

Experimental micropedological investigations of iron oxide-clay complexes and their interpretation with respect to the soil fabrics of Paleosols

J. B. DALRYMPLE

Department of Soil Science, University of Reading, Great Britain

INTRODUCTION

Micropedological techniques now range from the use of a low power binocular microscope to electron probes. Certain physical, chemical and biological properties of soil materials can now be measured and described in detail. Additionally, many of these techniques are ideally suited to experimental field and laboratory studies of soil processes, but not surprisingly, few workers have tried to apply such a wide range of micropedological techniques to the recognition and interpretation of modern soils, let alone to paleosols. Most studies of paleosols rely on the identification of fabric types from thin sections under a polarising microscope. These are then interpreted on the basis of the presently held concepts of taxonomy and classification evolved from investigations of fabric types from modern soils.

'LEHM' FABRICS AND 'LEHM' PLASMA MATERIALS AS RELICT FABRIC COMPONENTS OF PALEOSOLS AND TROPICAL PALEO-ENVIRONMENTS

One of the basic concepts of Kubiëna's [10-12] pioneer micromorphological work is that the total character of the fabrics of the B horizons of many soil types (Great Soil Groups) are distinctive and diagnostic. Kubiëna also considers that 'lehm' fabrics and 'lehm' plasma materials¹ are formed today predominantly under warm moist tropical and subtropical conditions, and, less commonly, under those of coastal Mediterranean areas. This, he considers, contrasts with the temperate climatic conditions necessary for the formation of those 'erde' fabrics and 'erde' fabric components¹ associated with braunerde soil types. This led many

¹ 'Lehm' fabrics and 'lehm' plasma materials include the generic concepts of braunlehm, rotlehm and gelblehm of Kubiëna [12] as well as the various kinds of fliessplasma described by Kubiëna [14]. Although it is agreed that the designation lehm and erde should not be used without a prefix when referring to soil types, these terms well express the significant and diagnostic properties of the fabrics of the B horizons of such soils.

early workers in Western Europe, such as Kubiëna [12, 13], Dalrymple [3, 4], Klinge [9] and Proudfoot [16], to interpret 'lehm' fabrics and 'lehm' plasma materials in paleosol horizons as diagnostic of relict soil features. Also, it was assumed that such soil fabric features were originally formed under a tropical or subtropical climate and, therefore, must be of Tertiary or early Pleistocene origin.

More recently such paleo-climatic interpretations have been challenged. On both theoretical and practical grounds it is steadily becoming more difficult to accept the basic tenet of applying zonality to soil fabrics. This is especially the case when such zonal concepts are assumed without paleo-catenary investigations of even the major paleosol horizons. One detailed example is illustrated by the differing interpretations of the many and varied 'lehm' plasma materials that have been identified from the B horizons of paleosols occurring in well exposed and carefully recorded stratigraphic sequences of loess deposits in Western Europe and South East England. Dalrymple [4-6], unlike Kubiëna [14], considers that there is little evidence to support either of the two alternate hypotheses that these 'lehm' plasma fabric features represent relict soil features or that they are inherited from their underlying and unweathered loess parent materials.

Concentrations of 'lehm' plasma materials do occur within the s-matrix of such paleosol B horizons. Certain kinds show little obvious relationship to the position of cavities and conducting channels, and their distribution pattern within the s-matrix appears to be random. These 'lehm' concentrates, however, must be pedogenetic in origin as no 'lehm' concentrates nor any other kind of 'lehm' plasma materials have been identified from thin sections of completely fresh loess at such sites as Ford, Hale or Pegwell Bay in Kent; Bobbit's Hole near Ipswich in Essex; "Grande Briqueterie de Chedeville" and "Briqueterie Bigot" at Saint Pierre-les-Elbeuf in northern France; Achenheim in Alsace; and Niedermendig in Germany. The words "unweathered parent material" cannot be overstressed as 'lehm' concentrates do occur in both partially oxidised and partially decalcified loess materials.

