

# **IMPREGNATION TECHNIQUES FOR SOILS AND CLAY MATERIALS**

## **The Problems and Overcoming Them**

**I. Salins and A. J. Ringrose-Voase**  
**CSIRO Division of Soils, GPO Box 639, Canberra, ACT 2601**

### **INTRODUCTION**

Soil scientists often need to examine the internal structure and fabric of soil by observing sections through undisturbed samples using a variety of microscopic and photographic techniques. However unlike rock, soils are usually plastic or friable and cannot be cut without disrupting the structure. Therefore, undisturbed soil samples are usually impregnated with an embedding resin, which allows the samples to be treated like rock material after the resin material has hardened. Impregnated samples are used to make both thin sections and polished blocks. The former are examined using a petrological microscope and used to aid studies of mineralogy, soil genesis and soil structure and fabric. They can also be examined by scanning electron microprobe. Polished blocks can be used to produce images of soil structure using fluorescence photography for meso to microscopic scales, or SEM in backscatter mode for higher magnifications.

A number of problems are encountered during the impregnation process. Firstly, it is difficult to obtain an undisturbed soil sample, especially if the soil is sandy or granular. Field impregnation is possibly the only way to sample sandy or granular soils successfully. Secondly, water usually has to be removed from the sample before the resin can be introduced, since the two are immiscible. Air drying can cause shrinkage and cracking on samples of clay soils. Solvent replacement of water can be used to overcome this problem. We have successfully removed water from a range of samples using dioxane. Thirdly, there are problems associated with impregnating samples with low permeability. We have overcome this using a drip vacuum impregnation technique with good results.

This paper will cover sampling methods, field impregnation techniques, dehydration methods and impregnation alternatives. A fuller description of methods for thin section preparation of soils is given by Murphy (1986).

### **SAMPLING**

Samples are taken using Brewer tins, cylindrical cores or as lumps of material. Brewer tins (150x90x50 mm) are pushed into the soil, carefully dug out and both ends covered with lids. Horizontal sections are obtained by inserting the tin into the surface or a ledge cut at the desired depth and vertical sections by inserting them into a soil pit face.

Alternatively, where there is no room to dig a pit such as in plots in an agronomic field experiment, cylindrical rings can be inserted vertically into the soil. We use 100 mm diameter x 75 mm high aluminium rings sharpened at one end and driven into the ground using a steel guide tube to assure insertion is in a straight line. The walls of the sampling rings should be as thin as possible to reduce the volume displacement and hence compaction necessary to insert the ring. The leading edge is sharpened from the outside to direct compressive forces during insertion.

outwards away from soil being sampled. The ring is usually lubricated with light oil to reduce friction. Least disturbance is achieved by using a constant, steady motion during insertion, for example by pushing by hand or jacking against a vehicle. It can help to initially wet the sample but this could later lead to some surface cracking. In many situations, where the soil is too hard to be sampled by hand and inaccessible to vehicles, a mallet or drop hammer has to be used. Compared to brewer tins, cores have the advantage of providing equidimensional samples which allow both horizontal and vertical polished faces to be obtained. However, their equidimensional shape also increases the distance over which the resin has to impregnate, which can cause problems in soils with low permeability.

If the soil is coherent, a lump of the material can be transported back to the laboratory.

### **FIELD IMPREGNATION**

Field impregnation (Moran *et al.*, 1989a) can be used if the material cannot be sampled successfully. As mentioned earlier, sandy or crumbly soils are difficult to sample without disturbance but can be impregnated in situ with an epoxy resin which sets in 24 hours even in moist soils. The resin mixture consists of the following ingredients from Ciba-Geigy (TM):

Base Resin	LC191	170g
Reactive diluent	Dy026	170g
Hardener	LC249	160g

It is important that quantities are measured accurately as variation can alter handling time. The mixture is applied by a dripper to the surface within 60 minutes of mixing. This time is reduced in high ambient temperatures. After 24 hours, when the resin is cured, the impregnated soil is excavated and transported to the laboratory. In the laboratory it is cut and surface impregnated if necessary, before preparation of polished faces or thin sections. The method has a few drawbacks such as the lack of penetration and incomplete impregnation, as only the pores continuous to the surface are impregnated.

