

APPLICATION OF ACETATE PEELS AND MICROCHEMICAL STAINING IN SOIL MICROMORPHOLOGY

by

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INTRODUCTION

The surface of a highly polished specimen retains a micro-relief which reflects the detailed fabric of the material, and which is readily transferred and recorded in the form of a peel. Such peels are widely used in geology in the study of detailed microstructure, particularly in palaeontology and sedimentary petrology, as a simple, quick and inexpensive alternative to thin-sections. This technique is readily adaptable to pedological studies (Jenkins, 1970) but despite its obvious advantages it does not yet appear to have been extensively used in soil micromorphology. In this paper the procedures, potential and limitations of this technique are discussed briefly, as are also such developments as the use of serial peels to study three dimensional features, and the combination of microchemical staining with peel preparation to follow the distribution of selected components within soil fabric.

PREPARATION AND USE OF ACETATE PEELS

Peels were first prepared by the evaporation of a solution of collodion or nitrocellulose applied to the specimen surface (e.g. Walton, 1928), but they are now more conveniently prepared by moulding a thin acetate film to the specimen surface by use of a solvent (Stewart & Taylor, 1965). The procedure for the preparation of peels is the same in its initial stages as that for thin-

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sections. A block of resin-impregnated soil is cut, ground flat, and polished. Progressively finer grades of powder are employed (e.g. 50, 10, 6, 3 & $1\mu\text{m}$), preferably finishing with automatic polishing with diamond pastes of $1\mu\text{m}$ grade, or finer ($\frac{1}{2}$ & $1/4\mu\text{m}$). At this stage, the surface may be lightly etched by exposure to a dilute acid. (e.g. HCl, HF) to selectively accentuate relief, but this is not usually necessary or desirable. A peel may also be prepared from a similarly polished surface of an uncovered thin-section, though this can sometimes damage the latter.

The polished surface is positioned level and facing upwards, and flooded with the solvent acetone. A piece of acetate film is applied to one edge and smoothed across the surface, pushing a meniscus of acetone before it, and then the sheet rubbed gently to expel any excess acetone and entrapped air. The quantity of acetone is critical as an excess results in wrinkling whilst too little results in an incomplete moulding of the sheet to the surface. The thickness of the sheet can vary from around 75 to $150\mu\text{m}$ the thinner material having better optical properties, but the thicker proving more convenient to handle. The peel generally dries and may be detached within 10-15 minutes, but tends to curl and it is therefore conveniently mounted between 50mm square photographic glass plates. If left to detach spontaneously (c. 60 minutes), the curling of the peel is more pronounced and less easily corrected.

The peels can be studied directly by transmitted light microscopy. Fine detail can be observed down to a level determined by the final grade of polishing which is usually more than adequate to reveal the detail of coarse clay size material in the s-matrix. Dark field, phase contrast, and other modes of viewing can also be used, but are not usually necessary. Peels obviously lack the optical refinements of colour, anisotropy, bi-

refringence, etc., shown by minerals in thin-sections, and this will generally preclude mineral identification, unless by distinctive morphology. Peels are however, useful for the study of the general fabric of soils and, in particular, of the size, shape, orientation, and distribution of such features as voids and skeleton grains. For this purpose the absence of distracting or obscuring pigmentation or opacity can be a positive advantage. Fig. 1 illustrates the preservation of detail in the peel as compared to the original thin section. Peels can also be projected as if they were photographic negatives and so provide a convenient magnified (e.g. $\times 25$) record on high contrast paper (e.g. Kodagraph P84). For this purpose a LEITZ PRADOVIT slide projector fitted with a COLORPLAN 90 mm flat field lens has proved particularly useful. Such routine prints are also suitable for void of grain analysis using, for example, the ZEISS TGZ3 Particle Size Analyser.

