AGENCY FOR INTERNATIONAL DEVELOPMENT
WASHINGTON, D. C. 20523

BIBLIOGRAPHIC INPUT SHEET

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Batch #22

1. SUBJECT CLASSIFICATION

A. PRIMARY: Agriculture — AF22-0000-0000

B. SECONDARY: Soil chemistry and physics

2. TITLE AND SUBTITLE
Influence of micromorphology on ion release in an eutrustox

3. AUTHOR(S)
Moura Filho,W.; Buol,S.W.

4. DOCUMENT DATE: 1971

5. NUMBER OF PAGES: 22p.

6. ARC NUMBER
ARC: BR631.416.F481a

7. REFERENCE ORGANIZATION NAME AND ADDRESS
N.C.State

8. SUPPLEMENTARY NOTES *(Sponsoring Organization, Publishers, Availability)*

9. ABSTRACT

BEST COPY AVAILABLE

10. CONTROL NUMBER
PN-RAB-367

11. PRICE OF DOCUMENT

12. DESCRIPTORS
~~Eutrustox?~~
Ions
Morphology

13. PROJECT NUMBER

14. CONTRACT NUMBER
CSD-2806 Res.

15. TYPE OF DOCUMENT

AID 590-1 (4-74)

BR
631.416
F481a

Cod 2806

Influence of Micromorphology on Ion Release in an Eutrustox[1]

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ABSTRACT

Natural aggregates smaller than 2 mm from an Eutrustox were separated into three size fractions (2-0.5 mm, 0-5-0.25 mm, and 0.25 mm) by dry sieving. Portions of each aggregate fraction and the whole soil were crushed in a ball mill to destroy the microstructure. Total chemical composition does not change with aggregate size but mechanical destruction of the microstructure increases the acid-extractable Ca, Mg and P. High base status is postulated to be the result of entrapment of bases within peds during the dry season.

Additional key words for indexing: base status, chemical composition, soil testing, oxisol.

SOIL BRIEF

Soil material from, 98% base saturated, pH 5.9, Eutrustox was fractionated into peds of 2-0.5 mm, 0.5-0.25 mm and < 0.25 mm diameter by dry sieving. Portions of each fraction and of the whole soil had their microstructure mechanically destroyed causing increases in acid-extractable Ca, Mg, and P to be observed. A mechanism of accumulating these ions in fine granular peds under leaching conditions is postulated.

Earlier studies of an Eutrustox, from Minas Gerais, Brazil, indicated a paradoxial situation of a mineralogically highly weathered soil with relatively high pH and base saturation.[3/] It is postulated that these circumstances could be engendered by the firm, fine granular peds seen in thin sections to consist of plasma arranged in concentric layers.

Queiroz Neto et al. (1966) working with similar soils found anlytical variations in clay, pH, OM, N, Ca, Mg, K, and CEC that could be related to aggregate size. Khalifa and Buol (1969) found that argillans in the argillic horizon of an Ultisol reduced plant growhh and hypothesized that this may be caused by restricted ion diffusion from ped interiors, through the argillans, into the inter-ped soil solution. Similar restrictions are postulated to exist in the Eutrostox sample, as a result of intra-ped plasma arrangement, and to be effective in restricting the leaching of bases and also influence the results obtained from short-term extractions of soil samples.

This study was designed to (1) evaluate the effect of the Eutrustox microstructure on the results of chemical analysis by a standard Brazilian soil testing procedure, (2) investigate possibilities of increasing reproductibility of laboratory analyses and (3) suggest a possible explanation for the presence of high base status in the mineralogically high weathered soil.

Materials and Methods

An Eutrustox (Table 1) in Minas Gerais, Brazil was sampled from the 0-20 cm depth. The soil material was previously determined to be composed of 19.6% free iron oxide and contain 4.8% organic matter. After free iron oxide removal

[3/] Moura Filho, W. 1968. Characterization of the physical, chemical and micromorphological changes engendered by the cultivation of a soil in the Triangul of Minas Gerais, Brazil. M.S. Thesis. N. C. State Univ. at Raleigh.

it was found to be composed of about 74% fine clay (<0.2μ), 9.4% coarse clay (2-0.2μ), 7.8% sand (>50μ), 8.6% coarse silt (50-20μ) and 0.1% fine silt (20-2μ). Base saturation was determined as 98.8% and pH in 1:1 water was 5.9[3/]. Kaolinite and gibbsite composed approximately 41 and 13% of the clays, respectively. X-amorphous material was determined to compose 20% of the clay fraction[4/].

