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Electron and petrographic microscopy applied to soil micromorphology

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INTRODUCTION

This work is a parallel study of the soil plasma fraction, which occurs as particles less than 2 μ in diameter, with the application of the petrographic and electron microscopes.

With the first technique we can observe mainly the colour, quantity and distribution, orientation of the plasma grains, the nature of the plasma separations and to have an idea of the nature of the constituents.

The electron microscope allows us to identify the components: clay minerals, salts, oxides and hydroxides ... etc. and still, the most important, it shows the shape and disposition of them *in situ*.

For the first work of this research line, we have made a selection of quite different soil types, from Braunlehm to Braunerde and we have paid special attention to the study of iron oxydes and hydroxides, association of clay minerals, crystallinity and size homogeneity. The knowledge of some of these properties, actually, are only possible with the application of the electron microscope.

The finality of this study is to have some insight in the soil plasma fraction as we cannot get it with the petrographic microscope when we consider its resolving power and the size of the plasma constituents. The knowledge of the nature, size and disposition of the plasma grains, will allow us to deduce the processes that have taken place in the genesis and development of soil.

TECHNIQUES USED IN THE ANALYSIS OF SOIL MATERIALS

1. MICROMORPHOLOGY ANALYSIS

Thin sections were obtained from the soil materials by impregnation of the samples with a polyester resin (Cronolita 1.108) following the conventional methods [4] and afterwards were examined under the petrographic microscope.

2. ELECTRON MICROSCOPE

The soil specimens have been either directly studied from a suspension in destilled water or making use of the replica technique. In the first case, all the minerals present in the sample were identified.

In the case of the replica technique there have been used two variants: the direct carbon replica and the composite carbontriafol techniques. When the direct replica technique is used the carbon film is broken by dispersion in the water when it is removed from the clay. To avoid this problem the carbon replica is protected with parlodion, which later is eliminated by dissolving it in amyl-acetate. In relation to the composite replica it was necessary to take great precautions to avoid the soil disintegration in the stripping of the triafol replica; so, in the mounting of the triafol replica, a soft support of cotton was employed.

Some specimens were studied under two Philips electron microscopes (models 200 and 300).

MATERIAL STUDIED

Sample 1. Location: km 26,200 road Ciudad Real — Valdepeñas (Ciudad Real). Rainfall: 597.93 mm. Average annual of the maximum temperatures 36.46°C, average annual of minimum temperatures 0.41°C.

Samples 2 and 3. Location: Villanueva da la Serena (Badajoz). Parent material: Slate (Cambrian). Rainfall: 569.3 mm. Average max. temps. 28.04°C, average min. temps. 7.91°C.

Samples 4 and 5. Location: Villafranca de los Barros (Badajoz). Parent material: Metamorphic schist (Palaeozoic). Rainfall: 501 mm. Average max. temps. 27.21°C, average min. temps. 7.72°C.

Sample 6. Location: Altos del Minguete (Soria). Parent material: Limestone (Miocene). Rainfall: 668.1 mm. Average max. temps. 18.3°C, average min. temps. 5.8°C. Material taken at 1.50 m depth, between layers of the original material.

Sample 7. Location: km 8.5 road Puente de Arganda-Cinchón (Madrid). Parent material: Limestone (upper Miocene). Rainfall: 440.4 mm. Average max. temps. 21.3°C, average min. temps. 7.3°C.

Sample 8. Location: Ciudad Encantada (Cuenca). Parent material: Limestone (middle Cretaceous). Rainfall: 984 mm. Average max. temps. 18.1°C, average min. temps. 5.8°C.

Sample 9. Location: Anzola (Vizcaya). Parent material: Limestone (Cretaceous). Rainfall: 1,053.6 mm. Average max. temps. 18.4°C, average min. temps. 8.3°C.

Sample 10. Location: km 20 road Evinayong — Kogo (Equatorial Guinea). Parent material: Granite (Palaezoic). Rainfall: about 2,500 mm. Average annual temp. about 26°C ranging between 20°C and 30°C.

Sample 11. Location: Campillo de Llerena (Badajoz). Parent material:

Slate (Silurian). Rainfall: 449 mm. Average max. temps. 27.01°C, average min. temps. 7.72°C.

Sample 12. The same location as sample 7. The material is considered a soil sediment in this case.