SERIAL PEELS

Serial sectioning is a well established technique that is employed to elucidate the 3-D structure of fossils. It can be readily adapted by use of acetate peels to the study of pedological features such as void systems. Peels are particularly useful in this context since they are much simpler and quicker to prepare than thin-sections, and can also be made at smaller intervals. For the study of soil voids, intervals of 100 μm have been found convenient, but this can be reduced to 50 μm should the scale of investigation demand it. The procedure for the preparation of the peels follows that already described with minor modifications. To facilitate the alignment of successive peels, three parallel steel rods (c. 1 mm ϕ) are inserted prior to resin impregnation around the periphery of the soil block, perpendicular to the proposed

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plane of sectioning. Predetermined thicknesses are ground off the impregnated samples, using a LOGITECH precision grinding machine (PCM ; 2) the surfaces polished and successive peels prepared.

The reconstitution of the 3-D data recorded in the serial peels presents difficulties. Ager (1965) projected serial outlines of fossils onto a skew grid to obtain in effect an oblique view, but was doubtful of its value. Images of the peels can also be projected onto transparent (perspex) sheets, the outlines of the voids traced, cut out, or painted in, and then the sheets superimposed to provide a magnified scale model. The result can still be obscure by virtue of its complexity, but by painting with successive shades of grey (basal) through to black (top), the clarity of the model can be improved. This approach is illustrated in Figure 2 which depicts a void system in the surface horizon (Ap) of a Brown Earth profile (Flint Series). The possibility of processing the data recorded in the serial peels such that they can be reconstructed by computer to give a perspective 3-D image is also being investigated. An alternative approach to the study of void systems has been described by Pittman & Duschatko (1970) for sediments; this involves the dissolution of the silicate grains from a resin-impregnated sediment with HF, leaving a resin cast to be viewed by scanning electron microscopy. This method has not, however, proved productive with soils, presumably because of the components which are not soluble in HF.

MICROCHEMICAL STAINING

Microchemical staining is a well established technique in petrographic analysis, particularly in the differentiation of the various carbonate minerals and of the different feldspar species. Staining, either single or

multiple, can be performed on the polished surface of either a block or a thin-section. These techniques are all equally applicable in soil micromorphology and can be used to detect or follow the distribution of specific elements or mineral species within the soil fabric. The stain can also be transferred subsequently to an acetate peel (Hawksey, 1969) which has the advantage of isolating the one component sought within the soil fabric, free from the masking or confusion caused by other pigmented components in the soil.

Staining generally proceeds in two stages, though they are often combined in practice. Firstly, the particular ion sought must be released from its host mineral lattices in the specimen surface. This is usually accomplished by acid treatment using either HF for silicates or HCl for carbonates and oxides. A reagent is then added to react with one of the ions released so as to produce a distinctive coloured complex specific to the element sought: the coloured complex can be soluble, adsorbed to a gel carrier (e.g. $\text{Al}(\text{OH})_3$) or precipitated. Ideally the reactions should remain localised with minimal diffusion of the stain from its point of origin so as to achieve maximum resolution. It is also possible to combine staining simultaneously with the preparation of acetate peels by diffusing acetone-soluble reagents through the acetate sheet which is effective in restricting the mobility of the coloured complexes. (Stewart Jones 1974). Once produced on a thin section or transferred to a peel, stains can be viewed by either bright or dark field transmitted illumination, or by reflected light.

Probably the best known staining procedures are those developed to identify carbonate minerals in sedimentary rocks (e.g. Katz & Friedman, 1965; Dickson, 1966). Following a brief (c. 30 second) etching in dilute (0.1N) HCl, the calcium released is stained with Aliza-

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rin Red E (1% aqueous : 1-5 minutes) to give a pink coloration to calcite grains ; magnesian calcites show a purplish tinge , but dolomite itself is not stained, whilst ferroan carbonates can simultaneously be tinged blue by combining a ferricyanide stain (2 % in 2 % HCl : 1-2 minutes) with the alizarin. This procedure has been found useful in following the distribution of detrital calcite and decalcification in Brown Earth profiles developed on calcareous tills. It is interesting however, that pedogenic calcites in these profiles did not take up the stain readily. The calcium in gypsum can also be stained with alizarin although this requires brief immersion in boiling NaOH rather than the acid etch: this procedure has been used to follow the distribution of gypsum in the matrix of samples from gypsorthid profiles. Wieder & Yaalon (1974) have also reported the useful application of microchemical stains to thin-sections in their study of pedogenic calcite nodules in soils from Israel.