If 'lehm' plasma materials are demonstrably not inherited from fresh loess, Kubiëna's alternative hypothesis of their relict pedogenetic origin must be considered. In these loess sites the 'lehm' concentrates do tend to occur more frequently in what are stratigraphically lower and, so, older paleosol B horizons. This of itself, however, does not necessarily mean a relict origin. It may be that further weathering and soil formation is involved after the burial of the paleosol by a subsequent aeolian deposition of loess. Even more significant is the fact that 'lehm' concentrates are normally present in the s-matrix of the B horizons of the present day soils at these loess sites.

There is, however, much more detailed evidence for the non-relict

nature of the 'lehm' fabric components in these paleosol B horizons. This relates to the 'lehm' plasma materials other than the concentrates. In total amount these 'lehm' materials far outweigh that of the 'lehm' concentrates, and, unlike the concentrates, they show a markedly preferred orientation under doubly polarised light. That is strongly anisotropic according to Cagauan and Uehara's [8] use of the term. In addition, much of the 'lehm' material is specifically aligned along conducting channels and around cavity walls. It is this spatial arrangement of layered colloids that strongly suggests their formation by lessivage and the majority of these 'lehm' plasma components are considered to represent iron oxide-clay complexes redeposited from the soil water. Again, similar micropedological features occur in the modern lessivé or grey-brown podzolic soils at many of these loess sites in South East England and Western Europe. Thus, a relict origin for the 'lehm' plasma materials in the fabrics of the paleosol B horizons at these loess sites is no longer tenable. Neither is there sufficient evidence to postulate that the paleoclimate at the time of formation of these paleosols was significantly different from that of today. Further support for this interpretation is that the gross micromorphological properties of the B_t horizons of these lessivé paleosols differ little from those of similar modern (Alfisol) soil horizons described by many workers from many parts of the world.

VARIATIONS IN THE OPTICAL AND MORPHOLOGICAL PROPERTIES OF 'LEHM' PLASMA MATERIALS AND THE ORIENTATION AND ALIGNMENT OF CLAY PARTICLE GROUPS

Dalrymple [7] has noted that there are important differences in detail in the optical and morphological properties of the 'lehm' plasma materials in the B_t horizons of lessivé soils. In the case of such modern and paleosol B_t horizons derived directly from loess parent materials, he considers that the pattern of distribution of the 'lehm' components directly depends on the characteristic porosity inherited from the loess. Since it is the size, shape and distinctive pattern of distribution of the pores that determine the specific paths for the movement of colloidal suspensions of iron oxide-clay complexes in soils, the translocation of the colloids is through rather than around the prismatic and blocky structural units of their B_t horizons. This gives rise to a marked contrast between the occurrence of a few and discontinuous cutans to these peds and the overwhelming redeposition of the iron oxide-clay complexes ('lehm' plasma material) within interpedal pores.

Most other differences in the optical and morphological properties of these 'lehm' plasma materials are thought to reflect variations in parameters other than porosity. For example, variations in the morphology of the iron oxide-clay concentrates from diffuse nodules with an irregular

shape to papules with a lamellar fabric or differences in the layering and banding of the plasma material in interpedal conducting channels are thought to depend more on variations in the length of time available for lessivage during the formation of each paleosol than on changes in any other single parameter. This may also partially explain the surprisingly uniform changes in colour and birefringence that occur in certain of these 'lehm' plasma materials [6, 7]. There is certainly evidence for thinking that these fabric features continue to form and develop after burial and that changes in the iron oxide to clay ratio are involved. Thus, these changes in colour and birefringence may relate more to the total length of time available that has elapsed since their burial rather than to the length of time available for formation before burial.

It is, however, the degree of anisotropy shown by different areas of iron oxide-clay complexes in the fabrics of these paleosol horizons that show the most striking and repetitive variations in both optical and morphological properties. Whatever the possible mechanisms involved in their genesis, detailed observations and measurement of the optical orientation and morphological alignment of these materials strongly suggest that there is a fundamental dichotomy. On the one hand, the s-matrix may contain groups of clay and iron oxide-clay complexes that are typically oblong or square in shape and that are relatively small in size with their long axis rarely measuring more than 10 μ . Each such group of clay particles shows an optical continuity and parallel orientation (strongly anisotropic) within the group and this means that the clay particles must have a face to face orientation within each group. However, the groups themselves have a predominantly edge to face orientation one to another and they show no specific alignment with respect to other components of the s-matrix such as mineral grains, conducting channels, cavities or concentrates and concretions. It is these morphological and optical properties of the groups of clay particles and iron oxide-clay complexes that diagnostically characterise the 'erde' fabrics and 'erde-like' components of the s-matrix in these paleosol B and B_t horizons.

