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IMPORTANCE OF CLAY FABRICS IN SOILS, AN APPROACH BY ELECTRON MICROSCOPY

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Abstract: This paper deals with clay microstructure studies using electron microscopy techniques. It focuses on recent developments of SEM and TEM when these are used to study clay microstructures for a better understanding of hydration, structural stability and rheological properties of clayey soil systems.
The following aspects are successively examined, (1) clay texture and (2) its consequences on hydration and shrinkage; (3) clay microstructure and its relation to structural stability and hydraulic conductivity and (4) the role of clay-associated substances in soil physical properties.

Key words: Clay, SEM, TEM, organic matter, fabric, behaviour.

Introduction

It is well known that soil texture, clay content and type of clay play a direct part in many soil properties including the dynamics of ions, the water retention properties as well as the soil structural stability. For soils with appreciable clay content the uptake and release of water gives rise to changes in bulk volume (swelling and shrinking), and this alters the clay microstructure at different levels of organization.

Changes in soil structure take place in relatively low levels of soil water tension, and especially in the domain of biological activity (tensions > – 1.6 MPa). This is also the range within which the clay constituents greatly affect the soil structure. Most of the work in literature has been limited to the study of pure clay minerals and to the clay layer and inter-layer characterization. While not ignoring the importance of the layer and interlayer, the role of clays in soils should also be concerned with crystals, crystal aggregates and their association with pore space. It was also demonstrated that clays in soils differ significantly from reference clays (Tessier & Pédro 1987; Robert et al. 1987). Understanding of highly hydrated clay water systems assumes the possibility of describing the solid phase and pores over a range of 1 nm to 1 cm. The use of both scanning and transmission electron microscopes implies the use of suitable preparation techniques that have been described and discussed elsewhere (Tessier 1984; Chenu &Tessier 1995; Kim et al. 1995).

This paper aims at demonstrating the relationship between the microstructure of soil clays and hydration and selected physical properties. The role of clay-associated substances, especially organic matter and oxides, on structural stability and the mechanical properties will also be discussed in terms of changes in microstructure.

Clay texture and fabric

1. Clay particles in soils

Recent work has shown that the clay particles of soils are different by their size and their nature. For 2:1 minerals, the < 2 μm fraction is rarely composed of individual crystals (Fig. 1a). Clay particles of 2:1 minerals are rather crystal aggregates and each aggregate results from the stacking of packets of thin *mica* crystals themselves composed of 2 to 10 layers. According to Robert et al. (1987), 3 layers per mica crystal appears to be a general feature for interstratified clays (Fig. 1b). For illites the number of layers per mica crystal reaches 5 to 10 (Fig. 1c), while for smectitic clays only hydrated layer spacings can be observed (Fig. 1d). Thus, in the case of 2:1 clays in soils a continuous solid solution from smectitic to illitic clay can be identified with all intermediate species between individual layers to thick mica crystals. It is also noticed that the 2:1 clay layers have always in soils a very limited lateral extent (<50 nm).