Another well established procedure is that developed to differentiate visually the otherwise similar feldspar minerals (e. g. Zschach 1977). The sample is lightly etched (1-10 mins. in HF (40 %) vapour at room temperature and then treated with sodium cobaltinitrite solution (60 % aqueous : 2 minutes) to give a yellow stain to the K-feldspars. Plagioclases are etched a chalky white and can be stained further by treatment with BaCl₂ and one of several dyes such as methylene blue, amaranth or rhodamine. Quartz remains unaffected. These procedures can be used to identify detrital feldspars within soil fabric, but the cobaltinitrite stain does not appear to work with the K-bearing micaceous minerals.

Iron in the form of poorly crystalline pedogenic hydrous oxides can be detected by either of two sensitive stains combined with an acid etch. Potassium ferrocyanide (2% aqueous : 5 mins) gives a distinctive blue-pre-

cipitate (c. f. Eabel 1964). Whilst potassium thiocyanide, (1% : 5 minutes) which can preferably be dissolved in acetone and diffused through an acetate peel, produces a red-brown colour. Associated aluminium in hydrous oxides can similarly be stained pink using a 0.01% ammoniacal ethanolic solution of alizarin-S. The two stains for Fe (blue) and Al (red) can be combined on one peel producing intermediate colours according to composition. The distribution of Mn could probably be followed with the fluorescent dye Arnold's base, and there are many other stains for specific elements in the literature which could no doubt be adapted to soil micromorphological studies. A related but more protracted technique which is described elsewhere (Bajwa & Jenkins, 1977) utilises the selective adsorption of radiotracers such as Ni⁶³ and Ca⁴⁵ on soil components in the polished surfaces of resin-impregnated blocks or sections, their distribution being recorded in the form of autoradiographs.

CONCLUSION

The microfabric of soil is inherently complex by virtue of the diversity of its constituents, both inorganic and organic, and the variety of pedogenic processes that have spatially re-organised these constituents. In studying the microfabric it is therefore desirable to be able to obtain as much information by as many different means as possible to supplement the basic data from the microscopic examination of thin-sections. The preparation of acetate peels can provide a simple, rapid and inexpensive alternative to thin-sections where details of size, shape or distribution of features, uncomplicated by pigmentation, are sought: individual soil components, however cannot always be identified. Serial peels are easily prepared and enable features such as voids to be followed

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into the third dimension, although difficulties arise in the presentation of data. The transference to peels of microchemical stains (e.g. for Ca, K, Fe, Al) can reveal the distribution of selected components (e.g. calcite, gypsum, feldspars, hydrous oxides) within the soil fabric. Such techniques are capable of development to meet different needs in soil micromorphology.