In contrast, in those areas of s-matrix interpreted as 'lehm' fabrics or 'lehm' plasma materials the groups of clay and iron oxide-clay complexes have both different morphological properties and a different optical orientation between the groups. Only the optical properties within individual groups are similar. For example, each optically continuous individual group can be of almost any shape, and in these paleosol horizons characteristically they are of the order of 100 μ across their longest axis. More significant, however, is the fact that these groups do not have a predominantly edge to face orientation one to another but are themselves orientated face to face and even, on occasions, edge to edge as well. These groups of clay particles and iron oxide-clay complexes, therefore, show an alignment within the s-matrix of their fabrics and it is this

property that diagnostically identifies 'lehm' plasma material. This property also differentiates the overall appearance of 'lehm' plasma material in one s-matrix from that in another. The degree of such organisation tends to be only moderately well developed in the paleosol B and B_t horizons characterised by either a high sand or a high silt content. In these s-matrices the alignment of the groups is largely restricted to a face to face orientation around the mineral grains and between one grain and the next. That is unless lessivage is operative when additional well developed alignment of such groups can be observed in the conducting channels and cavities. Here, the morphological alignment of the groups is parallel to the walls of the conducting channels and cavities, whilst the optical orientation of the groups is essentially face to face as only rarely have optical discontinuities been observed in this aligned material. The greatest degree of organisation of groups of clay particles and iron oxide-clay complexes has been observed in those paleosol B and B_t horizons containing more than 35% of clay-sized material. Here, each individual group tends to be very large and frequently the groups show a curved or pseudo-hexagonal alignment. Thus, it is the large size of many of the individual groups and especially the optical orientation and morphological alignment of the groups of clay particles and iron oxide-clay complexes that both identifies and differentiates 'lehm' fabrics and 'lehm' plasma materials. Such identification of 'lehm' plasma materials does not depend on the mechanism of their formation be this the result of lessivage, the *in situ* differential expansion and contraction of the clay-sized fraction, or a combination of such processes as oxidation, hydration and hydrolysis. Such mechanisms merely differentiate between the overall distribution pattern, size and amount of such material within the s-matrix, and particularly the nature of the morphological alignment of the 'lehm' materials.

'LEHM' PLASMA MATERIALS AND THE OPTICAL PROPERTIES OF LABORATORY PREPARED IRON OXIDE-CLAY MIXTURES

In order to substantiate these observations and interpretations of fabric components from paleosol B, and especially B_t horizons, an attempt has been made to compare them with thin sections of laboratory prepared iron oxide-clay mixtures. Previous workers have prepared 'soil fabrics' in the laboratory by adding clay to quartz sand before sectioning the resultant 'soil'. Methods used to add the clay to the sand include mixing [15], and by percolation and capillarity [2]. In each case birefringent fabric components with varying degrees of orientation were observed. Neither workers, however, used laboratory prepared iron oxide-clay mixtures with varying, but known, proportions of iron oxide to clay type.

In this work, such colloidal suspensions were prepared by precipitating known amounts of haematite and lepidocrocite onto the previously cleaned surfaces of clay minerals of differing size fractions below 2μ [17]. All the colloidal suspensions used in this study were prepared by Rowell, and possibly the most interesting were a series containing varying amounts from 0.5% to 50% by weight of haematite precipitated onto 0.1μ montmorillonite that itself had been separated from a sample of Wyoming bentonite². These dispersed colloidal suspensions were allowed to dry out as thin films on glass slides. In order that the optical properties of these thin films of iron oxide-clay mixtures could be directly compared with thin sections of soils, an attempt was made to keep their thickness within the range normal for soil studies. Of general help here is the nature of the birefringence of the silt-sized dust particles which invariably become embedded in the films unless they are allowed to dry out in a desiccator or similar dust free environment.