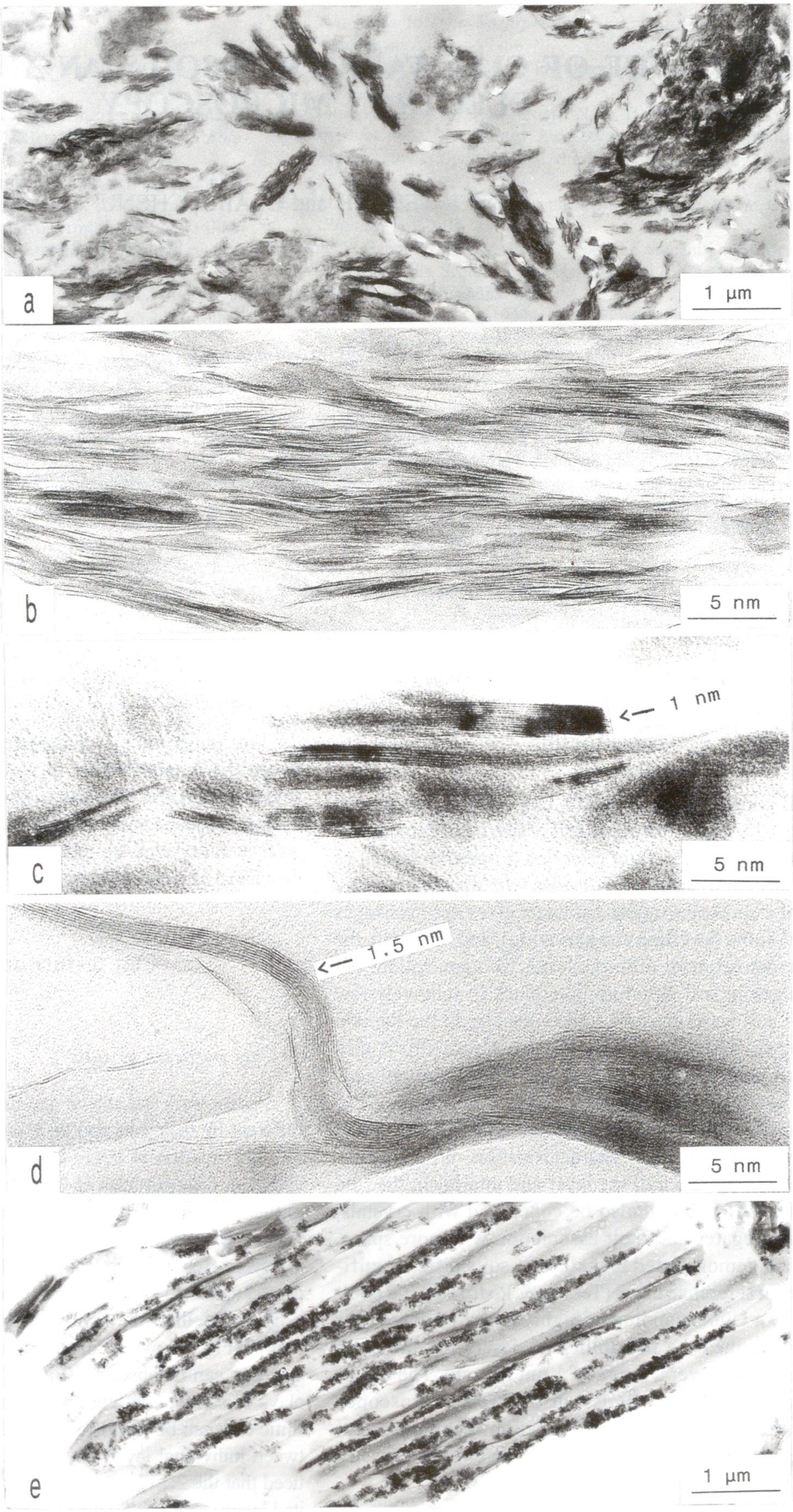

Fig. 1. TEM microphotographs of an interstratified clay at low (a) and high (b) magnification. Particles of an illite from Le Puy (France) (c), of Fourges clay (d), and clay from an oxisol from Amazonia in which kaolinite crystals are sandwiched with goethite (e).

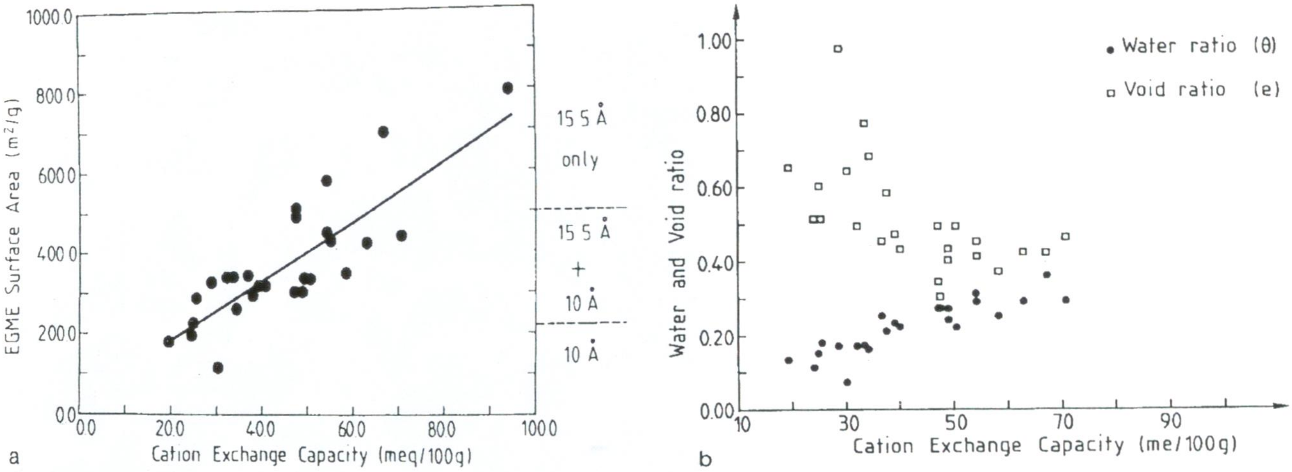

Fig. 2. Relationship between surface area as measured by EGME and CEC (a) and void ration (Vv/Vv) and water ratio (Vw/Vs) as function of CEC for soil clays of France at 48% relative humidity (*from* Tessier et al. 1992).

Similar results were obtained with kaolinitic clays (Tandy et al. 1990; Robain et al. 1990). For instance, in C horizons of Guyana oxisols giant (25 µm) kaolinite crystal aggregates (Fig. 1e) can be observed in contact with the geological parental material. In oxisols it was shown that crystal size decreases generally from the bottom to the top of the soil profile. In the latter, kaolinite crystals are generally very small and associated with extremely fine iron oxides diffused in the clay matrix. Very large crystal aggregates appear thus to be typical of oxisols and give their specific micropedic soil structure (Chauvel et al. 1991).

It is concluded that the clay fraction of soils is rarely made of individual crystals but rather associated in stable aggregates, when prepared in normal dispersion conditions. Only strong chemical products seem to be able to separate the individual crystals, i.e. the fundamental particles of Nadeau (1985), namely, the clay crystals.

2. Relation between mineralogy and surface area

Because clays in soils can be regarded as materials having more or less large crystals or crystal aggregates they differ also in their surface areas. Fig. 2a shows the correlation between surface area deduced from the adsorption of Ethylene Glycol MonoEthyl Ether (EGME) and the Cation Exchange Capacity (CEC) for different clays of clayey soils of France. X-ray diagrams and TEM have shown that in these clays the proportion of open 2:1 layers ranges from about 25 % for illitic clays, 40–50 % for interstratified clays, to about 75 % for smectitic clays (Robert et al. 1987).

This criterion can also be used to establish a relationship between clay texture and clay physical properties (Bruand et al. 1988).

Consequences on hydration and sweling

1. Swelling

It is established that the swelling mechanism of clay in soil is mainly associated to crystal and particle arrangement changes. Because most volume changes observed under field conditions occur at high matric potentials, swelling of soils is thus not directly related to layer distance changes, even for Ca- and Mg-smectites (Méring 1946; Aylmore & Quirk, 1971; Tessier 1984; Touret et al. 1990), but rather to changes in interparticle distances and pores (Fig. 3 a to c). The one exception is low electrolyte Na-smectitic clays.

When interpreted in physical terms, the swelling of clays occurs during hydration, as the initial volume of the clay (V_1) changes to a final volume (V_2), where $V_1<V_2$. This definition implies that both initial and final volume are known. By considering the minimal volume reached at the shrinkage limit and the volume at 1 kPa, the physical swelling ranges from 20% for coarse illites and kaolinites to about 150% for Ca-smectites while for Na-smectites prepared in dilute solutions the swelling reaches 500% (Tessier 1990).