### **DEHYDRATION**

If samples have not been field impregnated, they must be dehydrated before impregnation, as the presence of water can cause the resin to become cloudy and/or inhibit complete polymerization. Soils with low clay content are air or oven dried at around 40°C without shrinkage altering the structure. Clay soils, especially those with high proportions of shrink/swell clay, cannot be air-dried without their structure being disrupted by shrinkage. The simplest method to dehydrate such soils is solvent replacement in the liquid phase (Miedema *et al.*, 1974). Freeze drying (Stephan, 1969; Jongerius and Heintzberger, 1975) or solvent replacement in the vapour phase (FitzPatrick, 1974) can also be used. Both the latter techniques are only satisfactory with small samples. Murphy (1982) compared oven-drying, freeze drying and solvent replacement as methods of dehydrating clay soils and found that both oven drying and freeze drying caused shrinkage, and that freeze drying additionally caused artificial cracks to form in the centres of samples where freezing was slower.

### *Choice of Solvent*

Acetone is the most frequently used solvent, because it is inexpensive and relatively harmless. However, both in theory and practice we have found dioxane to be preferable. Greene-Kelly (1973) and Chartres *et al.*, (1989) found that in comparison to acetone, dioxane appears to cause less shrinkage, while acetone appears to increase the likelihood of samples collapsing, especially when they contain smectitic clays. Further evidence for the latter finding is the work of Brindley *et al.*, (1969) who measured the basal spacing of Ca-montmorillonite in various water-organic solvent mixtures. When the solvent was dioxane, the basal spacing decreased from that in pure water (19Å) to that in pure dioxane (15Å) when there was only a small proportion of dioxane in the mixture. In contrast, when the solvent was acetone, the basal spacing greatly increased to a semi-dispersed state (>50Å) when there was a small proportion of acetone. As this proportion increased, the spacing gradually decreased to that of pure acetone (17Å). Whilst this study was on separated clay, it implies that as acetone diffuses into a wet sample it may exert a high enough swelling pressure for the sample to collapse at a macroscopic scale.

Disadvantages of dioxane are its expense and greater health risk.

Another possible solvent is methanol, but we have not used it in this laboratory.

### *Equipment*

In our laboratory we have modified the solvent replacement technique to allow the handling of large batches of samples, increase the rate of exchange and recycle the solvent (Moran *et al.*, 1989).

The samples are placed in four tanks (450x350x150 mm) on a false floor of perforated stainless steel 20 mm above the base of the tank. The four tanks hold 48 cores, 100 mm diameter x 75 mm high. A 240V magnetic drive impeller pump with a high density polyethylene body is used to circulate solvent from a 60 litre reservoir into the bottom of the tanks (connected in parallel). The solvent returns to the reservoir via an overflow tube whose diameter is twice that of the inlet. This system ensures that the overflow rate equals the inflow, without needing adjustable valves, so that the tanks are kept full without overflowing the sides. The movement of solvent increases the rate of exchange (Murphy, 1985). In addition, the solvent is periodically, usually daily, drained from the tanks for 30 minutes and pumped back into the tanks. This procedure drains the macropore space thereby hastening equilibrium.

The tanks and associated hardware must be built from materials compatible with dioxane. High density polyethylene, polypropylene, nylon, Teflon or stainless steel are all suitable. Rubber, commonly found in pumps as a gasket material, and PVC are unsuitable.

### *Solvent Recycling*

As the solvent circulates it comes into contact with the desiccants placed in the reservoir. These desiccants remove water from the solvent which hastens the exchange process by ensuring the maximum concentration gradient between the solvent and the sample. In addition, the amount of solvent used is minimized, thereby reducing costs and any environmental hazards.

When water contents are greater than 3% a 5 kg bag of polyacrylamide (Alcosorb AB®) granules is placed in the reservoir. This desiccant can absorb large quantities

of water, provided it is not doing so against a large concentration gradient. The bag is replaced every 24 - 48 hours and regenerated, after evaporating off any surplus solvent, in an oven at 105°C.

We find it useful to have three bags so that, at any one time, one is in the reservoir, one evaporating and one in the oven. When the water content is less than 3%, polyacrylamide is no longer effective and is replaced by 8 kg of type 4Å molecular sieve in stainless steel, mesh baskets. This material is an artificial clay (zeolite) with a nominal 4Å pore size which are large enough to absorb water molecules but not solvent molecules against virtually any concentration gradient. However, it only absorbs relatively small quantities of water and must be regenerated at temperatures over 180°C. Therefore it is only used in the latter stages to reduce the water content from 3% to 0.5%, at which point samples are dry enough for impregnation. As with the polyacrylamide, we find it useful to have three baskets.