After drying from a dispersed state, such iron oxide-clay mixtures show a range of colours³ under ordinary light that vary in intensity with added amounts of precipitated iron oxide. That is from very pale brown (10 YR 8/3) for 1% additional iron oxide, through very pale brown (10 YR 8/4) for 5%, pale yellow (5 YR 8/4) for 10%, to reddish yellow (7.5 YR 7/8) for 50%. More significant are the optical and morphological properties seen under doubly polarised light. Here, the iron oxide-clay mixtures are strongly anisotropic. The face to face orientation of the large groups of clay particles is well marked and certain areas show additional organization into pseudo-hexagonal or a series of right-angled alignments. All the iron oxide-clay mixtures are strongly birefringent with yellow (5 Y 8/4) and pale yellow (5 Y 8/6) colours except for the laboratory prepared sample with 50% added iron oxide when the body colour of the iron oxide masks that of the highly birefringent clay to give a reddish yellow (5 YR 7/8) colour under doubly polarised light. These features together with a low relief all help to produce optical and morphological properties resembling those of 'lehm' plasma materials. In fact these properties are so markedly similar as to warrant not only further investigation but also to suspect an underlying similar mechanism for their genesis.

² The geological sample of Wyoming bentonite was supplied by F. W. Berke and Co. Ltd.

³ All the Munsell colour readings are of relative significance only. A daylight filter was used, but, by varying the intensity of the illumination or the size of the opening of the diaphragm, it is possible to obtain a difference of colour of either 1 hue, or 1 value or 2 chromas.

'LEHM' PLASMA MATERIALS AND DRYING MODELS OF IRON OXIDE-CLAY MIXTURES OF VARYING CONCENTRATIONS

In contrast with the experimentally produced 'lehm' materials it has been found very difficult to produce experimentally 'erde-like' fabric features from these same laboratory prepared iron oxide-clay mixtures. The first attempt was to flocculate the dispersed colloidal suspensions by the addition of 10^{-2} m calcium chloride. The flocs so produced tend to be very small and, if mounted directly onto a glass slide, they are not highly concentrated in the supernatant liquid. This means that on drying the flocs spread out in a thin film with small groups of iron oxide-clay particles orientating themselves parallel to the slide. Optically this results in well developed anisotropic features and other properties diagnostic of 'lehm' materials. Thus, under these experimental conditions the optical properties of an iron oxide-clay mixture dried from a previously wet flocculated state differ little from those of the same iron oxide-clay mixture dried from a previously wet dispersed state.

These specific iron oxide-clay colloidal suspensions, however, were thought not to be sufficiently concentrated by comparison with those likely to be present in the B and B_t horizons of paleosols and modern soils. This applies to the iron oxide-clay complexes formed *in situ* within structural units by a combination of such processes as oxidation, hydration and hydrolysis as well as to those likely to be translocated in the soil water and redeposited as cutans around structure faces and within interpedal pores. Thus, a concentrated clay suspension, a clay gel and a moist clay were obtained from both the previously wet dispersed and the previously wet flocculated iron oxide-clay mixtures. This was done by centrifuging and filtering on a porous plate with greater amounts of the solution being successively sucked out of the suspension. After allowing the concentrated clay suspension to dry on glass slides, the clay gels and the moist clays all showed a marked face to face orientation together with other typical optical properties of 'lehm' materials. This is irrespective of their previous wet state or concentration in suspension. Theoretically, however, this is what might be expected by the movement of a liquid/air interface onto a flocculated or dispersed iron oxide-clay mixture.

Such a drying model of a liquid/air interface is also what might well be expected to operate in the fissures and clefts between structural units and in conducting channels within such structural units in soil B horizons. Certainly the identification both in the field and in thin sections of the results of the translocation and redeposition of iron oxide-clay complexes would support such a model. In particular, this may in part account for the close similarity noted by Dalrymple [7] between the nature and pattern of distribution of interpedal pores and that of redeposited iron oxide-clay complexes ('lehm' plasma materials) in many modern and paleosol B_t horizons formed from loess and similar materials.