2. Aptitude to shrinkage

The clay materials of soils may be mostly differentiated by their aptitude to shrinkage (Tessier 1990). The above presented criterion of CEC helps to establish a relationship between clay texture and clay physical properties. This is clearly shown by the variation of the water content and the bulk volume of clays as a function of CEC for 2:1 clays in equilibrium with a relative humidity of 48% (Fig. 2b). It was noted that coarse illitic clay materials produce a shrinkage limit at a suction pressure of ~ 1 MPa. It also corre-

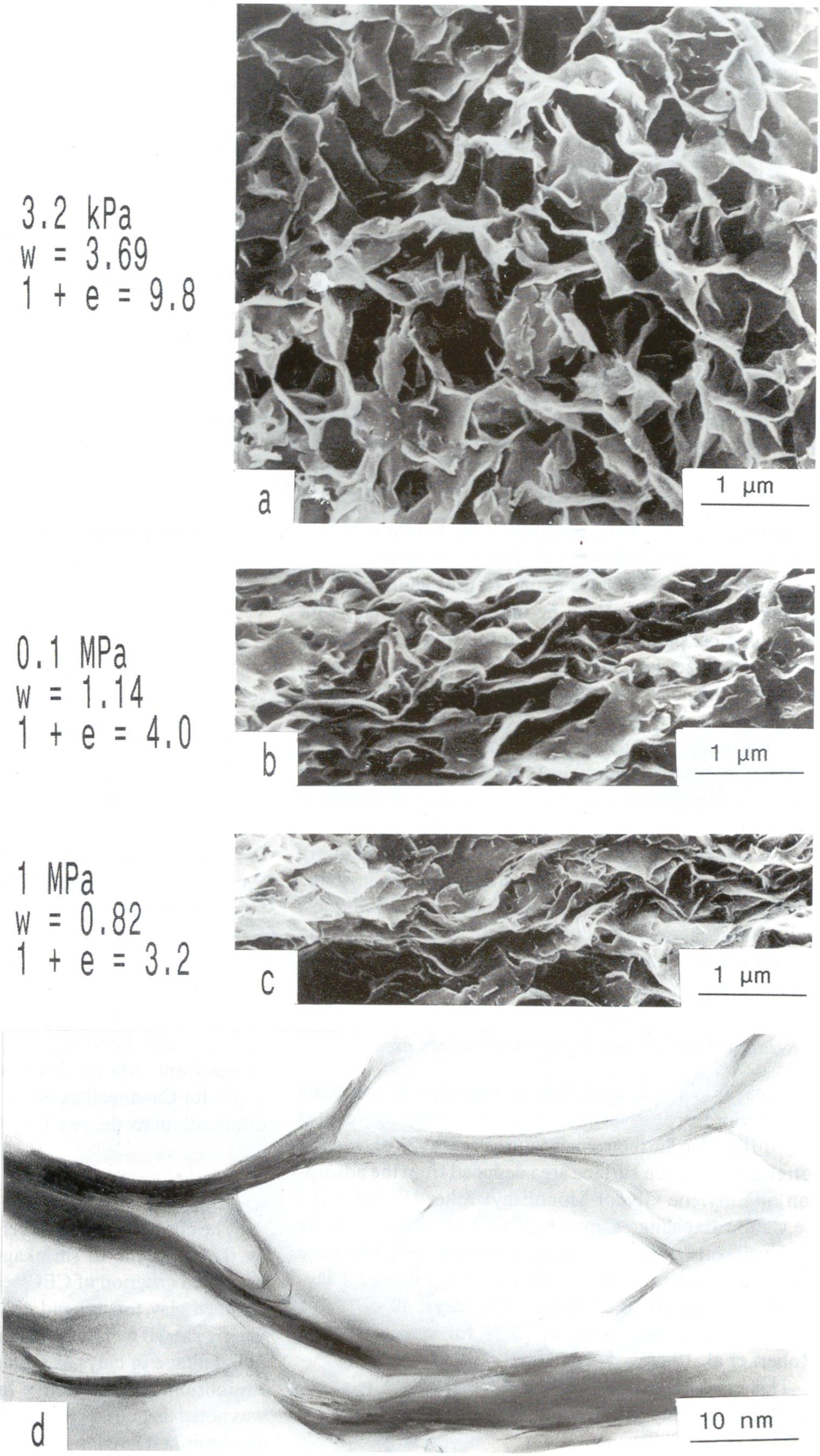

Fig. 3. Bulk volume and microstructure of Greek montmorillonite prepared with 1M NaCl solution at 0.0032 MPa (a), 0.1 MPa (b), and 1MPa (c), where w = water content and e = void ratio (Vv/Vs). Channels between pores at 0.1 MPa are illustrated in (d) (from Tessier 1984).

sponds to the appearance of the air-entry point, which indicates that the pores in the clay matrix are of the order of 150 nm. For a smectite, it is necessary to reach ~ 100 Pa before the air entry point appears to be significant (Tessier et al. 1992). In other words, when highly contrasted climatic conditions are met, the aptitude of clayey soils to be compacted, i.e. to reach high bulk densities, may be associated to the fabric and mineralogical nature of the clays (Wilding & Tessier 1988).

Consequences for hydraulic conductivity and structural stability

1. Microstructure and permeability

Hydraulic conductivity measurements give accurate informations about pore connectivity, and thus about the importance of the clay matrix on clay properties. Prost (1990) using the data of Hénin (1977) and Rouquerol (1968) was able to determine from hydraulic conductivity measurements the mean hydraulic radius, the tortuosity factor and the surface envelope of the crystal aggregates (Fig. 4 and Table 1). For each clay the extreme value of the hydraulic conductivity radius was obtained at swelling pressures of 10^{-3} and 1 MPa, respectively.

The very different value of hydraulic radius and its variation with swelling pressure may be interpreted in terms of microstructure.