Sample 13. Location: Santiago, El Barco de Valdeorras (Orense). Parent material: Slate (Silurian). Rainfall: 660 mm. Average max. temps. 18°C, average min. temps. 7.5°C.

Sample 14. Location: Poyales (Sierra de Gredos). Parent material: Granite (Acid plutonic rocks, geological map of Spain, scale 1:1,000,000) [6]. Rainfall: 1,640 mm. Average max. temps. 26.2°C, average min. temps. 5.99°C.

MICROSCOPIC STUDY OF THE PLASMA

Sample 1. Fossil sediment of Rotlehm. Plasma has a high luster and smooth surface (under a hand lens).

Thin section (petrographic microscope): Plasma dominant over skeleton grains, red (10 R 5/8) colour, good birefringence between crossed nicols, abundant doubly refractive streaks [7] (Plate I, Fig. 1). Deep chemical weathering of the soil.

Electron microscope: Montmorillonite — kaolinite predominant. The intimate union between montmorillonite and acicular goethite forms of different development is significant for the plasma aspect and colour origin; besides it is worth denoting the presence of bundles and large fibres of sepiolite (Plate I, Fig. 3) crossed among themselves (confirmed in the replica soil technique). The kaolinite observed is of a size really small forming agglomerates of difficult dispersion, there is a great bonding force between the mineral constituents; also a geliform-like fraction not identified and microcarbonates exist, the composition proving the double origin of the parent material (sedimentary rock influenced by volcanic sediments).

Sample 2. Rotlehm, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Abundant plasma, red (2.5 YR 4/8) colour, good birefringence, abundant doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: The clay minerals found are the typical ones from a slate: heterometric micas, thin illites, and feldspars; also frequent sepiolite — attapulgite fibres are observed in quite diverse fields of the slide. The presence of the last mineral species with its great adsorption capacity must have an influence on some soil physical properties.

There are also kaolinite and microgoethite but the iron predominates in the turite form, more or less spongy. The homogeneity is good in two levels: one of microkaolinites and the other of the rest of the components being the attapulgite fibres the largest. The crystallinity is low.

