | 0.10 | 0.06 | 0.03 | 0.02 | 0.01 | 0.34 | 0.04 | 0.02 |
| 19 | Bocl | 0.08 | 0.02 | 0.01 | 0.01 | 0.20 | 0.05 | 0.02 | 0.01 | 0.03 | 0.27 | 0.04 | 0.02 |
| 20 | Bo2 | 0.06 | 0.01 | 0.02 | 0.01 | 0.14 | 0.03 | 0.01 | 0.022 | 0.04 | 0.16 | 0.02 | 0.01 |
| 22 | BA | 0.02 | 0.02 | 0.03 | 0.04 | 1.50 | 0.03 | 0.01 | 0.02 | 0.04 | 1.50 | 0.05 | 0.02 |
| 22 | Bo1 | 0.02 | 0.01 | 0.03 | 0.04 | 1.38 | 0.01 | 0.01 | 0.02 | 0.02 | 1.35 | 0.03 | 0.01 |
| 22 | Bo2 | 0.02 | 0.03 | 0.02 | 0.02 | 0.95 | 0.01 | 0.01 | 0.02 | 0.01 | 1.10 | 0.05 | 0.02 |

Table continued on next page

Table 2. Continued

| Pedons ISCW | Horizon | SNLCS | | | Extr. Al ^a | AgTU | | | Extr. Al ^b | NH ₄ OAc | | | |
|----------------|---------|-------|------|------|--------------------------|------|------|------|--------------------------|---------------------|------|------|------|
| | | Ca | Mg | Na | | Ca | Mg | Na | | Ca | Mg | Na | K |
| 23 | Bogl | 0.03 | 0.02 | 0.02 | 0.03 | 1.95 | 0.01 | 0.2 | 0.02 | 1.95 | 0.14 | 0.02 | 0.01 |
| 23 | Bcvg | 0.02 | 0.01 | 0.03 | 0.02 | 1.67 | 0.04 | 0.01 | 0.02 | 1.80 | 0.07 | 0.02 | 0.01 |
| IIRCC-4RJ | BA | 0.04 | 0.02 | 0.01 | 0.01 | 2.35 | 0.03 | 0.02 | 0.05 | 2.34 | 0.13 | 0.02 | 0.01 |
| IIRCC-4RJ | Bol | 0.01 | 0.01 | 0.01 | 0.01 | 2.23 | 0.01 | 0.01 | 0.01 | 2.30 | 0.08 | 0.01 | 0.01 |
| IIRCC-4RJ | Bo3 | 0.08 | 0.02 | 0.01 | 0.01 | 0.80 | 0.07 | 0.03 | 0.04 | 1.40 | 0.15 | 0.03 | 0.01 |
| SBCS M-3 | BA | 0.01 | 0.07 | 0.10 | 0.09 | 2.10 | 0.01 | 0.06 | 0.10 | 2.00 | 0.06 | 0.08 | 0.07 |
| SBCS M-3 | Bol | 0.07 | 0.08 | 0.14 | 0.10 | 1.15 | 0.06 | 0.09 | 0.12 | 1.30 | 0.19 | 0.08 | 0.11 |

^a Extractable acidity titrated with 0.025N NaOH in KCl extract.^b Extractable acidity titrated with 0.025N NaOH in Ag TU extract.

Table 3. Apparent Effective Cation Exchange Capacity Using Three Different Extractors Referred to 100 g Soil

| Pedon ISCW | Horizon | SNLCS | NH ₄ OAc (meq/100 g soil) | AgTU |
|---------------|---------|-------|---|------|
| 1 | Bo2 | 0.92 | 0.93 | 1.22 |
| 2 | Bo1 | 0.32 | 0.28 | 0.42 |
| 3 | BA | 1.54 | 1.53 | 1.54 |
| 3 | Bo1 | 1.20 | 1.19 | 1.22 |
| 4 | Bo1 | 1.62 | 1.69 | 1.96 |
| 4 | Bo2 | 0.59 | 0.52 | 0.79 |
| 5 | Bto2 | 2.32 | 2.39 | 2.45 |
| 5 | Bo2 | 1.06 | 1.08 | 2.03 |
| 5 | BC | 2.83 | 2.87 | 3.26 |
| 6 | Bto2 | 6.13 | 5.83 | 5.79 |
| 6 | Bo1 | 2.43 | 2.47 | 2.85 |
| 6 | Bo2 | 4.60 | 4.57 | 4.47 |
| 6 | BC | 2.09 | 2.20 | 2.84 |
| 7 | Bo2 | 0.42 | 0.39 | 0.41 |
| 8 | Bo2 | 3.46 | 3.33 | 3.58 |
| 9 | Bo1 | 0.32 | 0.28 | 0.27 |
| 10 | Bo2 | 0.37 | 0.33 | 0.44 |
| 11 | Bo3 | 0.17 | 0.14 | 0.17 |
| 12 | Bov1 | 0.16 | 0.15 | 0.32 |
| 13 | Bo1 | 1.00 | 0.99 | 1.17 |
| 13 | Bo2 | 0.79 | 0.79 | 1.27 |
| 14 | Bo2 | 3.55 | 3.52 | 3.52 |
| 15 | Bto1 | 1.82 | 1.77 | 1.84 |
| 16 | Bto1 | 1.90 | 1.81 | 1.88 |
| 16 | Bo | 1.51 | 1.45 | 1.40 |
| 17 | Bo3 | 1.06 | 1.05 | 1.03 |
| 18 | Bo | 0.32 | 0.29 | 0.41 |
| 18 | Boc2 | 0.21 | 0.18 | 0.46 |
| 19 | Boc1 | 0.32 | 0.28 | 0.38 |
| 20 | Bo2 | 0.24 | 0.19 | 0.26 |
| 22 | BA | 1.61 | 1.60 | 1.60 |
| 22 | Bo1 | 1.48 | 1.44 | 1.41 |
| 22 | Bo2 | 1.04 | 1.04 | 1.15 |
| 23 | Bog1 | 2.05 | 2.16 | 2.01 |
| 23 | Bcvg | 1.75 | 1.78 | 1.89 |
| IIIRCC-4RJ | BA | 2.43 | 2.52 | 2.46 |
| IIIRCC-4RJ | Bo1 | 2.27 | 2.34 | 2.34 |
| IIIRCC-4RJ | Bo3 | 0.92 | 1.01 | 1.60 |
| SBCS M-3 | BA | 2.37 | 2.40 | 2.26 |
| SBCS M-3 | Bo1 | 1.54 | 1.65 | 1.68 |

