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Charge Characteristics in Relation to Mineralogy of Selected Soils from South-east Asia

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ABSTRAK

Satu kajian mengenai mineralogi dan ciri-ciri cas telah dijalankan terhadap 7 jenis tanah dari Filipina, Indonesia dan Malaysia. Tanah-tanah tersebut termasuk 1 Entisol, 3 Alfisol, 1 Andisol dan 2 Oksisol. Smektit, mika dan kuarza didapati wujud di bahagian lempung pada tanah Entisol. Smektit dan kaolinit ialah mineral-mineral dominan di tanah Alfisol. Kebanyakan mineral di tanah Andisol ialah haloisit, manakala kaolinit dan oksida banyak didapati di tanah Oksisol. Kuantiti kaolinit meningkat dari bawah ke permukaan tanah, sedangkan kuantiti haloisit semakin menurun. Ini menunjukkan haloisit telah ditukarkan kepada kaolinit semasa proses luluhawa. Perbezaan mineralogi bagi tanah yang berlainan adalah jelas dipengaruhi oleh perbezaan ciri-ciri cas. Tanah yang mengandungi smektit (Entisol dan Alfisol) mempunyai cas negatif yang tinggi. Tanah Andisol mengandungi haloisit dan tanah Oksisol mengandungi kaolinit dan oksida, mempunyai jumlah cas positif yang tinggi tetapi jumlah cas negatif yang sederhana. Ketersediaan Ca dalam tanah bergantung kepada Ca tukarganti dan keupayaan pertukaran kation berkesan.

ABSTRACT

The mineralogy and charge characteristics of 7 soils from the Philippines, Indonesia and Malaysia were studied. The soils consisted of an Entisol, 3 Alfisols, an Andisol and 2 Oxisols. Smectite, mica and quartz were present in the clay fraction of the Entisol. In the Alfisols, smectite and kaolinite were the dominant minerals. The Andisol was dominated by halloysite, whereas the Oxisols were dominated by kaolinite and oxides. The amount of kaolinite increased towards the surface, while halloysite decreased, indicating the transformation of halloysite to kaolinite during the course of weathering. Differences in mineralogy of the various soil types were reflected clearly in the differences in charge characteristics. Soils with smectite (Entisol and Alfisol) had high a negative charge. The Andisol, which contained halloysite, and the Oxisol, with kaolinite and oxides, had high amounts of positive charge, but moderate amounts of negative charge. The availability of Ca in the soils depended upon exchangeable Ca and the effective cation exchange capacity (ECEC).

INTRODUCTION

Many soils in the Philippines and Indonesia and some soils in Malaysia are derived from volcanic rocks of recent to Pleistocene age. Depending on the age and composition of the parent rock, and the stage of weathering, volcanic soils in the tropics contain allophane, halloysite, smectite, kaolinite, goethite and gibbsite (Eswaran 1979; Delvaux *et al.* 1989). Volcanic soils classified as Andisols are known to contain large amounts of halloysite (Mohr *et al.* 1972;

Allen and Hajek 1989). Imogolite and X-ray amorphous hydrous aluminosilicates (collectively known as allophane) are common in these soils (Wada 1989). Geographic distribution of allophane and imogolite has been connected with areas of recent volcanic activity throughout the Pacific ring.

The charge of Andisols containing allophane and imogolite is pH dependent (Okamura and Wada 1983). The cation exchange capacity (CEC) is known to increase with increasing soil pH and/or ionic strength (Gillman and Hallman 1988). For instance, the CEC increased when fertilizer such as sulphate of ammonia was applied, via replacement of OH^- by SO_4^{2-} (Guadalix and Pardo 1991). The CEC of these soils can be determined accurately by Ca or Ca plus Al adsorption (Gillman and Sumpter 1986) or by a compulsive exchange method (Gillman and Hallman 1988). Such an estimate gives a true CEC value under field conditions; this information is considered useful in soil management. The charge and ion retention properties chiefly govern the soil cation dynamics. In view of the lack of baseline data for these soil types, the objectives of this research were to characterize the mineralogy of a range of volcanic soils, occurring under different climatic conditions in the Philippines, Indonesia and Malaysia, and to establish the relationship between their mineralogy and charge characteristics.