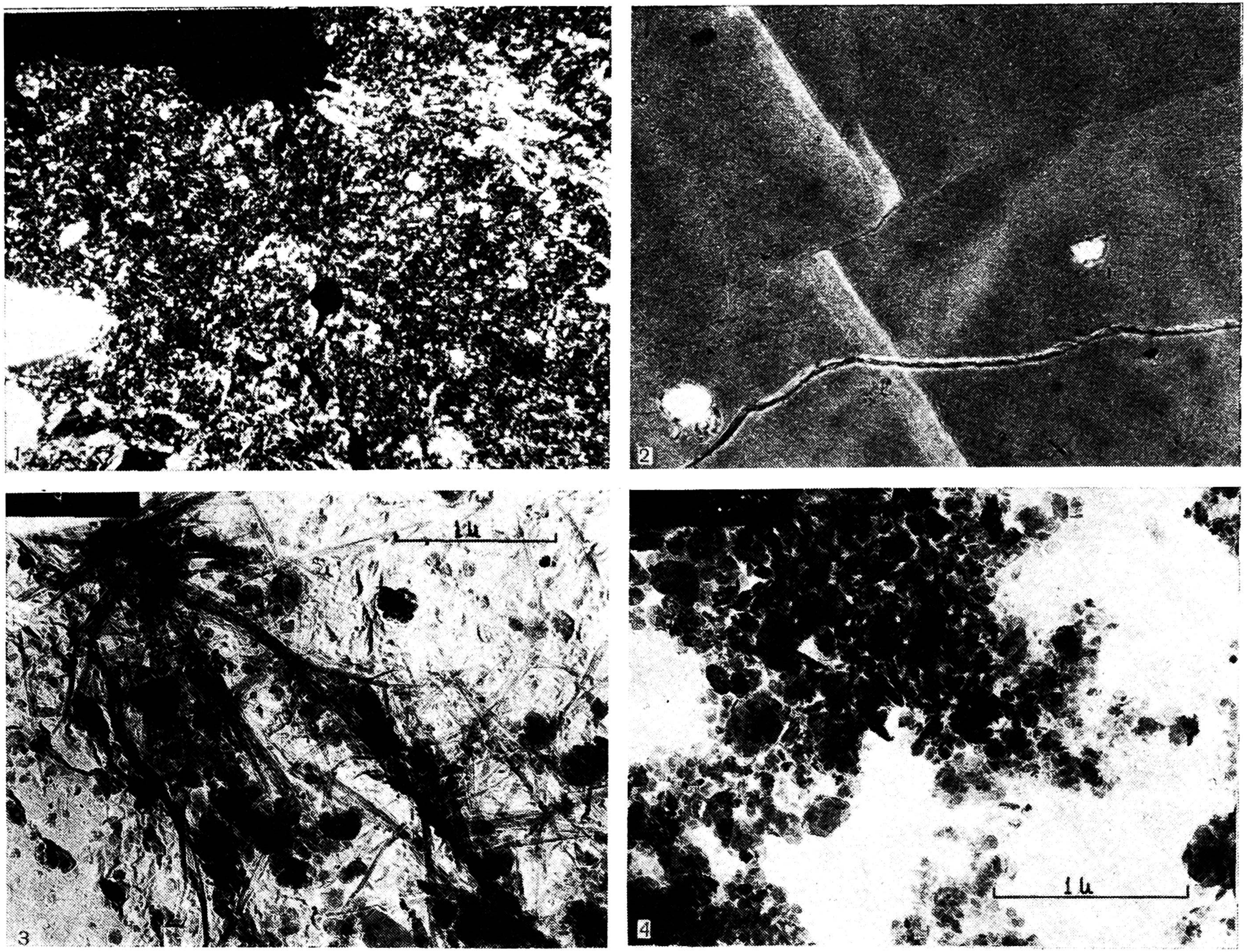

Plate I. Fig. 1. Doubly refractive streaks are clearly seen. Thin section under crossed polarizers. Sample 1; × 130. Fig. 2. Plasma dominant over skeleton grains. Thin section. Sample 6; × 130. Fig. 3. Sepiolite fibers crossed among themselves showing a trend towards a fluidal disposition. Replica technique. Sample 1; × 30,800. Fig. 4. Abundance of kaolinite intermixed with goethite. Normal dispersion technique. Sample 6; × 37,600.

Sample 3. Browned rotlehm, A horizon. Plasma has low luster and rough surface.

Thin section: Abundant plasma, red (2.5 YR 4/6) colour, good birefringence, there are not doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: Mica and illite are predominant, there are abundant iron spots throughout all the slide in the goethite form, as well as in the turite form, the last species shows a tendency to get the shape of acicular goethite; the presence of attapulgite persists. The size of the kaolinite is larger, the sample is less homogeneous and the crystallinity higher than in the (B) horizon.

Sample 4. Rotlehm, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Abundant plasma, red (2.5 YR 4/6) colour, good birefringence, abundant doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: The material consists of montmorillonite, illite, scarce kaolinite and rounded heterometric micas. Montmorillonite is found in two different grades of crystallinity, the same as it happens with muscovite and biotite. The iron is mainly grain shaped turite, frequently it is adsorbed by montmorillonite; there are scarce anatase or rutile and it is frequent to observe fragmented biotites.

The proportion of turite is scarce and shows certain tendency to give microneedles (turite-goethite). There is presence of undetermined salts.

Sample 5. Browned rotlehm, A horizon. Plasma has low luster and rough surface.

Thin section: Abundant plasma, red (2.5 YR 5/6) colour, good birefringence, scarce doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: Montmorillonite is still present, as in the above sample and shows tendency to include turite and salts in its own mass. In this horizon it seems to be higher increase of goethite than in the (B) horizon.

Sample 6. Terra rossa, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Dominant plasma (Plate I, Fig. 2), red (2.5 YR 5/8) colour, good birefringence, abundant doubly, refractive streaks. Slight chemical weathering of the soil.

Electron microscope: In this sample, perhaps the most compact among the ones studied, we have applied all the techniques mentioned in this paper obtaining promising results and it seems to corroborate the possibilities of this research line. So in an ordinary dispersion we see that heterometric kaolinite prevails (Plate I, Fig. 4) though generally of small size and from middle to good crystallinity; there is an intimate association of iron with the above mineral being the iron predominantly goethite according to the replica forms after Beutelspacher and Van der Marel [2]. There is as well broad turite in aggregates keeping its own individuality within the aggregates; mica is also observed becoming kaolinite in some cases.