- For Na-smectite prepared with *dilute* solution (NaCl $<\sim$ 0.3 M), the pore system assuring water flow is mainly an inter-layer pore, the size of which is the order of the distance between the layers when the diffuse double layer is fully expanded (up to 10 nm) (Fig. 4).
- Fig. 3 shows the effect of the swelling pressure (3.2 kPa, 100 kPa and 1 MPa) on both volume and microstructure of Na-montmorillonite prepared with *concentrate a* solution ($> \sim$ 0.3 M NaCl). The values of the hydraulic pore radius thus correspond, for suction pressures < 1 MPa, to pores between crystals or crystal aggregates. These pores are considerably larger than those between the layers, which are 2.0 nm wide for suction pressures lower than 5 MPa (Ben Rahïem et al. 1987).
- With clays having collapsed layer spacings (kaolinite, illites), the magnitude of the pore size is associated to the presence of inter-crystal and inter-crystal aggregate pores (order of 0.01 to 1 μm). SEM and TEM observations as well as hydraulic conductivity measurements suggest that the intra-aggregate water flow is relatively motionless and thus not really concerned with water flow through the system.

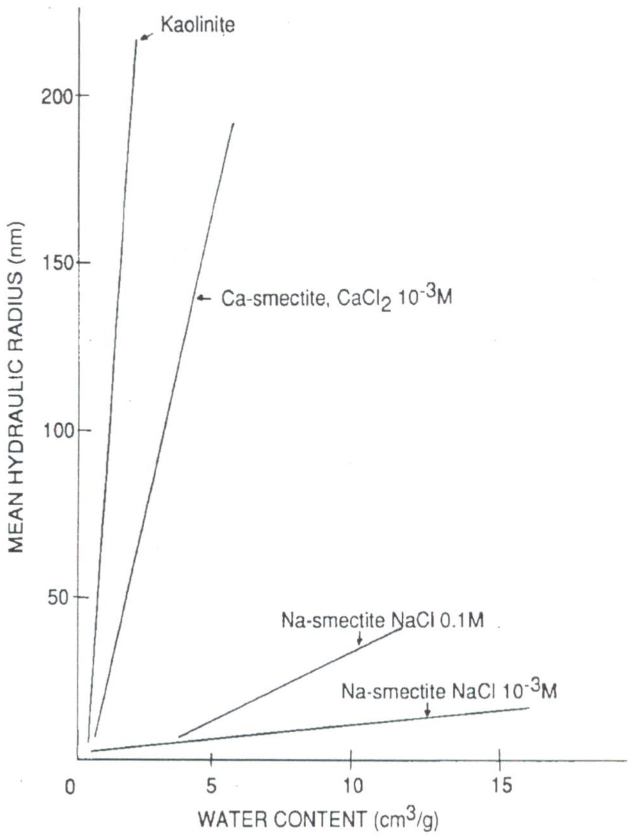

Fig. 4. Mean hydraulic pore radius of clays prepared at various water contents (adapted from data of Prost 1990).

Table 1: Mean hydraulic radius, tortuosity factor, surface envelop, layer number and type of particle for different clay minerals. (adapted from Tessier 1990 and Prost 1990).

Clay Sample	Hydraulic radius (nm)	Tortuosity factor	Surface envelop (m^2/g)	Layer number	Particle type	Layer spacing (nm)
Kaolinite	40–200	1	12	90	rigid	closed (7 nm)
Ca–Hectorite 10^{-3} M $CaCl_2$	3–200	1	8	100	very flexible aggregate	1.8 to 2.1
Ca–Hectorite 10^{-1} M $CaCl_2$	3–50	1	43	20	flexible aggregate	1.8
Na–montmorillonite 10^{-1}M NaCl	2–25	6	43	20	tactoid	3.5 to 5.0
Na–montmorillonite 10^{-3}M NaCl	2–10	6	815	1	tactoid	3.5 to 10