MATERIALS AND METHODS

The Soils

An Entisol (Philippines), 3 Alfisols (Philippines), an Andisol (Indonesia) and 2 Oxisols (one each from the Philippines and Malaysia) were examined in the field, classified (Soil Survey Staff 1990) and sampled. Table 1 gives their location, annual rainfall, rock type and geologic

TABLE 1

Location, annual rainfall, rock type, geologic age and classification of soils from Indonesia, Malaysia and the Philippines

Country	Location	Annual Rainfall (mm)	Rock Type	Geologic Age	Classification
Philippines	Setio Bueno, Tarlac	1986	basalt	Holocene	Lithic Ustorthents
	UPLB, Laguna	2006	dolerite, tuff	Pleistocene	Typic Hapludalfs
	Rosario, La Union	2297	basalt	Pleistocene	Typic Hapludalfs
	VISCA, Leyte	2499	basalt	Pleistocene	Typic Palendalf
	CMU, Bukidnon	1828	basalt	Pleistocene	Kandiustalfic Eutrustox
Indonesia	Carita 11, Java	2874	basalt	Tertiary	Typic Hapludands
Malaysia	Kuantan, Pahang	2757	basalt	Tertiary	Typic Acrudox

TABLE 2
Chemical properties of soils from Indonesia, Malaysia and the Philippines

Soil	Hor	pH(1:1)	pH_o	Exch Ca	ECEC	B.S	Fe_2O_3	C_{org}	Clay
		(H_2O)		$cmol_c/kg$			(%)		
Entisol	A	5.2	2.7	16.3	23.5	97.2	2.4	1.8	28.6
Alfisol	Ap	6.2	3.7	25.1	34.4	99.9	2.1	1.1	37.7
	Bt_2	5.3	nd	29.7	42.7	99.3	1.6	1.3	53.2
Andisol	A_1	4.3	3.5	1.4	2.3	95.6	7.3	1.4	76.4
	Bw_2	4.7	nd	1.3	3.6	44.3	7.8	1.0	73.5
Oxisol	A	5.7	3.3	12.4	17.1	99.4	8.6	3.3	65.1
(Eutrustox)	Bt_2	4.7	nd	6.7	8.1	91.4	10.6	1.3	74.3

nd = not determined

age. Four soils were selected for detailed investigation into their mineralogy and charge properties. Chemical properties are given in Table 2. Note that for Table 2 both the Alfisol (UPLB) and Oxisol (CMU) were from the Philippines, and the Entisol is a shallow soil without B and C horizons. In addition, the Oxisol is a special type, Eutrustox, which has a very high base value (91.4 – 99.4%) compared with normal Oxisols, which have a base value of less than 35%.

Soil Analysis

The pH of a 1:1 solution of soil in water was determined after 1 h of intermittent shaking and standing overnight. Exchangeable Ca and Mg were extracted by 1 M NH_4OAc and determined by atomic absorption spectrophotometry, while exchangeable K and Na in the same extract were determined by flame photometry. Exchangeable Al was extracted by 1 M KCl and determined colorimetrically (Barnhisel and Bertsch 1982). Effective cation exchange capacity (ECEC) was calculated as the sum of basic exchangeable cations and exchangeable Al, while base saturation (BS) was calculated on the basis of ECEC. Free iron oxide content was determined by the method of Mehra and Jackson (1960). The Walkley-Black method (Nelson and Sommers 1982) was used to measure organic carbon (C_{org}).

Clay content of the soils was determined by the pipette method of Day (1965). To obtain the clay, the soil was first treated with H_2O_2 to remove organic matter. It was later dispersed with dilute Na_2CO_3 solution. This clay ($< 2\mu m$) was later used to study the mineralogy of the soils by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The XRD analysis was conducted by an automated Phillips diffractometer equipped with a graphite monochromator, operated from 3 to 50 degrees 2-theta, using Cu Kα radiation and scanning speed of half a degree per minute. The X-ray diffraction analysis was carried out on Mg-saturated and glycolated samples.

Charge characteristics of the soils were determined by the method of Gillman and Sumpter (1986). In this method, negative charge as measured by Ca adsorption was termed CEC_B, while that measured by Ca and Al adsorption was termed CEC_T. The positive charge as measured by Cl adsorption was termed AEC. Soil weathering index (WI) was calculated (at soil pH) as follows (Tessens and Shamshuddin 1983):

$$WI = \frac{\text{negative charge} - \text{positive charge}}{\text{negative charge}} \times 100$$

The negative charge (CEC_B) and the positive charge required for WI determination were estimated from the charge curves. PH_o, defined as the pH of the variable charge colloid at which the net charge is zero, was determined by the method of Gillman and Sumpter (1986).