In the replica technique, we have seen absence of cavities and compactness of kaolinites around which we found needles of goethite-hematite (Plate II, Figs. 1 and 2).

Sample 7. Terra rossa, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Dominant plasma, red (2.5 YR 5/8) colour, good birefringence, abundant doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: This sample because its colour and compactness is quite similar to number 6 and it has also been throughly studied. In the direct observation we identify illite, some thin mica and other ones dark thick and heterometric, turite, salts, kaolinite of low and high crystallinity showing great heterometry, and smaller proportion of iron microneedles (goethite) than in the above sample.

Here we find turite grains throughout all the sample. There is scarce quantity of montmorillonite and quartz, as well as undetermined salts. Turite shows certain trend to the crystallinity.

In the replica technique we see the great relation between the above quoted minerals and the fluidal disposition of iron which encloses mainly kaolinites and illites.

Sample 8. Terra rossa, (B) horizon. Plasma has low luster and rough surface.

Thin section: Abundant plasma, yellowish red (5 YR 5/8) colour, scarce birefringence, common doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: Illite and some kaolinite are found but the sample is characterized by the turite and goethite arrangement in microaggregates, both of them are in similar proportion. In some of the aggregates we have observed needles of goethite in parallel disposition. The average crystallinity of the clay minerals is lower than in samples 6 and 7.

Sample 9. Terra fusca, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Abundant plasma, strong brown (7.5 YR 5/8) colour, good birefringence, abundant doubly refractive streaks. Deep chemical weathering of the soil.

Electron microscope: In normal dispersion, illite is identified as the predominant clay mineral, there are also many micas; kaolinite and possible chlorite are present, both of them with the morphology previously observed in the insoluble residue of triassic limestones [1, 10].