LEGENDS TO FIGURES

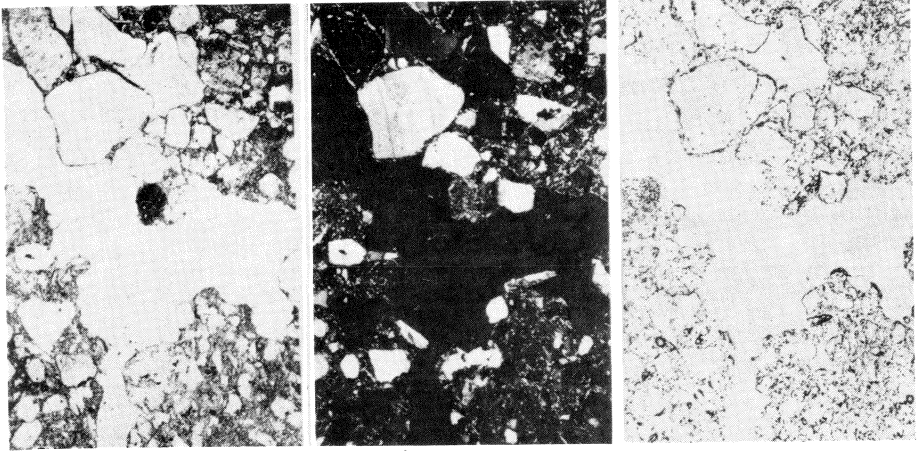
Figure 1 : Microfabric of a Brown Earth (Flint Series: Bw) recorded in an acetate peel (3) as compared to thin-section (1 & 2).

Figure 2 : Representation of a void system in a Brown Earth (Flint Series: Ap) recorded on serial peels taken at 100 μ m intervals.

SUMMARY

Acetate peels, widely used in palaeontology and sedimentary petrology, are a valuable source of information about soil fabric and have the advantage of being easy, quick and inexpensive to prepare. Considerable detail is preserved in the peel at the microscopic level, and although the refinements of optical relief, colour, birefringence, etc., used in thin-sections microscopy are missing, the absence of pigmentation can prove convenient in instances where the latter obscures the fabric. Peels may be prepared from the polished surfaces (1 μ m diamond) of either the resin impregnated block or an uncovered thin section: the peels can be studied directly under the microscope or used as a negative and enlarged photographically. Serial peels (e.g. at 100 μ m intervals) can be used to study void systems in three dimensions.

Microchemical staining is also a well established technique in sedimentary petrology and is equally appli-

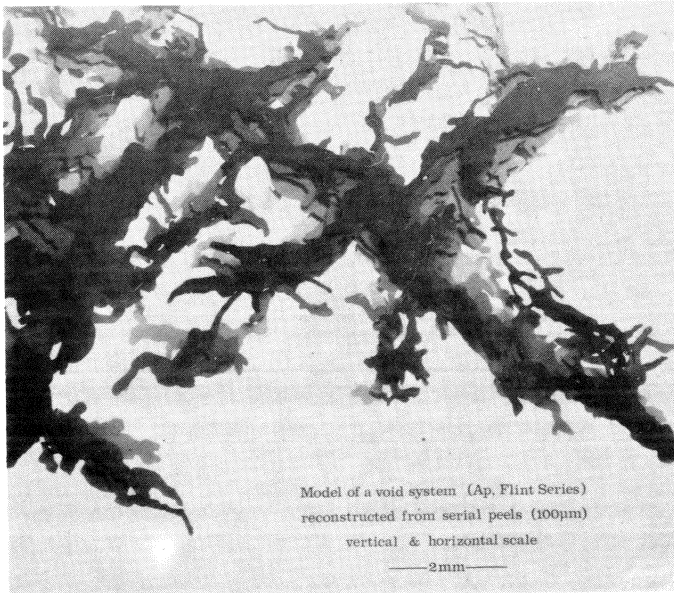


1 plane polarised

2 crossed polars
—200µm—

3 acetate peel

Fig. 1



Model of a void system (Ap. Flint Series)
reconstructed from serial peels (100µm)
vertical & horizontal scale
——2mm——

Fig. 2

cable to soil micromorphology. Elements which can be successfully detected and located by this technique include K (e.g. in feldspars), Ca (in calcite and gypsum) and Fe & Al (e.g. in the amorphous oxide fraction). The stain can be viewed on the thin-section or, in some cases more conveniently, when transferred from the thin section or block surface to an acetate peel where its distribution is unobscured by the pigments in the soil matrix; staining and peel production can also be carried out simultaneously.

This paper will discuss the procedures, scope and limitations of these techniques, illustrated by reference to specific applications.

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