Extraction of Soil Solution and Analysis

Distilled water was added to the air-dried soils and the wetted soils were subsequently incubated for 1 day at a matric suction of 10 kPa (Menzies and Bell 1988). This study assumed that a state of equilibrium was reached between the liquid and solid phase of the soils during the incubation period. Soil solutions were extracted by centrifugation at 2000 rpm for 1 h using specially designed centrifuge tubes. The pH and electrical conductivity (EC) of each soil extract were immediately determined from 2-ml subsamples. The remainder of the extract was stored at 5°C for later determination of Ca, Mg, K, Na, Fe, Al, S and P by inductively coupled plasma atomic emission spectroscopy (ICPAES).

RESULTS AND DISCUSSION

Soil Types

Differences in the lithology, age of parent material, climate, drainage conditions and vegetation for the soils in the different regions (Table 1) were reflected by the differences in their chemical properties (Table 2), as the soils had undergone different rates of chemical weathering. Hence, several groups of soils ranging from Entisol to Oxisol are found in the region. According to their chemical properties and profile morphology, the least weathered of all the study soils was sited at Setio Bueno, Tarlac, the Philippines (Entisol), while the most weathered soil was found at Bukidnon, the Philippines and Kuantan, Malaysia (Oxisol). The other soils, which had high basic exchangeable cations, were classified as Alfisols (Philippines). The soil which had low bulk density and a high amount of amorphous materials was classified as an Andisol (Indonesia). The Oxisol and Andisol also contain high amounts of Fe_2O_3.

Mineralogy

Overall, the peaks on the X-ray diffractograms of the soils were clear although the clay samples were likely to be coated with the amorphous materials (including allophane and/or sesquioxides). Furthermore, the low scanning speed was slow enough to allow differentiation of the individual minerals on the X-ray diffractograms. Although allophane is common in volcanic soils (Mohr *et al.* 1972; Wada 1989) it was not confirmed by the XRD analysis or TEM observation.

The X-ray diffractogram of the Entisol had reflections at 1.52, 1.0, 0.713, 0.5, 0.445, 0.426, 0.418, 0.356, 0.334, 0.245 and 0.212 nm (*Fig. 1*). The strong and clear reflections at 0.426 and 0.334 nm indicated the presence of quartz in the clay fraction. Mica (1.0, 0.5 nm), smectite (1.52 nm) and kaolinite (0.713, 0.356 nm) were also present, but in lesser amounts. A small amount of goethite (0.418, 0.245 nm) and hydrated halloysite ($Al_2Si_2O_5(OH)_4.2H_2O$) were present. The presence of the hydrated halloysite was shown by the 1.0, 0.445 and 0.346 nm reflections (Dixon 1989). The TEM micrograph (*Plate 1A*) gives a visual illustration of the presence of kaolinite, mica and/or smectite in the soil. The smectite in the soil was probably an alteration product of mica weathering under impeded drainage conditions (Velde 1992). This smectite can be interstratified with the hydrated halloysite as shown by Delvaux *et al.* (1989) in volcanic soils of Cameroon.

Smectite, halloysite, kaolinite and goethite were present in the topsoil of the Alfisol (*Fig. 1*). The 1.5 – 1.8 nm peak was strong and intense, suggesting that smectite

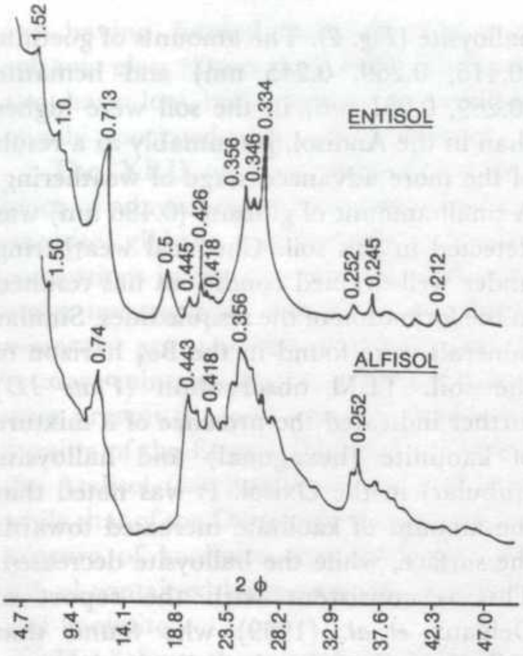

Fig. 1 X-ray diffraction patterns of Mg-saturated clay fraction from the A horizon of the Entisol and Alfisol.