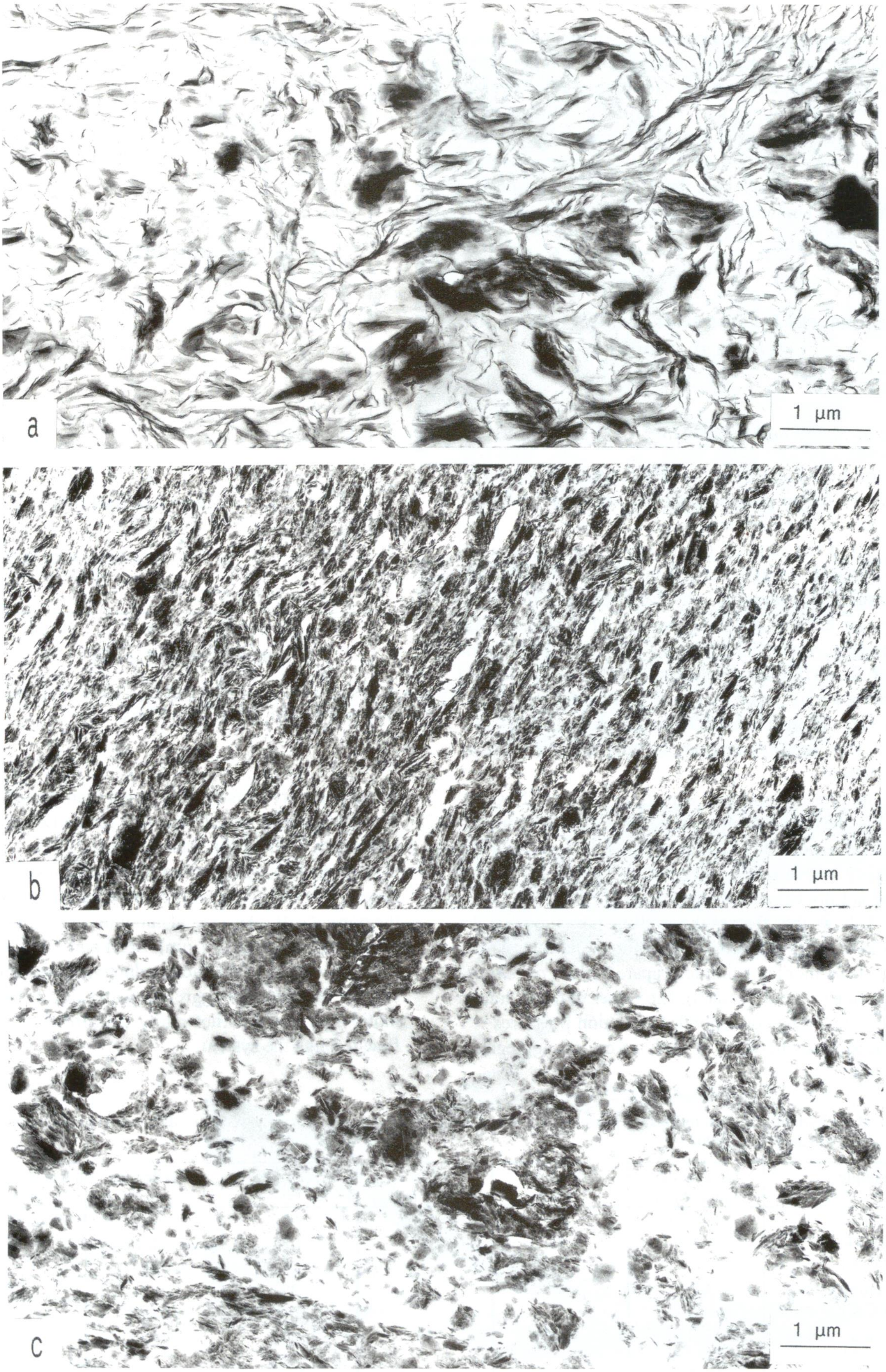

Fig. 5. Clay microstructures after rapid rewetting of Ca-montmorillonite from Wyoming (a) and of slow (b) or rapid (c) rewetting of illite from Le Puy.

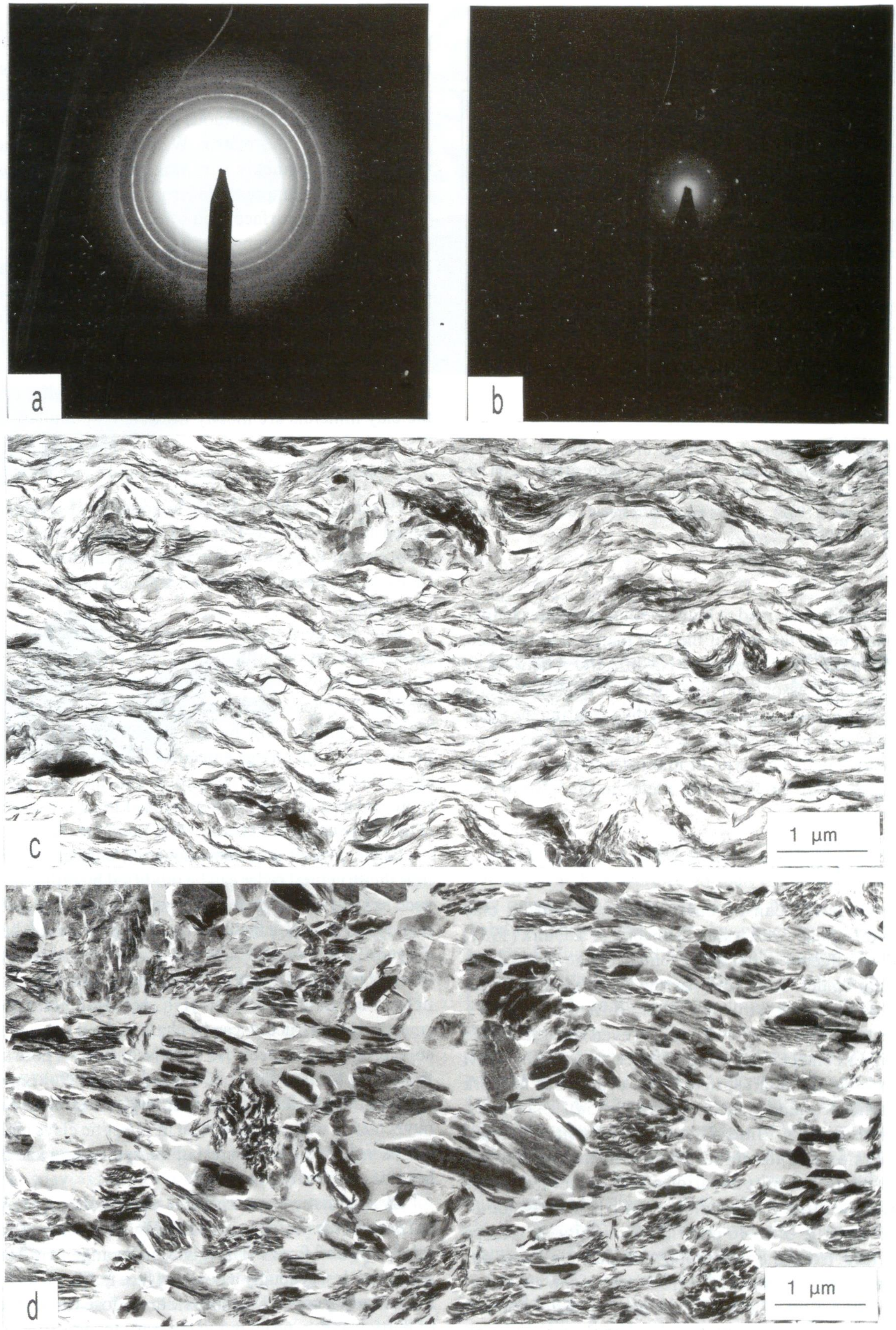

Fig. 6. Electron diffraction diagram showing a–b layer stacking with (a) desordered crystal and (b) ordered crystal, and corresponding microstructures at 1MPa for kaolinite (c) and smectite (d).