#### *Measurement of water content and solvent*

The water content of the solvent mixture is monitored periodically. We use two methods, depending upon the concentration of water in the mixture. In the early stages, when the concentration of water in the solvent is greater than 5%, equal quantities of solvent/water mixture and kerosene are mixed and the aqueous layer allowed to settle out (Moran *et al.*, 1989b). This layer is drained off and its volume is measured. The approximate water concentration in the solvent/water mixture can be obtained from a calibration curve. The method is not precise as the composition of kerosene is not fixed.

Later in the drying procedure, the concentration is determined enthalpimetrically, by measuring the temperature change on dissolving a 3 ml of distilled water in 100 ml of mixture using a thermistor circuit (Moran *et al.*, 1989b). The water is placed in a small vial inside a flask and surrounded by solvent mixture. The flask is left for two minutes for the temperature to equilibrate. The circuit is then zeroed and the vial upset so that the water mixes with the solvent mixture. The dissolution is endothermic when the water concentration in the mixture is less than 5%. As the temperature changes so does the resistance of the thermistor, causing the current in the circuit to change. The concentration is found by comparing the maximum deflection of the ammeter in the circuit with a calibration curve. This method has the advantage of becoming more sensitive as the water concentration in the solvent decreases.

The solvent dehydration procedure generally takes about six weeks.

### **LABORATORY IMPREGNATION**

The final stage of sample preparation, before it can be sectioned, is impregnation with a clear embedding resin. Impregnation stabilizes the soil so that it can be cut on rock sectioning equipment. We employ two methods : a standard method for 'easy to impregnate' soils and a vacuum drip technique for solvent dehydrated samples or dense samples.

### **RESIN**

There are two broad types of resin suitable for soil impregnation : epoxy resins and polyester resins. A resin must have suitable optical properties and cure without shrinkage or overheating. It must also have low enough viscosity and remain liquid for long enough to penetrate the pore space in the sample. For this reason we

prefer to use polyester resin, which can be diluted with methyl methacrylate monomer to make it less viscous and whose rate of curing is controlled by the amount of catalyst added. By using small quantities of catalyst, the mixture remains liquid for longer and has longer to penetrate the sample. In contrast, the curing rate of epoxy resins is controlled by temperature and is generally much shorter.

In addition, we add a fluorescent dye to the mixture, to allow pore space in thin sections or polished faces to be identified and photographed under UV light.

The mixture we use consists of :

<b>Ingredient</b>	<b>Quantity (by weight of CR64)</b>
CR64 clear, polyester, casting, embedding resin (Daystar Pty Ltd)	1 part
methyl methacrylate monomer	0.5 - 1 parts
Cumene hydroperoxide catalyst	0.005 - 0.001 parts
UVtex OB fluorescent dye (Ciba-Geigy Pty Ltd)	0.0015 parts

As heat will accelerate polyester resin polymerization, although less so than for epoxy resins, resin storage and impregnation should occur in a controlled temperature environment (approximately 20C).

### **STANDARD IMPREGNATION**

Samples are placed in a polypropylene tub. For standard impregnation the quantity of methyl methacrylate is generally 0.5 parts to 1 part resin with 0.005 parts catalyst. Initially the resin mixture is poured around the base of the sample and not over it. This minimizes air entrapment and allows the resin to be drawn up from the base of the sample by capillary action. After a period of time, usually several hours depending upon the material, the surface wets up, after which the sample is covered with a 2 - 3 cm layer of resin mixture.

The sample is then carefully moved to a vacuum chamber and placed under vacuum at about -85kPa. For the first 30 minutes the vacuum is cyclically increased and reduced to atmospheric pressure in order to remove entrapped air bubbles. It is then left at -85kPa for a period of 5 - 6 hours to remove any entrapped air.

Polymerization usually takes 3 - 4 weeks in order to prevent heat generation and allow time for the resin to penetrate fully. After polymerization is complete, samples are oven cured at 40°C to remove any tackiness. They are then ready for sawing and sectioning.

## **VACUUM DRIP IMPREGNATION**

We have found that the standard method gives poor impregnation when samples are from soils with degraded structure and low permeability and/or have been dehydrated by solvent replacement. In such cases, the resin may not penetrate to the centres of the samples and solvent is frequently present. To improve penetration and ensure complete solvent removal, since solvent will prevent resin fully curing, we developed a vacuum, drip method of impregnation.