In contrast, it would seem unlikely that a liquid/air interface could be postulated as the drying model for the small groups of iron oxide-clay complexes occurring in those areas of the s-matrices of B horizons that are not adjacent to interpedal conducting channels or to the larger interpedal cavities. In this case the mechanism of drying would be by taking liquid out of the wet clay or clay gel by overall shrinkage rather than from a liquid/air interface. In order to test this hypothesis it was necessary to produce thin sections of the range of experimentally produced iron oxide-clay mixtures by some means other than by drying on a slide. Of the various methods tried the simplest was to allow a blob to dry in a dust free environment on the end of a thin piece of thread. This proved successful for the concentrated clay suspensions, the clay gels and the moist clays. Only in the case of the originally produced colloidal suspensions with a low concentration was it necessary to allow a blob of the colloid to dry inside a small mould. Each blob was then sectioned in a number of different directions both using standard techniques and by cutting thin slices with a scalpel. Both the thin slithers and the thin sections of each blob showed similar optical and morphological properties. For example, each thin section of the dried blob of the laboratory prepared 2.5% haematite precipitated onto $0.1\ \mu$ montmorillonite has only a moderately well developed birefringence. Typically it shows reddish yellow (5 YR 6/8; 7.5 YR 6/8 and 7/8) or even red (2.5 YR 5/8) polarisation colours, but there are always small diffuse areas with a more pronounced birefringence (yellow (10 YR 8/6, 8/8 and 7/8) colours) that are apparently randomly interspersed throughout their fabrics. This means that there are in these dried blobs small groups of clay particles present with a face to face orientation within each optically continuous group, but the significant and dominant pattern is of small groups with a predominantly edge to face orientation of one small group to another. These optical properties are present in the sections of all the blobs, that is irrespective of the degree of concentration of the clay particles in suspension and irrespective of whether they were in a dispersed or flocculated state before drying. Thus, even without the presence of sand and silt-sized material, such experimentally produced iron oxide-clay mixtures have the relatively random optical orientation and the lack of a marked morphological alignment so typical of clay-sized material in 'erde' areas of soil B horizon fabrics.

CONCLUSIONS

These experimental results show that in terms of colloidal chemistry it is not correct to describe 'lehm' fabrics and 'lehm' plasma materials as dispersed iron oxide-clay complexes. Nor is it correct to describe 'erde' fabric components as being flocculated iron oxide-clay complexes. These

terms belong solely to colloidal suspensions. The nature and character of the clay-sized material seen in thin sections of paleosol and modern soil B horizons must be described and interpreted in terms of the optical and morphological properties of dried colloids. Further, it has been demonstrated that many of the differences observed in the optical properties of the experimentally produced iron oxide-clay mixtures depend primarily on the specific drying mechanism of these colloids. This is particularly the case with respect to the optical orientation of their groups of clay particles. On the one hand there are groups of clay particles with a face to face orientation both within and between the individual groups that result from the drying down of a liquid/air interface onto the iron oxide-clay mixture. On the other there are groups of clay particles with a face to face orientation within groups but with an edge to face orientation between groups. This kind of optical orientation results from a drying model of overall shrinkage of the iron oxide-clay mixture. This holds true irrespective of the wet state of the colloidal suspension or of their concentrations right through to clay gels and moist clays.

Such drying models can be related to the iron oxide-clay complexes in the soil fabrics of B and B_t horizons of both paleosols and modern soils. This is justified on the grounds that the optical and morphological properties of both the laboratory made samples of iron oxide-clay mixtures and the iron oxide-clay complexes are so markedly similar. Thus, it would seem certain that repetitive hydration and dehydration must be an extremely important mechanism in helping to produce the nature of the organisation of the iron oxide-clay complexes in their s-matrices. There are two contrasting positions where iron oxide-clay complexes commonly occur and dehydrate. Liquid/air interfaces are to be expected in fissures and clefts between structure faces and within interpedal conducting channels and cavities, whilst overall shrinkage is to be expected elsewhere within structure units away from such potential lines of translocation and redeposition of iron oxide-clay complexes. It follows that the clay-sized fraction in soil fabrics should be of two main types with the formation and occurrence of 'lehm' plasma material in those areas where dehydration operates as a liquid/air interface, and 'erde-like' features in those areas likely to overall shrinkage. Such a dichotomy in optical and morphological properties has been observed in soil fabrics from a wide range of B horizons from many parts of the world.