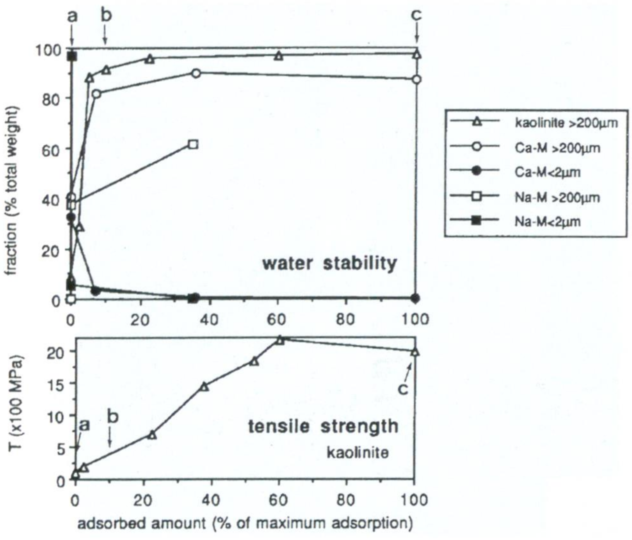

Fig. 7. Water stability and tensile strength of clay minerals as affected by an adsorbed polysaccharide. Both properties are measured on air-dried millimetric samples (adapted from Chenu & Guérif 1991 and Chenu & Jaunet, (1992). Arrows a,b,c correspond to the complexes observed in cryoSEM

TEM results obtained at high swelling pressures help to understand better the role of the clay matrix. For instance, at 0.1 MPa the TEM shows for Na-smectite a network of particles mainly associated face-to-face (Fig. 3d). The size of the transmission pores (channels) thus assuring the water flow compared to the pore size of the inter-crystal pores seems very small. This type of microstructure can explain the magnitude of the tortuosity factor in the case of Na-smectites.

2. Physical stability

This question can be envisaged by rewetting clay samples on contact with water. Pannabokke & Quirk (1957) showed the effect of initial water content on the breackdown of soil aggregates. Depending on the clay, the effect of re-wetting on clay microstructure is very different:

- Smectitic clays remain very stable, even when subjected to direct contact with water (Tessier et al. 1992). TEM shows that the microstructure is a continuous network of particles arranged face-to-face (Fig. 5a).
- On the contrary, illitic clays are very sensitive to rapid immersion and the initial water content and the rate of re-wetting results in various microstructures. After slow re-wetting the assemblage of particles is homogeneous (Fig. 5b). No other discontinuity than those resulting from the assemblage of particles was observed. Direct contact with free water led to a fissuration of the material, delimiting aggregates of about 5 µm. With illitic clay materials a single wetting was enough to bring about a breakdown of the microstructure (Fig. 5c).

Such a disruption of illites upon rapid wetting can be related to different factors: (1) in the dry material, 90 % of the pore volume is filled with air, (2) the particles being rigid the surface area of contact between adjacent particles is small, and (3) a continuous and large pore system takes place within the clay matrix. Water getting in contact with the sample can quickly very migrate into the porosity by capillarity and compress the air initially present in the material. Since the surface area of contact between clay particles is small, short range forces cannot overcome this pressure and re-wetting leads to a disruption of the illitic samples. Hence, the microstructure of illite explains its physical instability on the contrary to the behavior of a smectite. Kaolinitic materials as regard to their microstructure behave like illites. These results contribute to an understanding of the physical instability of temperate silty soils, in which the clay minerals are mostly illites and kaolinites.

3. Geometry of the clay matrix and pore continuity

Because clay particles are aggregates of crystals, associated mainly face-to-face, they can be characterized by their size in a–b and c directions. Furthermore, clay particles can be described by their stacking order in *a–b* and *c* directions.

Low charge smectites are usually regarded as having a turbostratic crystal structure, i.e. the rotation of one parallel layer relative to another around the *c* axis is completely random. In other words, the stacking order relative to the *a–b* plane is random (Fig. 6a). To the contrary, diffraction diagrams from high charge smectites, illitic or kaolinitic clays show distinctive spots indicating that the clay layers are more ordered as a result of increased inter-layer attraction (Fig. 6b) (Suquet & Pezerat 1987; Stucki & Tessier 1991).

In the case of smectites, however, cohesion forces and thus the structural order can be modified by different factors such as salts, exchangeable cations and also drying and wetting cycles. Because the layer is very thin they can be easily curved. Clay layer overlap and give extraordinarily large assemblages in a–b directions. As a consequence the particles are mainly connected (Fig. 6c). In other words, the smectite microstructure can be characterized as a continuous solid phase which gives rise to a discontinuous pore system. According to Ben Ohoud & Van Damme (1990) only a part of the surface area and the porosity is accessible to different molecules. It was concluded that in smectitic clays molecules can be insulated from the rest of the soil system and thereby become physically protected.

By contrast, in coarse illitic and kaolinitic clays, the stiffness of the particles is ensured by the very high layer to layer cohesion forces. This gives an arrangement based on rigid particles, generally associated face to face, which marks the limits of pores where the width is close to the thickness of particles (Fig. 6d). On the contrary to smectites, the porosity and surface area of the material appears to be totally accessible to various molecules.