In this procedure, the quantity of monomer used is increased to 1 part/1 part resin to reduce viscosity further. The quantity of catalyst is also reduced to 0.001 parts/1 part of resin to slow the rate of polymerization and allow longer for the solvent to escape and the resin to penetrate.

The samples are placed in a tub inside a vacuum chamber with a 5 mm diameter inlet tube which allows resin to be slowly introduced, drop by drop, to the bottom of the tub after the chamber has been evacuated. The drip rate is controlled so that the samples are covered after 6 - 8 hours. During evacuation, the chamber is bled slightly via the purge valve. Whilst this reduces the vacuum somewhat from -70 to - 80 kPa, it draws off the solvent vapour to ensure that the atmosphere in the chamber does not become saturated with solvent and prevent further evaporation from the sample.

## **SAFETY ISSUES**

The major hazards in the above procedures are from solvent drying, in particular when dioxane is used as the solvent. Dioxane is listed as a possible carcinogen and should be treated with caution. Both dioxane and acetone present a fire hazard. In order to minimize the hazards, the solvent tanks and reservoirs are sealed as much as possible and housed in a fume cupboard. Additional precautions include wearing a powered respirator with solvent extractors and mounting the electric pump away from the tanks.

The materials used for the resin mixture have a fire hazard; can irritate skin and have noxious vapour. Apart from the standard precautions such as wearing protective clothing and carrying out operations in a fume cupboard where possible, wearing a respirator also reduces health dangers.

## **CONCLUSIONS**

Although soils are usually loose or soft, they can still be successfully sectioned after resin impregnation. A variety of sampling, dehydration and impregnation techniques can be used depending on the structure and clay content of the soil. The most difficult samples to impregnate are those with high clay content and low permeability. Such samples require dehydration by solvent replacement and vacuum, drip impregnation. Using such techniques, we can generally achieve good impregnation.

## REFERENCES

- Brindley, G.W., Wiewora, K. and Wiewora, A. (1969)  
Intracrystalline swelling of montmorillonite in some water organic mixtures (Clay organic studies XVII). *American Mineralogist* **54**, 1635 – 1644
- Chartres, C.J., Ringrose-Voase, A.J. and Raupach, M (1989)  
A comparison between acetone and dioxane and an explanation of their role in water replacement in undisturbed soil samples. *J. Soil Sci* **40**, 849 – 863
- FitzPatrick, E.A. (1974)  
'Micromorphology of Soils'. (Chapman and Hall: London.)
- Greene-Kelly, R. (1973)  
The preparation of clay soils for determination of structure. *Soil Sci* **24**, 277 – 283
- Jongerius, A. and Heintzberger, G. (1975)  
Methods in Soil Micromorphology. A technique for the preparation of large thin sections. Soil Survey Papers No. 10. (Netherlands Soil Survey Institute: Wageningen, The Netherlands.)
- Miedema, R., Th. Pape and van der Waal, G.J. (1974)  
A method to impregnate wet soil samples, producing high quality thin sections. *Neth. J. Agric. Sci.* **22**, 37 – 39
- Moran, C.J., McBratney, A.B. and Koppi, A.J (1989a)  
A rapid method for analysis of soil macropore structure. Specimen preparation and digital binary image production. *Soil Sci Soc. Am J.* **53**, 21 – 928
- Moran, C.J., McBratney, A.B. Ringrose-Voase, A.J. and Chartres, C.J.(1989b)  
A method for the dehydration and impregnation of clay soil. *J. Soil Sci.* **40**, 569-575
- Murphy, C.P. (1982)  
A comparative study of three methods of water removal prior to resin impregnation of two soils. *J. Soil Sci.* **33**, 719 –735
- Murphy, C.P. (1985)  
Faster methods of liquid phase acetone replacement of water from soils and sediment prior to resin impregnation. *Geoderma* **35**, 251 – 272
- Murphy, C.P. (1986)  
'Thin Section Preparation of Soils and sediments'. A.B. Academic: Berkhamsted, Herts, UK
- Stephan, S. (1969)  
Gefrietrocknung und andere bei der Herstellung von Bodendunnschliffen benutzbare Trocknungsverfahren.  
*Z. Pflanzenernahr., Dungung, Bodenkde.* **123**, 131 - 140