All of the air-dried sample was carefully passed through a 2-mm sieve with practically no crushing. A few larger concretions were discarded. The aggregates less than 2 mm in diameter were separated into three size fractions by dry sieving for five minutes on a Ro-Tap shaker. Aggregate sizes separated were 2-0.5 mm, 0.5-0.25 mm, and smaller than 0.25 mm. The size fractions represented 58%, 20%, and 22% of the total sample, respectively.

Free-Fe was removed by the dithionite-citrate method (Jackson, 1956). Mechanical analysis was carried out on Fe-free samples. The sand was separated by wet sieving, and clay and silt were determined by sedimentation using the pipette method (Kilmer and Alexander, 1949).

Total elemental analysis was conducted using the dissolution procedure described by Shapiro and Brannock (1956). Atomic absorption spectrophotometry was used to determine Si, Al, Fe, Mn, Ti, Ca, Mg, and K contents in all solutions. The molybdatevanadate method (Olsen and Dean, 1965) was used to determine P content in all extracts. The weight lost upon heating from 110°C to 900°C was recorded as percent loss on ignition.

[4/] Moura, Filho, W. 1970. Studies of a Latosol Roxo (Eutrustox) in Brazil. Clay mineralogy, micromorphology effect on ion release, and phosphate reactions. Ph. D. Thesis. N. C. State Univ. at Raleigh.

The remaining studies were conducted using samples of the whole soil, and of each aggregate size fraction in their natural condition and after crushing. Crushing was done by placing portions of each aggregate size fraction and of the whole soil in a ball mill until more than 50% of the sample passed a 300-mesh sieve. Since previous petrographic analysis [4/] determined that the sand fractions (> 50μ) consisted almost entirely of quartz and magnetic iron minerals, some crushing of these sand particles was assumed not to affect the results of the analysis performed.

Soil reaction in 1:1 soil-water and 1:1 soil-N KCl was measured by means of a pH meter equipted with a glass electrode. Exchangeable H and exchangeable Al were determined by the titration of N KCl leachates using methyl orange and phenolphthalein as indicators (Coleman et al.,1959).

Basic cations and phosphorus were extracted by three separate procedures using a dilute solution of 0.05 N HCl and 0.025 N H_2SO_4. Adsorbed P was also estimated using a 0.025 $(NH_4)_2SO_4$ solution. The following procedures were used:

1. Suspensions of 1:10 soil-extractant solution were shaken for five minutes and left undisturbed overnight (Vettori, 1969). This is the standard extraction procedure for most of the Brazilian laboratories. Aliquots for analysis were taken from filtrates rather than from the supernatant as described by Vettori.

2. Suspensions of 1:10 soil-extractant solution were shaken for five minutes, 1, 6, 24, and 72 hours. The suspensions were filtered immediately after the shaking was completed and aliquots were taken from the filtrates for analysis.

3. Procedure 2 was repeated using a 0.025 N $(NH_4)_2SO_4$ solution instead of the dilute acid solution to estimate the amount of adsorbed P.

4. Exhaustive leaching with the dilute acid solution was carried out in columns 5 cm deep. The oven-dried equivalent weight of the soil sample was used in each extraction. Each leachate was collected separately for analysis and the results were calculated on an oven-dried basis. Extraction time could not be equal for all samples due to permeability differences but each extraction was started at the same time for all samples.

Calcuim, magnesium and phosphorus were determined in all extracts. Determination of K was discontinued after it was found not to differ among extractions, samples, or size fractions. Iron was also determined in the extracts of the crushed samples of procedure 2.

Thin sections were prepared from undisturbed, oven-dried (110°C), samples impregnated under vacuum with styrene and laminac resin as described by Buol and Fadness (1961).

Results and Discussion

Comparing the 2-0.5-mm and the smaller than 0.25-mm aggregates, the free-Fe content decreased from 21.7% to 19.4%, the sand content decreased from 9.8% to 7.4%, the clay content decreased from 85.3% to 77.9% and the silt content increased from 4.8% to 14.6%. The 0.5-0.25-mm aggregates and the total soil were intermediate in all analyses.

No differences were noted in total chemical composition among aggregate size fractions, although relatively high P and Ti contents were noted in all samples (Table 2).

There was a slight inverse relation between pH values, both in water and KCl, and aggregate size (Table 3). The pH values in water were always greater than those in N KCl. Mechanical alteration (crushing) of the structure increased the pH values in all samples but the increase was never more than 0.25 of a pH unit. No exchangeable H was detected. Exchangeable Al content was low but increased in all samples when they were crushed.