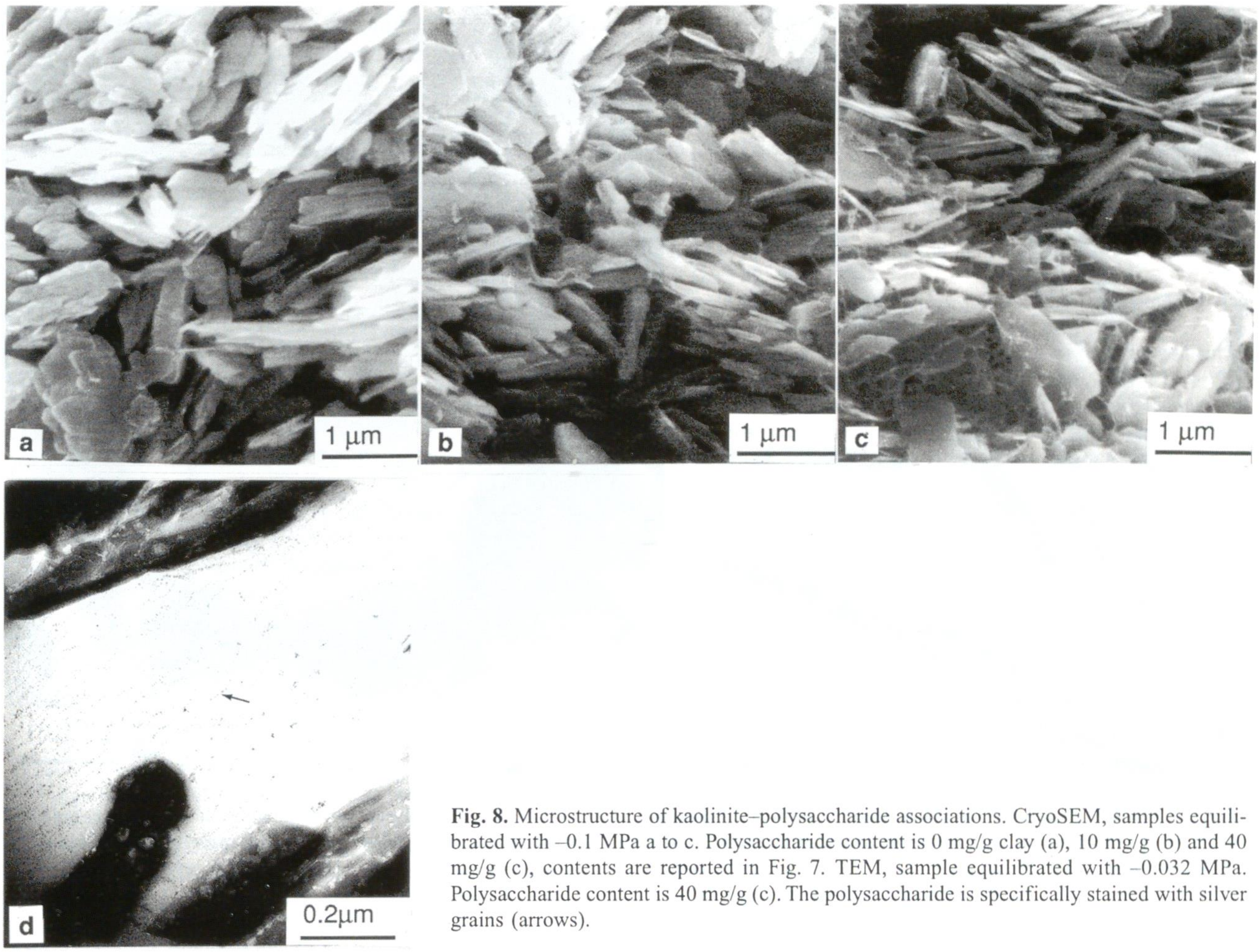

Fig. 8. Microstructure of kaolinite–polysaccharide associations. CryoSEM, samples equilibrated with –0.1 MPa a to c. Polysaccharide content is 0 mg/g clay (a), 10 mg/g (b) and 40 mg/g (c), contents are reported in Fig. 7. TEM, sample equilibrated with –0.032 MPa. Polysaccharide content is 40 mg/g (c). The polysaccharide is specifically stained with silver grains (arrows).

When illitic and kaolinitic clays reach higher specific surfaces (interstratified clays, 350 to 500 m^2/g) their capability for shrinkage increases. The more or less flexible character of the crystals and crystal aggregates, dependent on their thickness, appears to play a decisive role in the continuity of the solid phase and the pore system and thus the macroscopic properties.

Clay-associated substances, their effect on clay fabric and consequences on physical properties

Organic matter, oxides and hydroxides are soil constituents that may associate to clay minerals and modify the overall properties of soils. Organic compounds may be located, depending on their nature and size, in interlayer spaces of clay minerals or on the surfaces of clay particles (Theng 1979). They may not lead to changes in clay microstructure, or cause the association of clay particles or even result in strong microaggregation (Chenu 1989; Habib et al. 1990; Jambu et al. 1987).

Polysaccharides have a prominent role in soil structural stability which is ascribed to their direct interaction with clay minerals. As shown in Fig. 7, the gain in structural stability is considerable for clay minerals of low stability, such as kaolinites or illites, and only significant with Na- or Ca-smectites (Chenu et al. 1994). The tensile strength of kaolinite and montmorillonite–polysaccharide complexes increases with the amount of polysaccharide (Fig. 7), hence the resistance of the material to slaking and dispersion upon wetting is due to a stronger interparticle cohesion with polysaccharide (Chenu & Guérif 1991). Examination of the clay–polysaccharide complexes with cryoSEM and TEM (Fig. 8) reveals that polysaccharides have a fibrous shape and are attached to the clay particles and strongly interconnected (Chenu 1989). Interparticle cohesion and structural stability with polysaccharide may then be explained by a *bridging mechanism*. The progressive increase in interparticle cohesion with polysaccharide content traduces the increase in the numbers of bridges linking the particles (Fig. 8a–c). In addition such polymers have the ability to form intermolecular linkages, as clearly observed