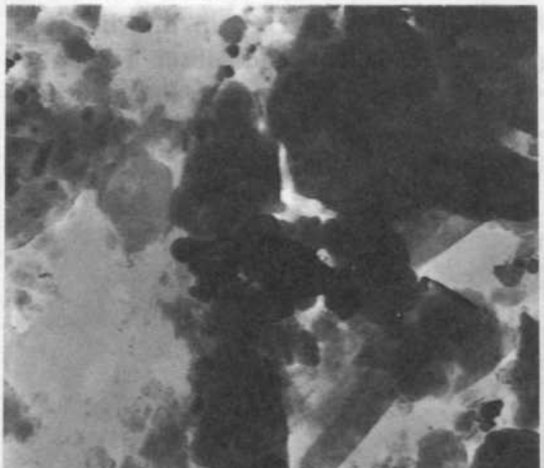

Plate 1A. TEM micrograph of the clay fraction from the A horizon of the Entisol

was present in large amounts. This is consistent with the high ECEC value (*Table 2*) and is a common feature of a volcanic soil with moderately well drained conditions existing under a udic moisture regime. The presence of amorphous materials in the clay sample tended to reduce the sharpness of the XRD peaks. *Plate 1B* shows the occurrence of halloysite, kaolinite and/or smectite being coated by the amorphous materials. The halloysite in the soil was similar in morphology to the ferruginous halloysite from Hokkaido, Japan (Wada and Mizota 1982).

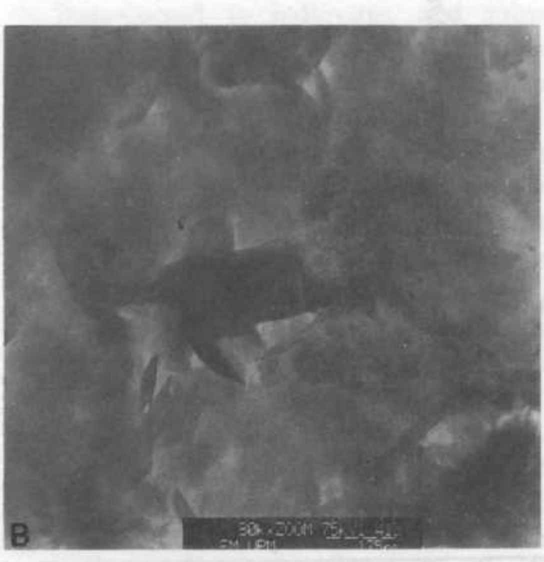

Plate 1B. TEM micrograph of the clay fraction from the Ap horizon of the Alfisol

The X-ray diffractogram of the Andisol (*Fig. 2*) showed the dominance of halloysite (0.73, 0.445 nm) with minor amounts of kaolinite (0.358 nm), goethite (0.418, 0.269, 0.245 nm) and hematite (0.270, 0.252 nm). The sample from the C horizon (not shown) gave a similar XRD pattern, indicating a similar mineralogy in both horizons. The peaks 0.73 and 0.445 nm belong to dehydrated halloysite, which has a formula of $Al_2Si_2O_5(OH)_4$ (Dixon 1989). This halloysite is common in youthful soil developed from volcanic rock (Dixon 1989; Allen and Hajek 1989). TEM observation further indicated that the clay fraction was dominated by halloysite, without clear manifestation of the presence of allophane and/or imogolite (*Plate 1C*). Further TEM observations (not illustrated here) suggest