Table 4. Apparent Effective Cation Exchange Capacity Using Three Different Extractors Referred to 100 g Clay

| Pedons ISCW | Horizons | SNLCS (meq g soil/cm ³) | NH ₄ OAc (meq/100 g soil) | AgTU (meq/cm ³) |
|----------------|----------|--|---|--------------------------------|
| 1 | Bo2 | 2.22 | 2.27 | 2.98 |
| 2 | Bo1 | 0.48 | 0.42 | 0.63 |
| 3 | BA | 4.40 | 4.37 | 4.49 |
| 3 | Bo1 | 3.53 | 3.41 | 3.59 |
| 4 | Bo1 | 2.16 | 2.25 | 2.61 |
| 4 | Bo2 | 0.77 | 0.68 | 1.04 |
| 5 | Bto2 | 2.97 | 3.06 | 3.14 |
| 5 | Bo2 | 1.41 | 1.44 | 2.71 |
| 5 | BC | 4.10 | 4.07 | 4.72 |
| 6 | Bto2 | 7.86 | 7.47 | 7.42 |
| 6 | Bo1 | 3.15 | 3.21 | 3.70 |
| 6 | Bo2 | 6.05 | 6.01 | 5.88 |
| 6 | BC | 2.78 | 2.93 | 3.79 |
| 7 | Bo2 | 0.55 | 0.51 | 0.54 |
| 8 | Bo2 | 4.74 | 4.56 | 4.90 |
| 9 | Bo1 | 0.78 | 0.66 | 0.66 |
| 10 | Bo2 | 1.68 | 1.50 | 2.00 |
| 11 | Bo3 | 0.19 | 0.16 | 0.20 |
| 12 | Bo1 | 0.25 | 0.25 | 0.50 |
| 13 | Bo1 | 5.88 | 5.82 | 6.88 |
| 13 | Bo2 | 4.39 | 4.39 | 7.06 |
| 14 | Bo2 | 4.93 | 4.88 | 4.89 |
| 15 | Bto1 | 4.55 | 4.42 | 4.60 |
| 16 | Bto1 | 3.72 | 3.33 | 3.69 |
| 16 | Bo | 3.14 | 3.02 | 2.92 |
| 17 | Bo3 | 1.96 | 1.94 | 1.91 |
| 18 | Bo | 0.40 | 0.36 | 0.51 |
| 18 | Bo2 | 0.28 | 0.24 | 0.64 |
| 19 | Bo1 | 0.38 | 0.34 | 0.46 |
| 20 | Bo2 | 0.30 | 0.23 | 0.33 |
| 22 | BA | 3.35 | 3.33 | 3.33 |
| 22 | Bo1 | 3.08 | 3.00 | 2.94 |
| 22 | Bo2 | 2.36 | 2.36 | 2.61 |
| 23 | Bogl | 3.41 | 3.60 | 3.35 |
| 23 | Bcvg | 2.96 | 3.01 | 3.20 |
| IIIRCC-4RJ | BA | 5.40 | 5.60 | 5.47 |
| IIIRCC-4RJ | Bo1 | 4.83 | 4.98 | 4.98 |
| IIIRCC-4RJ | Bo2 | 1.80 | 1.98 | 3.14 |
| SBCS M-3 | BA | 2.96 | 3.00 | 2.83 |
| SBCS M-3 | Bo1 | 1.81 | 1.94 | 1.98 |

Table 5. Correlation Coefficient (*r*) between ECEC Values

| Extractors | <i>r</i> (100 g soil) | <i>r</i> (100 g clay) |
|-----------------------------|--------------------------|--------------------------|
| SNLCS x NH ₄ OAc | 0.998** | 0.997** |
| SNLCS x AgTU | 0.980** | 0.960** |
| NH ₄ OAc x AgTU | 0.983** | 0.964** |

** Significant at the 0.01 level

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Table 6. Comparison of Means According to Tests of Paired Observations between ECEC Values

| Extractors | Means* | Means |
|---------------------|--------------|--------------|
| | (100 g soil) | (100 g clay) |
| AgTU | a | 1.7025 |
| SNLCS | b | 1.5680 |
| NH ₄ OAc | b | 1.5557 |

* Means with the same letter are not significantly different.