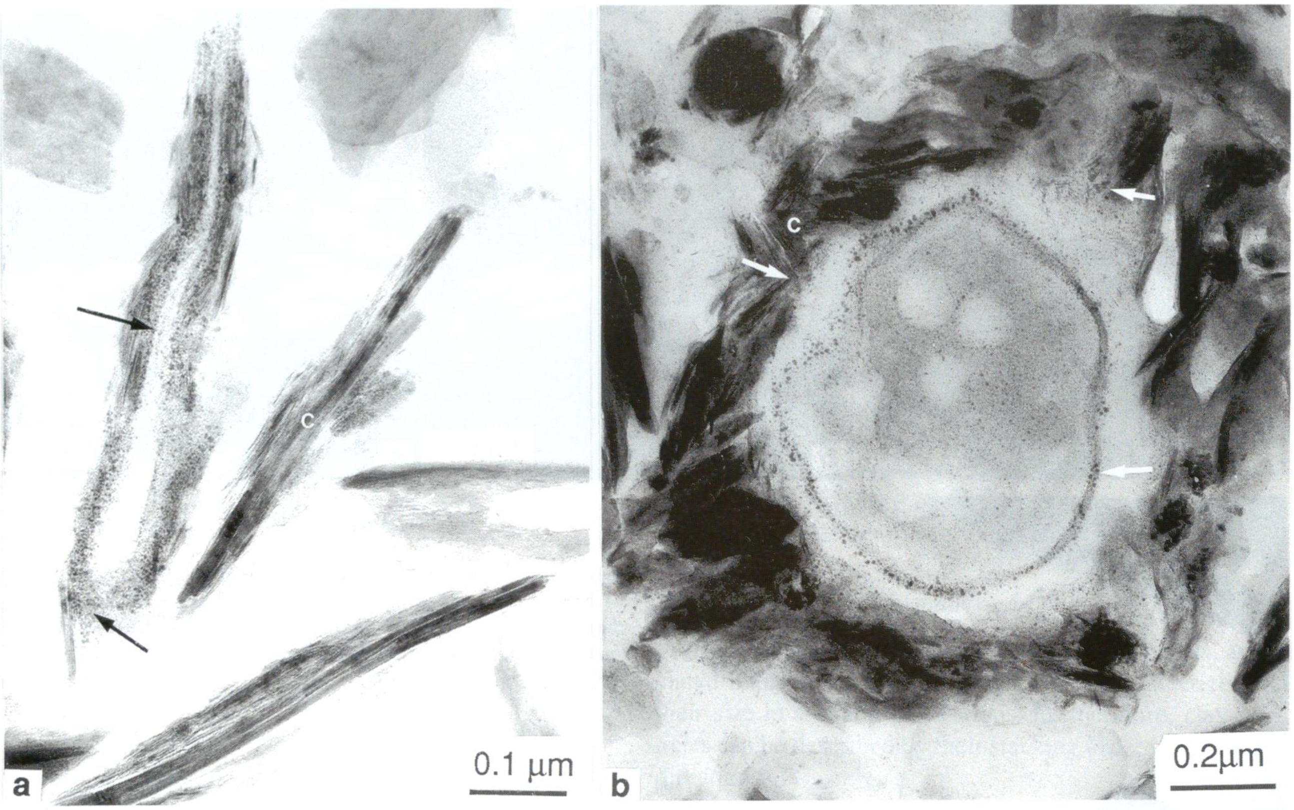

Fig. 9. Distribution of clay-associated substances in soil materials. TEM observation. (a) Polysaccharide coating on soil clay particle, (b) bacterial micro-aggregate with a clay–polysaccharide shell (silver grains that are used to stain polysaccharides are indicated with arrows), (c) iron coating.

by TEM on Fig. 8d, that enhance the strength of the bridging process.

In this example the location of organic matter and its own structure modify the microstructure of the clay into an organo-mineral network, in which the continuity of the solid phase is ensured by the organic matter. The effect of polysaccharides is considerable with kaolinite and illite, while in the case of smectites the organic bridges add little to the high degree of solid phase continuity that previously exists. Enhanced water retentions and decreased hydraulic conductivity of such materials are also in relation to the organo-mineral network microstructure (Chenu 1989; Chenu et al. 1994).

A great deal of attention has been paid in the literature to the organic interlayer complexes of clay minerals (Theng 1979). However, changes in physical properties often correspond, as shown by combined use of electron microscopy and X-ray diffraction, to changes in microstructure at the level of the arrangement of clay particles, i.e., to interparticle location of organic matter, rather than to clay-interlayer locations (Emerson et al. 1986; Chenu 1989).

When considering now soil fabrics, the distribution of organic matter appears very heterogeneous (Foster 1981). Organic constituents may occur as particles independent from the mineral phase, as clay coatings (Fig. 9a) or as cements in microaggregates, for example as networks between clay particles in bacterial micro-aggregates (Fig. 9b). The observed microaggregates resisted to high energy ultrasonic dispersion, they have thus a very high physical stability. Similar observations can be made with hydrous oxides (Fig. 1e and Robert et al. 1987). Different microstructures of clay–organic or clay–oxydes associations are likely to lead to different physical properties. Hence, a better knowledge of the location of organic matter and oxyhydroxides in soils, at the scale of clay constituents, seems required to understand their role on soils properties.

Conclusions

Soil physical properties of clayey soils are governed by soil fabric and in particular, by the arrangement of clay particles. In most soils shrink-swell occurs in response to changes in shape and pore volume of interparticle porosity upon changing hydration state.

Soil structural stability mainly depends on solid continuity. When short range forces concern a significant surface of the minerals (calcium smectites) or when constituents such as oxides and organic matter associated to clays ensure a certain continuity of the solid phase between the clay particles, mechanical properties as well as structural stability may be considerably improved.

The study of the microstructure of clay materials gives keys for the understanding of their properties in relation to mineralogy or presence of associated substances. At our state of knowledge, the electron microscopic examination of soils alone does not allow to predict their physical behavior. Thorough case studies with reference clayey materials are required to build up the links between the nature of soil constituents on one hand, and the macroscopic properties of soils on the other hand.

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