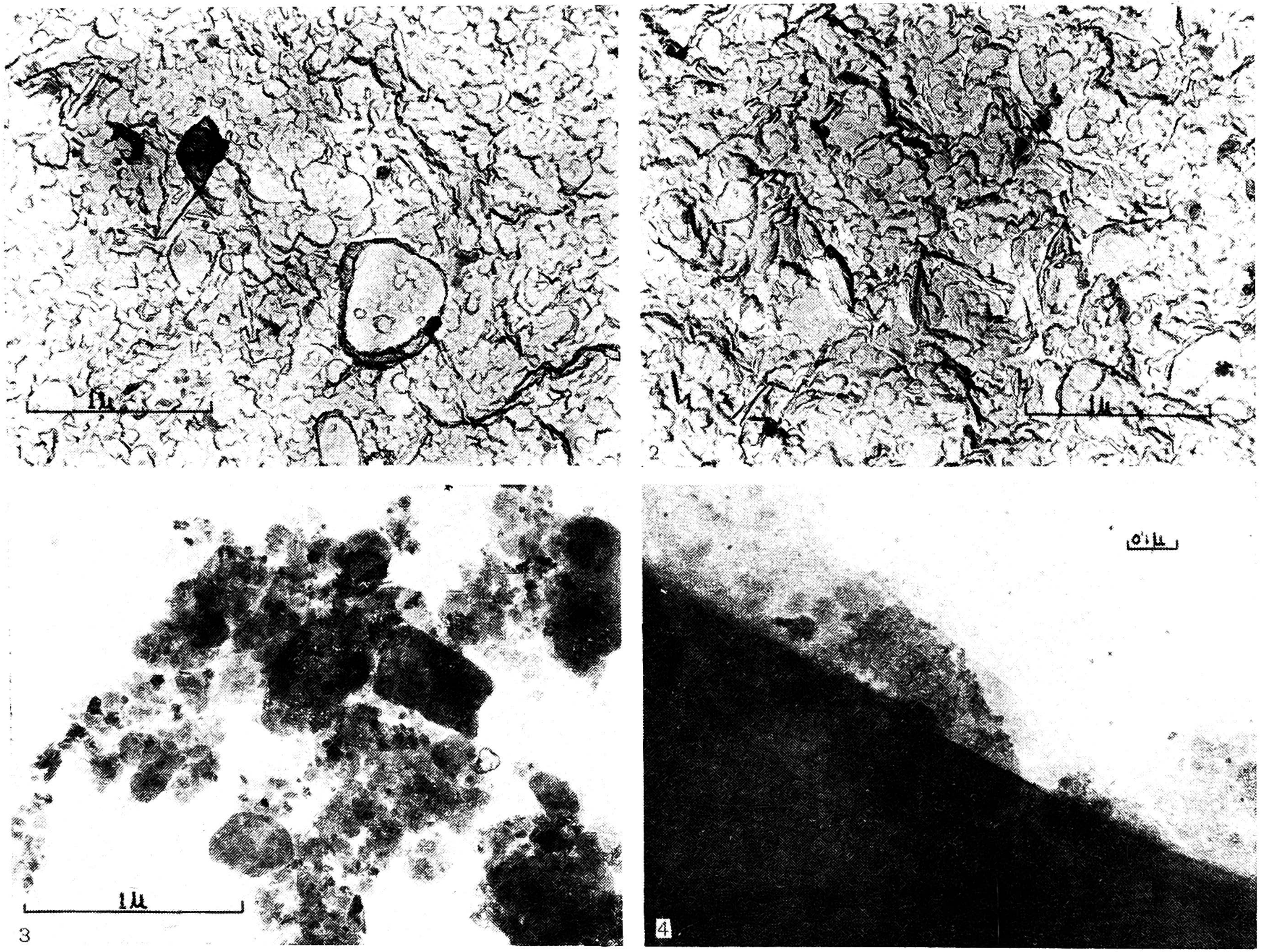

Plate II. Figs. 1 and 2. The sample is constituted practically by heterometric kaolinite and needles of goethite; there is absence of cavities. Replica technique. Sample 6; × 34,500. Fig. 3. Kaolinite with large size and rounded forms of mica and illite; the iron is adsorbed on these minerals. Normal dispersion technique. Sample 11; × 41,400. Fig. 4. Turite between muscovite flakes. Normal dispersion technique. Sample 14; × 99,000.

Among the iron minerals turite is dominant, frequently it shows high crystallinity; there is a minor proportion of goethite, this hydroxide is found in the shape of needles arranged more or less in parallel.

Using the replica technique we see the neat morphology of thin kaolinite with variable size, large mica flakes and the iron throughout all the sample around the just mentioned minerals.

Sample 10. Equatorial braunlehm, (B) horizon. Plasma has low luster and rough surface.

Thin section: Abundant plasma, brownish yellow (10 YR 6/8) colour, scarce birefringence, abundant doubly refractive streaks. Deep chemical weathering of the soil.

Electron microscope: Heterometric kaolinites are predominant existing large crystals with high crystallinity as well as incipient hexagons, so some of this form could be attributed to boehmite; halloysite is scarce and there are micas as well. Perhaps the most significant fact is the iron abundance, being the turite in very small grains predominant together with a gel like substance; the amount of goethite is low. Practically the turite and the gel are associated with the clay minerals.

Sample 11. Pseudogley braunlehm, (B) horizon. Plasma has a high luster and smooth surface.

Thin section: Dominant plasma, the colour ranges from brownish yellow (10 YR 6/8) to red (2.5 YR 4/8), good birefringence, abundant doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: There is much iron prevailing turite which shows certain tendency to pass into goethite. We find iron adsorbed on the clay minerals. There is also iron in the goethite-hematite form. The kaolinite is very typical with pronounced heterometry and there are also rounded mica and illite (Plate II, Fig. 3). The crystallinity is high.

Sample 12. Earthy braunlehm, (B) horizon. Plasma has no luster and rough surface.

Thin section: Very scarce plasma, yellowish red (5 YR 4/8) colour, good birefringence, abundant doubly refractive streaks. Very slight chemical weathering of the soil.

Electron microscope: The sample appears in direct observation as constituted by illite, thin and dark heterometric micas and neat kaolinite. With the double replica technique we have seen thin and angular frequently fragmented micas with minerals inside, well developped kaolinites and iron. This iron appears in two typical forms: one mobile in goethite-hematite streaks around the micas and illite, and the other as turite aggregates. It is worth denoting the absence of cavities.

Sample 13. Braunerde, (B) horizon. Plasma has no luster and rough surface.