Acid-extractable Ca, Mg, and P contents were determined by the standard Brazilian procedure increased as aggregate size decreased. The crushed samples gave higher analytical results than the respective uncrushed aggregates (Table 4) suggesting that the arrangement of the individual particles in aggregates, all less than 2mm in diameter, slows the extraction of basic cations and P from this soil. The results of K determinations did not show any difference either upon crushing or among size fraction. This apparent anomaly was attributed to a fire the year previous to sampling which probably contributed a sizeable quantity of K to the underlying soil.

Increasing the shaking time (procedure 2) generally increased the amount of Ca, Mg, and P extracted. In most cases, contents of these elements also increased with decreasing aggregate size (Table 4). The effect of crushing was very evident on the contents of extractable P and to a much lesser extent on content of extractable Ca and Mg. The acid-extractable K varied from 0.54 to 0.64 me/100 g of soil but it did not follow any trend. Portions

of each extract from the crushed samples were analyzed for Fe content. None of the extracts exceeded 0.009% Fe. Variation in Fe content of the extracts was not related to differences in acid-extractable Ca or P, suggesting that no iron interaction was involved in the P release.

The results from procedure 2 suggests that the larger aggregates presented more resistance to the acid extraction of P by shaking than the smaller aggregates. The differences in extractable P as related to aggregate size were reduced by increasing the shaking time but this was not true for extractable Ca. Increased shaking time, up to 72 hours, failed to eliminate differences related to crushing (Table 4).

The P extracted by the 0.025 N $(NH_4)_2SO_4$ (procedure 3) is referred to here as adsorbed P. Increasing time of shaking increased the release of adsorbed P, with maximum extraction occurring at about 24 hours of shaking. The effect of aggregate size was not well defined but crushing increased release of adsorbed P in every comparison (Table 5).

Assuming that P extracted by 0.025 N $(NH_4)_2SO_4$ is the adsorbed fraction of the total P extracted by the weak acid, the relative contribution of adsorbed P to the acid-extractable P was maximum at the beginning of the shaking and gradually decreasing as the shaking time advanced, with some exceptions noted in the crushed samples (Tables 4 and 5).

Exhaustive extractions by leaching (procedure 4) did not eliminate the increase in acid-extractable Ca, Mg and P imposed by the mechanical alteration of the structure (Figure 1). Release of extractable Ca approached zero after the fourth extraction. The difference between

natural and crushed samples was maximum in the 0.5-0.25-mm aggregates and smallest in the whole soil and in aggregates smaller than 0.25 mm. The total amount of Ca extracted was greater in the small peds than in the larger peds as was the result with the standard extraction (Table 4). The whole soil yielded intermediate results. Extractable Mg followed about the same trends as Ca, with amounts in the extracts approaching zero after three extractions. Aggregates smaller than 0.25 mm released more acid-extractable Mg than the larger peds. These results suggested that four successive extractions were enough to evaluate the acid-extractable Ca and Mg in this soil.

Extractable P continued to be released at about the same rate, after the first two extractions, through eight successive extractions. After the first two extractions, each extract yielded about 16 ppm P. The first two extractions had lower concentrations and appeared to reflect a slightly greater dependency upon mechanical alteration of the structure than was evident in later extractions. Crushing did not appear to significantly influence the rate of P release in successive extractions.

From the examination of thin sections under the polarizing microscope it is possible to say that the soil is composed fo discrete small peds separated by compound packing voids (Brewer, 1964). Internally the peds have their high fine clay (<0.2μ) and iron oxide contents compacted and arranged in a semi-concentric fabric (Figure 2). These granular-shaped peds contain small grains of quartz and opaque magnetic minerals.

The semi-concentric oriented nature of the ped fabric (Figure 1) may be caused by the wetting and drying under the monsoon nature of the climate.

The semi-concentric arrangement was most evicent in the upper portion of the profiles. There was no evidence of illuvial argillans, but charcoal with cracks filled with clay and iron oxide is common in thin sections from the site suggesting that clay and iron are mobile to some extent in the profile.