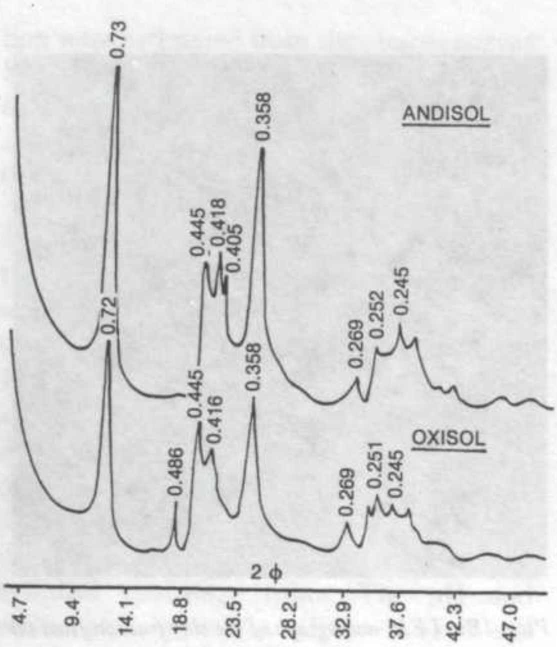

Fig. 2 X-ray diffraction patterns of Mg-saturated clay fraction from the A horizon of the Andisol and Oxisol

that halloysite was more dominant in the subsoil than in the topsoil.

The XRD peaks on the diffractogram of the Oxisol were different from those of the Andisol in that clear peaks were observed at 0.72 and 0.358 nm, showing that kaolinite was more dominant than halloysite (*Fig. 2*). The amounts of goethite (0.416, 0.269, 0.245 nm) and hematite (0.269, 0.251 nm) in the soil were higher than in the Andisol, presumably as a result of the more advanced stage of weathering. A small amount of gibbsite (0.486 nm) was detected in this soil. Chemical weathering under well-drained conditions has resulted in the formation of the sesquioxides. Similar minerals were found in the Bo_4 horizon of the soil. TEM observation (*Plate 1D*) further indicated the presence of a mixture of kaolinite (hexagonal) and halloysite (tubular) in the Oxisol. It was noted that the amount of kaolinite increased towards the surface, while the halloysite decreased. This is consistent with the report of Delvaux *et al.* (1989) who found that halloysite in volcanic soils in Western Cameroon was formed earlier than kaolinite in the weathering sequence.

Clear X-ray reflections at 0.404 and 0.248 nm were observed in the diffractograms of the Andisol and Alfisol. The mineral which gives the reflections was identified as cristobalite, which is commonly associated with volcanic deposits of Tertiary

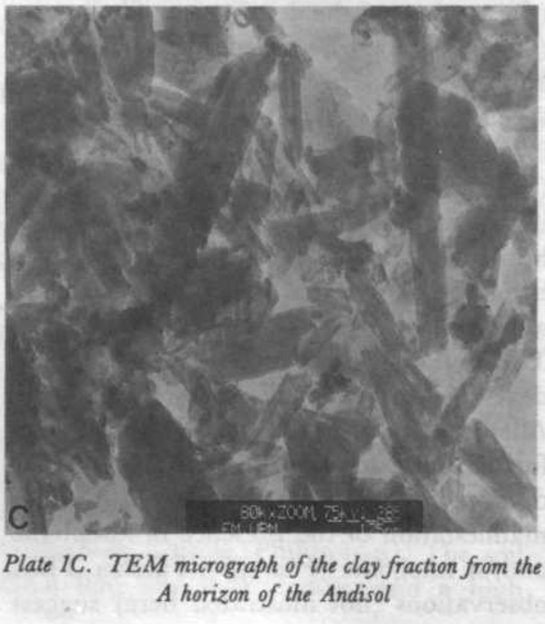

Plate 1C. TEM micrograph of the clay fraction from the A horizon of the Andisol

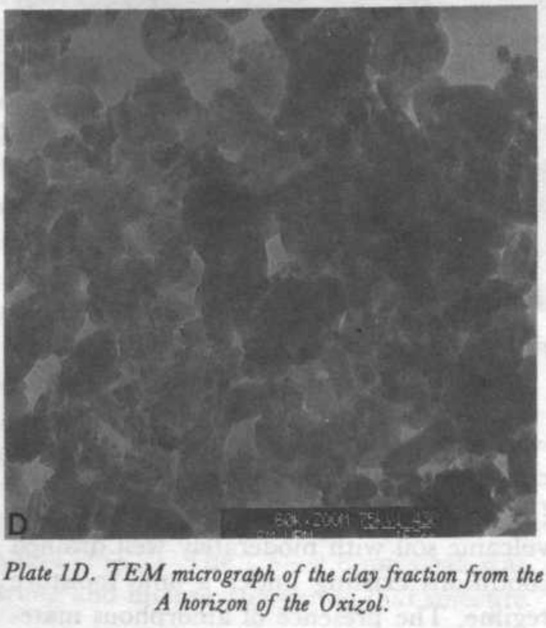

Plate 1D. TEM micrograph of the clay fraction from the A horizon of the Oxizol.

age having formed from dissolution of volcanic glass (Drees *et al.* 1989). Both soils also have low bulk density, an attribute usually associated with andic properties.