Thin section: Common plasma, predominantly of dark brown (7.5 YR 4/4) colour, due to the abundance of amorphous flocculated iron [7], good birefringence, there are no doubly refractive streaks. Slight chemical weathering of the soil.

Electron microscope: The sample has different morphology from the ones already described. The mica is the most abundant mineral with very different shapes and electron transparency..... etc. We find also illite, kaolinite, vermiculite and little halloysite.

The iron appears in three forms: as a gel adsorbed mainly on the micas, as an heterometric goethite and as turite. The last form shows a high crystallinity even so the size of either grains or needles is incipient. We have seen also goethite twins inside altered micas in the shape of star-like and turite-goethite aggregates.

Sample 14. Braunerde, A horizon. Plasma has no luster and rough surface.

Thin section: Very scarce plasma, predominantly of yellowish red (5 YR 4/8) colour, scarce birefringence, there are no doubly refractive streaks. Very slight chemical weathering of the soil.

Electron microscope: Similar to sample 13, it contains more or less opaque big flakes of mica. Turite is also very much alike to the last sample but the tendency to be transformed into goethite is not so clear. We find turite between muscovite flakes (Plate II, Fig. 4), it is more typical and does not show the needle shape. Needles of goethite are observed and there are fragmented micas, kaolinite and scare halloysite.

DISCUSSION AND CONCLUSIONS

Due to the lack of papers on this subject and because of the great difference that there is between the clay minerals in suspension and the same minerals within the soil fabric, perhaps there would occasionally be possible other interpretations of the results.

In the studies with the electron microscope, an iron mineral appears very often; on account of its morphology (Plate II, Fig. 4), we think that it is turite: "a fine-grained or very fine-grained physical mixture of hydrohematite (sometimes hematite also) and goethite-hidrogoethite" [11].

The data stated in Table 1 are ordered mainly by its abundance. It is observed the presence of a gel-like fraction that could be iron in the samples 10-13 and 14 which corresponds to Braunlehm and Braunerde soils. It is possible that in the first sample the gel is amorphous peptized iron hydroxide while in the last ones it is amorphous flocculated iron hydroxide, as it has been identified [9] using other techniques.

We have only noticed mobile iron, under the electron microscope, in the samples 6 and 12. As a matter of fact, these samples show also doubly refractive streaks in their thin section, but we found as well the same phenomenon in many other samples.

In a work on very similar soils, Hoyos and Rodriguez Sanchidrian [5]

Table 1. Soil classification and plasma constituents

Sample	Horizon (depth, cm)	Colour (thin section)	Classification acc. Kubiëna	Parent material	Iron minerals	Clay minerals
1	E/ (B) fos, 390–440 cm	Red (10 R 5/8)	Fossil sediment of rotlehm	—	Goethite	Kaolinite, montmorillonite, sepiolite
2	(B), 25–55cm	Red (2,5 YR 4/8)	Rotlehm	Slate	Turite-hematite, goethite	Mica, illite, kaolinite sepiolite-attapulgite
3	A, 0–25cm	Red (2,5 YR 4/6)	Browned rotlehm	Slate	Turite, goethite	Mica, illite, kaolinite, attapulgite
4	(B), 20–40 cm	Red (2,5 YR 4/6)	Rotlehm	Metamorphic schist	Turite	Montmorillonite, illite
5	A, 0–20 cm	Red (2,5 YR 5/6)	Browned rotlehm	Metamorphic schist	Turite, goethite	Montmorillonite, illite
6	(B), c. 150 cm	Red (2,5 YR 5/8)	Terra rossa	Limestone	Goethite-hematite, turite	Kaolinite
7	(B), c. 400 cm	Red (2,5 YR 5/8)	Terra rossa	Limestone	Turite, goethite	Illite, mica, kaolinite, salts
8	(B), 2–20 cm	Yellowish red (5 YR 5/8)	Terra rossa	Limestone	Turite, goethite	Illite, kaolinite
9	(B), 25–75 cm	Strong brown (7,5 YR 5/8)	Terra fusca	Limestone	Turite, goethite	Illite, chlorite, kaolinite, mica
10	(B), 20–40 cm	Brownish yellow (10 YR 6/8)	Braunlehm	Granite	Gel, turite, goethite	Kaolinite, halloysite, mica
11	(B), 30–85 cm	Brownish yellow (10 YR 6/8) Red (2,5 YR 4/8)	Pseudogley braunlehm	Slate	Turite-goethite,hematite	Kaolinite, mica, illite
12	(B), 20–50 cm	Yellowish red (5 YR 4/8)	Sediment of earthy braunlehm	—	Goethite-hematite, turite	Illite, mica, kaolinite
13	(B), 10–75 cm	Dark brown (7,5 YR 4/4)	Braunerde	Slate	Gel, turite-goethite	Mica, kaolinite, halloysite
14	A, 0–5 cm	Yellowish red (5 YR 4/8)	Braunerde	Granite	Turite, goethite, gel	Mica, halloysite, kaolinite