From these and other observations a mechanism where by bases accumulate in the fine, firm, granular peds of this Eutrustox is postulated as follows. The soil is formed in a warm, humid climate of about 1600-mm mean annual rainfall with a pronounced three-month dry season. During the dry season the peds desiccate, by evaporation, to moisture contents somewhat less than permanent wilting. At the onset of the rainy season, ions derived from decomposing vegetation at the soil surface move into the dry peds from percolating soil solution. The soil solution once in the ped interior is held at high capillary tensions due to the extremely small particles and arrangement of the plasma. Fifteen-bar moisture content is about 25% on a volume basis; total porosity is greater than 55% of the soil volume with more than half of it composed of pores smaller than 0.05 mm; bulk density is about 1.0 g/cc; and more than 70% of the particles are smaller than 0.2μ[3/]. Liquid phase movement of water from ped interiors to the larger inter-ped pores or root channels would be very slow or nonexistent thus most of the water is postulated to move out of the peds as vapor, during the dry season, leaving the bases behind. Leaching would be ineffective in removing these bases from the ped interior, since the percolating water occupies the large proes and has been measured to move at infiltration rates of 60 cm/hour[3/], thus leaching only the ped surfaces (Table 3). This mechanism acting over the years is hypothesized

to be responsible for the presence of high base status in this mineralogically highly weathered soil.

Conclusions

Total chemical composition does not change with aggregate size. There appears to be a slight concentration of silt in the finer aggregates (<0.25 mm) at the expense of both sand and clay.

Mechanical alteration of the microstructure caused an increase in acid-extractable P and in most cases also in the amount of Ca and Mg extracted. This difference could not be completely overcome by any of the the procedures used. Alteration of the structure also increases the amount of exchangeable Al measured and the pH values. The amounts of acid-extractable Ca, Mg and P were found to be greater from small aggregates than from larger ones. Thus, the reproducibility of the routine standard Brazilian procedure for soil test extractions of P from this soil may be affected by both aggregate segregation and crushing in sample preparation. It is suggested that either increasing the shaking time or the use of successive extractions will tend to increase reproducibility. The latter being easier and faster is recommended for further research.

High base status is postulated to be the result of entrapment of bases within peds as percolating water wets the soil after the prolonged dry season. The intra-ped pores are so small that leaching within the peds is retarded and water leaves the peds primarily as a vapor, thus not removing the bases.

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Table I. Soil Profile Description.

Classification: Typic Eutrustox (Latosol Roxo)
Position: Middle of a long, gentle slope
Altitude: 480 m
Relief: Gently undulating, long 2% slope, slightly convex landscape
Parent material: Basaltic lava
Drainage class: Well drained
Erosion: Uneroded
Vegetation: Tropical seasonal rainforest (cut in November, 1966); presently cotton and rice
Climate: 1,600 mm mean annual rainfall; 21°C mean annual temperature
Description by: Camargo, W. Moura Filho, and S. W. Buol
Date: 7/27/68 (dry season)

Profile Description

A1	0- 15 cm	Very dusky red (10R 2/3-moist) dusky red (10R 3/3-crushed moist) dark reddish-brown (2.5YR 3/4-dry) and yellowish-red (5YR 4/7-crushed dry) clay; moderate coarse to very fine granular and subangular blocky structure; slightly hard; very firable; very sticky and plastic; abundant roots (secondary roots of about 1 cm diameter and fine roots); gradual and smooth boundary.
A3	15- 40 cm	Dusky red (10R 3/3-moist) dusky red (10R 3/3-crushed moist) dark reddish-brown (2.5Y 3/4-dry) and yellowish-red (5YR 4/8-crushed dry) clay; weak coarse broken into strong fine granular and subangular blocky structure; slightly hard; very friable; very sticky and plastic; common roots (secondary roots of about 1 cm diameter and fine roots); diffuse and smooth boundary.
B1	40- 60 cm	Dusky red (10R 3/3-moist) and yellowish-red (5YR 4/8-crushed dry) clay; strong medium to very fine granular and subangular blocky structure, and presence of extremely firm clay nodules of 5-10 mm diameter; soft; very friable; very sticky and plastic; roots common (secondary roots of about 1 cm diameter and fine roots); diffuse and smooth boundary.
B21	60- 110 cm	Dusky red (10R 3/4-moist) and yellowish-red (5YR 4/8-crushed dry) clay; strong very fine granular structure; soft; very friable; very sticky and plastic; few roots (predominantly secondary roots of 0.5 - 1.0 cm diameter); diffuse and smooth boundary.
B22	110-180 cm+	Dusky red (10R 3/5-moist) and yellowish red (5YR 4/8-crushed dry) clay; strong very fine granular structure; soft; very friable; very sticky and plastic; few roots (predominantly secondary roots of 0.5 - 1.0 cm diameter).