The XRD analyses showed the presence of admixtures of halloysite, kaolinite, smectite, gibbsite and goethite in various proportions in the clay fraction of the soils under investigation. The majority of those minerals are variable-charge minerals. Primary minerals such as mica and quartz were present in small amounts in the clay fraction of the Entisol. The clay fraction of the Andisol was dominated by halloysite, while that of the Oxisol was dominated by a mixture of kaolinite and halloysite. The Alfisol contained large amounts of smectite and kaolinite.

Fig. 3 shows the suite of clay minerals in the different soil types arranged in order of decreasing abundance. Volcanic materials first weather to form either Andisol or Entisol, depending on the mineralogical composition and moisture regime. According to Mohr *et al.* (1972), volcanic glass under well-drained conditions first changes to allophane, which on further weathering is transformed to halloysite, and subsequently to kaolinite. This transformation can be visualized in the development of an Andisol.

In the other pathway, mica in the Entisol first weathers to smectite. Further weathering results in a complete destruction of mica and the formation of smectite, which in turn weathers to halloysite and/or kaolinite. Such a sequence typifies the development of an Alfisol. In the Oxisol, kaolinite, oxides and halloysite are present, but smectite is absent. We found that the amount of goethite and hematite increased with weathering. Additionally, kaolinite in the Alfisol, Andisol and Oxisol increased towards the surface, while halloysite decreased. These observations support the scheme of mineral weathering shown in *Fig. 3.*

Charge Properties

The CEC_B of the Entisol and Alfisol was high, with values comparable to those reported by Gillman and Hallman (1988) for the volcanic soils of Papua New Guinea. The high negative charge in the Entisol and

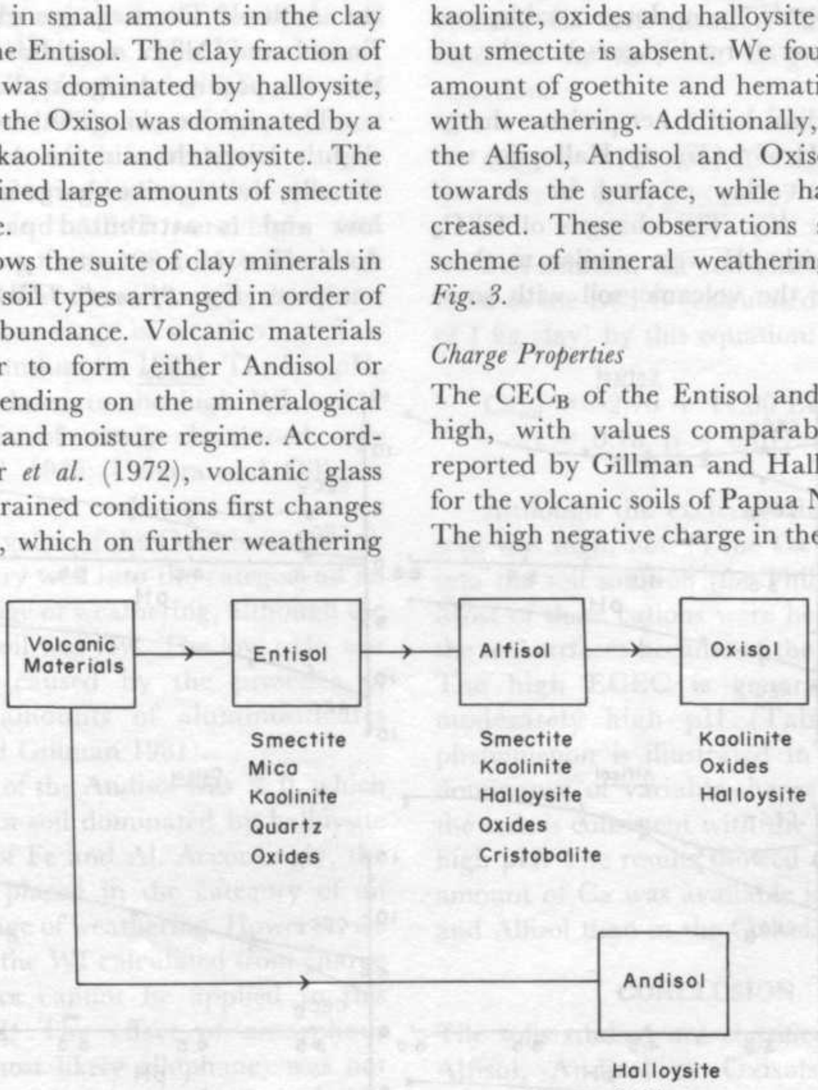

Fig. 3. Soil minerals arranged in order of decreasing abundance in the various soil types