Also, there is evidence for thinking that specific variations in the nature of the distribution pattern and alignment of the groups of iron oxide-clay complexes in 'lehm' plasma materials relate to the specific mode of their formation or of their translocation and redeposition in the s-matrix prior to the dehydration of a liquid/air interface onto the iron oxide-clay material. Much further work is required but this could well account for the apparent causal relationship between the nature of the

morphological alignment of 'lehm' plasma materials and the size, shape and distinctive pattern of the interpedal pores in certain B_t horizons of paleosols formed from loess in South East England and Western Europe. Here, the genesis of these specific 'lehm' plasma materials is lessivage.

Hypothetically it is thought possible to match up most of the differing kinds of 'lehm' plasma materials found in paleosol B horizons by varying the nature and amounts of iron oxide precipitated onto cleaned clay minerals. The laboratory manufacture of such soil fabric features is at present under investigation. The closest resemblance in optics and morphology so far obtained is between such properties described by Kubiëna [12] for relict braunlehms and those of colloidal suspensions, clay gels and moist clays of cleaned montmorillonite that contain not more than 10% precipitated haematite or lepidocrocite and that have been allowed to dry as thin smears on glass slides. However, whatever further correlations there may be between the occurrence of reddish coloured iron oxide-clay complexes with a marked micro-morphological alignment and strong anisotropic characteristics, there is certainly no evidence to suppose a causal relationship between the genesis of large groups of such clay particles with a face to face optical orientation both within each group and between each group and processes of soil formation specifically restricted to tropical climatic conditions. Nor is it necessary to suppose that the occurrence of such 'lehm' fabrics and 'lehm' plasma materials requires the invoking of a relict origin in the Tertiary or early Pleistocene. What can be inferred is the likely drying mechanism of the iron oxide-clay complexes and, in many cases, something about the kinds and interaction of the soil forming processes operating at the time of formation of the paleosol.

Acknowledgements. I wish to thank Dr. D. L. Rowell for providing the laboratory prepared iron oxide-clay mixtures and for numerous discussions, and Mr. J. Dillon for technical assistance.

SUMMARY

Soil fabric components from paleosol B horizons are compared with thin sections of laboratory prepared iron oxide-clay mixtures composed of varying but known proportions of iron oxide to clay type. Optical and morphological observations show that there is a dichotomy in the nature of the orientation and alignment of the iron oxide-clay material in both cases. In particular, face to face orientation between individual groups of clay and iron oxide-clay particles combined with well developed alignment of the groups ('lehm' plasma material) contrasts with predominantly edge to face orientation between individual groups combined with a marked absence of any morphological alignment of the groups ('erde' fabric fea-

tures). It is shown experimentally that these optical and morphological features could result from a difference in the drying mechanism of the iron oxide-clay complexes. Thus, 'lehm' features would appear to result from the drying down of a liquid/air interface onto the iron oxide-clay material, and 'erde' features from the overall shrinkage of the iron oxide-clay material.

These experimental results are related to pedogenesis and to paleopedology, and they further substantiate previous field and laboratory interpretations from paleosol B horizons in Western Europe and New Zealand. That is that there is no longer any justification for inferring a causal relationship between the occurrence of 'lehm' plasma material in the fabrics of such paleosols and their pedogenetic origin under tropical climatic conditions, or their supposed relict origin in the Tertiary or early Pleistocene. What can be inferred is the likely drying mechanism of the iron oxide-clay complexes and something about the kinds of soil forming processes operating at the time of formation of individual paleosol B horizons.

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