found, from the high proportion of free F_2O_3, total F_2O_3 and from the illuviation index calculated for iron, silica and aluminium that the iron remains in the state of hydrated oxides (which could be our turite), without being converted into crystalline compounds: i.e. stable hydroxides that stay *in situ*, while the aluminium and silica have a higher mobility as adsorbed colloids on the clay minerals. So, the plasma mobility seen in thin section is due partially to the iron and partially to the silica, aluminium and sepiolite. Whether or not, each part is going to be mobilized depends on soil factors, such as pH, redox potential, etc.

In the comparation of two horizons of rotlehm (samples 2 and 4) and browned rotlehm (samples 3 and 5) within the same profile we have at first sight a change in the colour, though they keep the same hue. Another difference observed in the studies with the electron microscope, is the higher crystallization that appears in the browned rotlehm; this fact is attributed to a possible partial dehydratation of the iron compounds, that takes place under the actual climate, drier than it is necessary for the genesis of a rotlehm.

There is also a difference in the size of the minerals; it is appreciated an obvious heterometry in the browned rotlehm. The possible cause of this fact is the allochtonous material of these horizons.

With respect to the correlation among colour, iron and clay minerals, there is not a clear interconnexion: the colour is a complex factor depending mainly on the crystallization of the iron minerals, their connection with the clay minerals (in some cases there is a very close union) and the nature of the clay and other plasma constituents (organic substance, Ti and Mn oxides... etc.).

In the Terra rossa samples with an intense red colour we appreciated the abundance of microcrystalline kaolinite and its high iron content (mainly goethite).

In relation with the birefringence of the plasma in thin section, it is induced not only by the anisotropy of its constituents but also by the thickness of the slide, due to the orientation of the soil particles [3]. Samples 10 and 14 have a low birefringence among the ones studied. Both contain iron in state of gel: in the first case all the iron is very tightly associated with the clay and in the other, turite shows a low crystallinity.

We have tried to see the analogies that show the samples according to their soil classification. Both brownerde samples show uniformity (Nos. 13 and 14): there is a preponderance of iron adsorbed on great flakes of mica, which become nearly opaque. In the clay minerals there is predominance of illite and mica with a very pronounced heterometry.

The Terra fusca has a tendency to differentiate from the other soils. It would be convenient, to study more samples to see if it keeps the same trend.

The samples of rotlehm and braunlehm do not show uniform characters like it happens in the case of the Terra rossa studied, the numbers 6 and 7 are very similar while they differ of the sample 8. It has been quoted the goethite and hematite as the predominant iron minerals in rotlehm [8, 9]. It is possible, for instance in sample 2, that a part of the turite is hematite and we do not refuse to admit that our samples could have a higher quantity of hematite, for the instruments used make the exact identification of minerals which have very small size and such a varied morphology sometimes quite difficult.

Among the clay minerals there is a predominance of kaolinite, usually of small size and pronounced homometry in the rotlehm as well as in the Terra rossa.

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SUMMARY

We carry out a parallel study on thin sections of soils by means of the usual micromorphologic and electron microscopy techniques.

The aim of the work is not only to identify the material lesser than two microns and to see its arrangement, facts beyond the resolving power of the petrographic microscope. We also make an attempt to investigate the different aspects of the iron compounds seen with both the petrographic and electron microscopes and to apply systematically the direct or double replica method to the selected soils.

We have chosen for the present paper a number of different soil types: Braunlehm, Rotlehm, Terra rossa, Terra fusca and Braunerde.

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