Alfisol was attributed partly to the presence of smectite in the soils (*Fig. 4*). The ECEC of these two soils was high (Table 2). The CECB value of both soils increased as the pH increased. CECT appeared to increase in the Entisol and Alfisol at pH below 3.5 (*Fig. 4*). A portion of the Al measured in the soil solution could have been dissolved from the inner part of the phyllosilicates (smectite or mica) rather than on the exchange sites. At very low pH some of the soil minerals are no longer stable, and Al in the minerals readily goes into the soil solution.

The Andisol had a net positive charge at the soil pH of 4 (*Fig. 4*). Halloysite was the dominant clay mineral in the soil (*Fig. 2*; *Plate 1C*). The changes of CEC$_B$ and AEC with pH were similar to those reported for the volcanic soil with some allophane and/or imogolite (Okamura and Wada 1983). Like the Entisol and Alfisol, the CEC$_T$ of the Andisol increased below pH 3.5. The high positive charge present in the soil was partly caused by the Fe oxides (goethite and hematite), as indicated by their 7.3% content in the topsoil (Table 2). On the other hand, the Entisol and Alfisol, which had quartz, mica and smectite with minor amounts of kaolinite, exhibited different charge characteristics to those of the Andisol. The negative charge in the Entisol and Alfisol was, however, higher than the positive charge at the soil pH.

The positive charge in the Oxisol was slightly lower than in the Andisol. Additionally, the negative charge in the soil was low and is attributed partly to the dominance of low activity clays. XRD analyses (*Fig. 2) and TEM observations*