Observed porosity: Many pores smaller than 1 mm diameter throughout the entire profile, and few channels of about 5 mm diameter in A1, A3, B1, and B21.

Table 2. Results of the total dissolution of total soil and different sized peds.

	SiO_2	Al_2O_3	$\frac{SiO_2}{Al_2O_3}$	Fe_2O_3	MnO	TiO_2	CaO	MgO	P_2O_5	K_2O	loss on ignition
	%	%	ratio	%	%	%	%	%	%	%	%
Total soil	21.3	26.6	1.4	26.8	0.25	10.5	2.3	0.26	0.75	0.73	13.18
2.0 mm–0.5 mm	21.3	28.5	1.3	27.2	0.25	10.6	2.3	0.25	0.75	0.71	11.98
0.5–0.25 mm	21.0	27.1	1.3	26.8	0.23	11.0	2.3	0.26	0.76	0.75	12.39
<0.25 mm	21.6	27.1	1.4	26.3	0.23	11.0	2.3	0.27	0.75	0.75	12.06

Table 3. Results from acid extraction by the standard procedure showing the effect of aggregate size and mechanical destruction of the structure on ion release and pH value.

Sample	Al	Ca	Mg	K	P	pH value 1:1 water	pH value 1:1 M KCl
	me/100 g soil				ppm		
Whole sample							
natural	0.56	7.50	2.17	0.53	9.4	5.40	5.30
crushed	0.83	7.75	2.21	0.56	13.0	5.60	5.40
2.0-0.5 mm							
natural	0.68	7.25	2.21	0.58	7.5	5.32	5.20
crushed	0.79	8.25	2.29	0.56	10.0	5.57	5.40
0.5-0.25mm							
natural	0.64	7.50	2.21	0.57	9.5	5.42	5.30
crushed	0.87	8.50	2.33	0.57	15.0	5.65	5.50
<0.25 mm							
natural	0.64	9.00	2.37	0.58	11.3	5.60	5.43
crushed	0.89	10.00	2.46	0.56	17.5	5.70	5.55

Table 4. Results from acid extraction by procedure 2 showing the effect of varying shaking time, aggregate size and mechanical destruction of the structure on the release of Ca, Mg and P

Sample	Shaking time					Shaking time					Shaking time				
	5 min	1 hr	6 hr	24 hr	72 hr	5 min	1 hr	6 hr	24 hr	72 hr	5 min	1 hr	6 hr	24 hr	72 hr
	Ca (me/100 g soil)					Mg (me/100 g soil)					P (ppm)				
Whole sample															
Natural	6.75	6.75	7.37	7.37	7.75	2.00	2.06	2.16	2.33	2.54	4.4	6.3	8.1	12.5	15.0
Crushed	6.25	6.75	7.25	7.50	8.00	1.90	2.00	2.10	2.15	2.20	8.7	10.0	11.2	13.1	20.0
2.0-0.5 mm															
Natural	6.62	6.62	6.87	7.25	7.50	1.94	2.08	2.25	2.37	2.54	4.4	6.3	8.1	11.3	15.0
Crushed	6.50	6.75	7.25	7.50	8.00	2.05	2.17	2.25	2.25	2.25	5.6	7.5	9.4	12.5	16.9
0.5-0.25 mm															
Natural	6.60	6.87	6.75	7.25	7.50	2.02	2.10	2.25	2.37	2.50	5.0	6.9	9.4	12.5	15.0
Crushed	6.50	7.25	7.50	8.00	8.27	2.05	2.27	2.27	2.27	2.27	10.0	11.2	13.1	16.2	20.6
<0.25 mm															
Natural	8.00	8.50	8.50	9.00	9.00	2.20	2.33	2.58	2.66	2.66	7.5	9.4	11.3	13.5	15.6
Crushed	8.00	8.50	9.25	9.75	10.00	2.20	2.40	2.40	2.40	2.40	11.2	11.9	13.7	16.2	20.6

Table 5. Data from the 0.025 N $(NH_4)_2SO_4$ extractions by procedure 3 showing the effect of varying shaking time, aggregate size, and mechanical destruction on PO_4'' replaced by SO_4''

Samples	Shaking time				
	5 min	1 hr	6 hr	24 hr	72 hr
	P by 0.025N $(NH_4)_2SO_4$ extr.				
	ppm				
Whole Soil					
natural	2.2	2.2	2.8	3.0	3.4
crushed	2.7	2.8	5.2	5.7	5.0
2-0.5 mm					
natural	1.2	2.0	2.8	3.0	2.8
crushed	1.7	2.2	4.0	4.4	3.8
0.5-0.25 mm					
natural	1.2	2.0	2.8	3.2	2.7
crushed	2.8	3.2	5.7	7.0	6.2
<0.25 mm					
natural	1.7	2.2	2.8	3.5	3.0
crushed	2.8	3.2	5.2	6.0	5.5