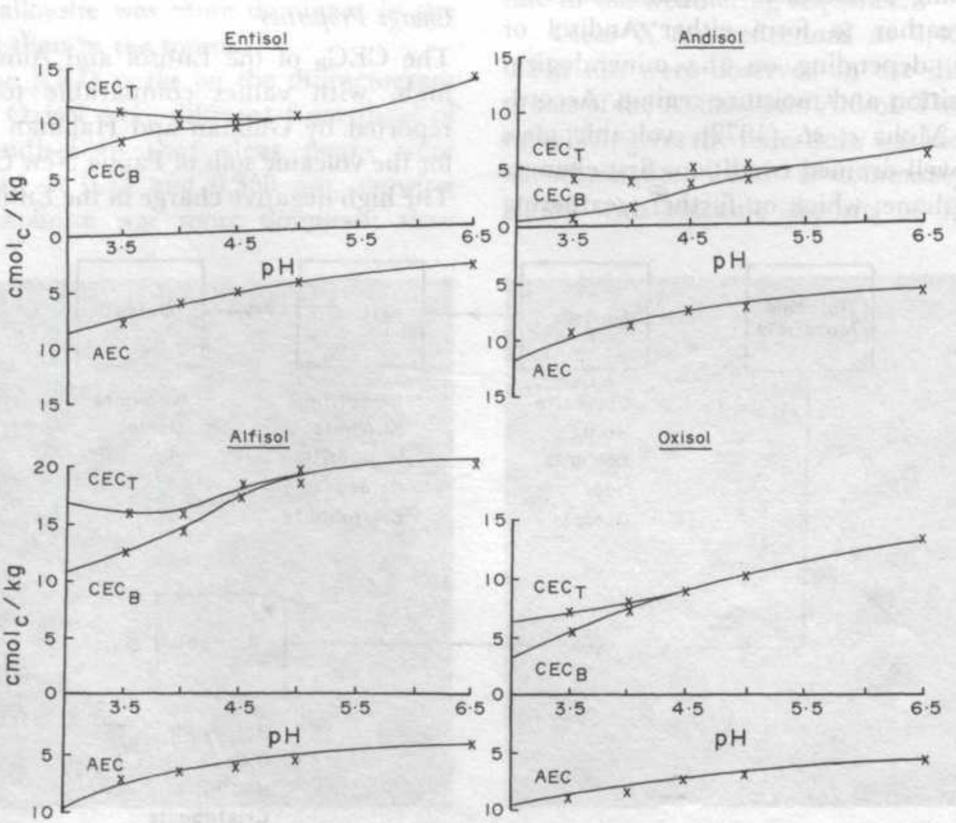

Fig. 4. Charge characteristics of the Entisol, Alfisol, Andisol and Oxisol

(Plate 1C and 1D) showed that kaolinite, halloysite, hematite and goethite were present in large amounts. The high amount of Fe_2O_3 contributed a certain amount of positive charge to the soil surfaces. The CEC_B and AEC of the topsoil are quite similar to those of the Andisol. However, the CEC_T did not increase below pH 3.5. The positive charge in the Bo_4 horizon of the Oxisol (data not shown) was higher than the negative charge at pH of 3.0 to 6.5. This charge pattern was similar to that of the soil at Kuantan, Malaysia. The Kuantan soil, classified as Typic Acrudox (Tessens and Shamshuddin 1983), is one of the most highly weathered soils in Malaysia.

The weathering index (WI) values for the Entisol and Alfisol were high, with values ranging from 100 to 50. The high value means that the soils are in the recent or intermediate stage of weathering (Tessens and Shamshuddin 1983). The low pH_o is directly related to the high WI and is characteristic of newly developed soils (Gallez *et al.* 1976; Uehara and Gillman 1981).

The WI value of the Oxisol was 50 – 0, which fits very well into the category of an advanced stage of weathering, although the pH_o of the soil was low. The low pHo was presumably caused by the presence of significant amounts of aluminosilicates (Uehara and Gillman 1981).

The WI of the Andisol was < 0, which is typical of a soil dominated by halloysite and oxides of Fe and Al. Accordingly, the soil can be placed in the category of an advanced stage of weathering. However, we believe that the WI calculated from charge characteristics cannot be applied to this kind of soil. The effect of amorphous materials (most likely allophane) was not taken into consideration in the formulation of WI. Allophane is normally associated with relatively young volcanic soil (Mohr *et al.* 1972).

Soil Solution Attributes

The soil solution Ca concentrations in the soils of the Philippines were high, but low in soils from Indonesia and Malaysia. The highest value of soil solution Ca concentration was observed in the Bt_2 of the Alfisol. The high value of the soil solution was consistent with the high exchangeable Ca in that horizon; the exchangeable Ca in the Bt_2 horizon of the Alfisol was 29.7 $cmol_c$/kg (Table 2). The relationship between the soil solution Ca concentration and exchangeable Ca in the soils is given by this equation:

$$Ca_{sol} = 0.79 + 41.40\ Ca_{exch},$$
$$(r = 0.75,\ p < 0.05)$$

Soil solution Ca concentration was related to the ECEC (calculated on the basis of 1 kg clay) by this equation:

$$Ca_{sol} = -2.70 + 11.00\ ECEC,$$
$$(r = 0.78,\ p < 0.01)$$

Although the exchangeable Ca in the soils was high, not all the Ca was released into the soil solution (for Philippine soils). Most of these cations were held tightly by the soil surfaces because of the high ECEC. The high ECEC is generated by the moderately high pH (Table 1). This phenomenon is illustrated in *Fig. 4.* The dominance of variable-charge minerals in the soils is consistent with the high CEC at high pH. The results showed that a higher amount of Ca was available in the Entisol and Alfisol than in the Oxisol.

CONCLUSION

The soils studied are classified as Entisol, Alfisol, Andisol or Oxisols. The clay fraction of the Entisol contains smectite, mica and quartz, while the Alfisol is dominated by smectite and kaolinite, with minor amounts of halloysite and goethite.

Halloysite is abundant in the Andisol, while the Oxisol contains a mixture of kaolinite and halloysite with significant amounts of sesquioxides.

The mineralogy of the soils significantly affects their charge characteristics. The Entisol and Alfisol have a high negative charge, but the Andisol has a high positive charge. Generally, the charge characteristics of the Andisol are similar to those of the Oxisol. The soil solution Ca is correlated to the exchangeable Ca and the charge characteristics. The availability of Ca is therefore governed by the amount of exchangeable Ca and the ECEC of the soils.

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