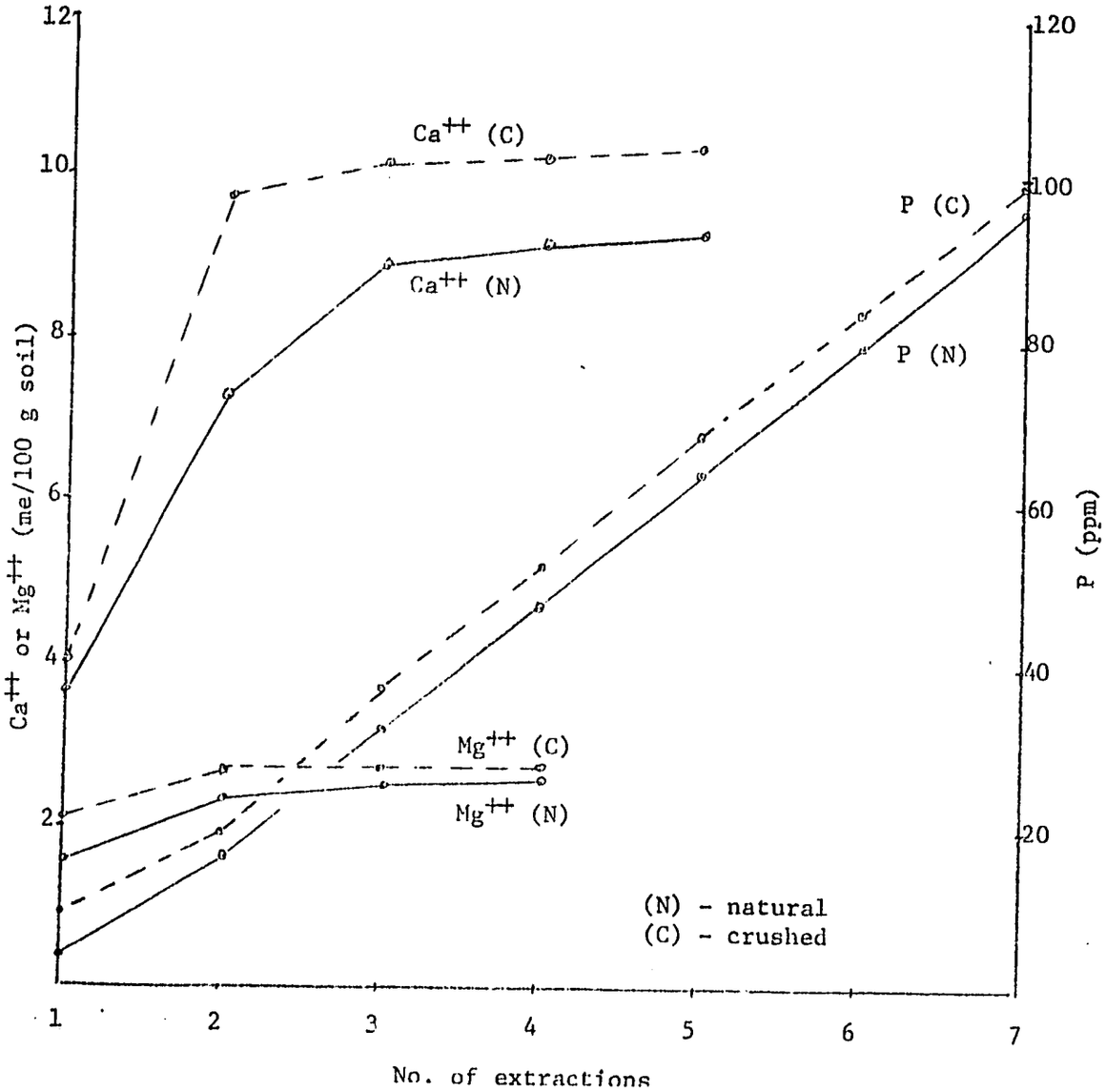

Figure 1. Cumulative curves showing results from exhaustive extractions in leaching columns (procedure 4) of the whole soil

Figure 2. Photomicrograph under crossed polarizers showing the semi-concentric fabric of a ped in the Eutrustox soil. 380X Magnification