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SEPARATION OF SUB-MICRON PARTICLES FROM SOILS AND SEDIMENTS WITHOUT MECHANICAL DISTURBANCE

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Abstract—A method is described for the separation of the finest particles from soils and sediments without mechanical disturbance. Particles are separated through the induction of osmotic stress. Generally, samples are treated with a concentrated sodium salt solution and then exposed to water by diffusion. Naturally sodic samples are simply exposed to water. Solid samples and the swollen and dispersed material they produce are confined by dialysis tubing. Examples show that the method gives a size gradient of particles in a vertical column of suspension. The compositions of particles can vary with size. The method can be used to show the effects on separated particles of ions other than Na^+ and also of other physico-chemical treatments of soils and sediments. It is inexpensive and requires little labor.

Key Words—Clays, Dialysis, Dispersion, Mobility, Particles, Sediments, Separation, Size, Soils, Submicron, Swelling.

INTRODUCTION

The finest clay material almost invariably dominates both chemical and physical properties of soils even though the coarser sand and silt fractions are often predominant by weight or volume (e.g., Robert and Chenu 1992). Characterisation of the fine material often requires its separation from the whole soil. Separation by centrifugation is common. However, mechanical disturbance of the soil inevitably accompanies centrifugation and may change the nature and distribution of separated particles. A method described here which uses dialysis tubing enables the separation of sub-micron particles from soils and also from sediments. The method avoids mechanical disturbance, is inexpensive and requires little labor. It also provides a visual demonstration of swelling, dispersion and the subsequent diffusion of fine particles in soils and sediments and enables the measurement of extent of occurrence of these phenomena.

PRINCIPLE OF THE METHOD

In this method, particles are separated by inducing sufficient osmotic stress between them to cause dispersion. Subsequent diffusion against gravity produces a size gradient of particles for sampling.

The use of osmotic stress as a "chemical hammer" (Clapp and Emerson 1965) to assess the strength of bonds between soil aggregates was initiated by Emerson (1954). By its use, degree of dispersion of a sample is measured when the easily exchangeable cations are replaced by Na^+ ions and electrolyte concentration is reduced.

In the new method, osmotic stress is normally applied to samples by treating them with a concentrated solution of a sodium salt to replace exchangeable cations with Na^+ and then exposing the Na^+-exchanged sample to water. Osmotic stress can also be induced in soils and sediments which are naturally sodic, and hence dispersive (Gupta and Abrol 1990), by their contact with water without additions of sodium.

PROCEDURE

The gadgetry used in conjunction with dialysis tubing has undergone evolution since the inception of the method. The complexity of the procedure to be followed depends on the aim of the application of the method.

Procedure for demonstration purposes

A concentrated solution of NaCl is added slowly to an air-dried whole sample in a dry dialysis tube which is sealed at both ends by tying them tightly with rubber bands. The tube containing the treated sample is hung vertically in a cylinder overnight. The cylinder is then filled with deionised water which is replaced regularly by siphoning until enough electrolyte has been removed from the equilibrated solution that it shows no precipitate with $AgNO_3$ solution. As the electrolyte concentration is decreased the sample may swell, with dispersion generally beginning before final dilution. In the example shown in Figure 1, a sample of a Mollisol (Oxic Argiudoll) or Prairie soil (Stace *et al* 1968) from Glen Innes, New South Wales, was treated with 1 M NaCl and dispersion was allowed to occur for 34 days.

Procedure for analytical purposes

The outfit we now use is illustrated in Figure 2, and shown in operation with a saline sediment from Chowilla, South Australia in Figure 3.

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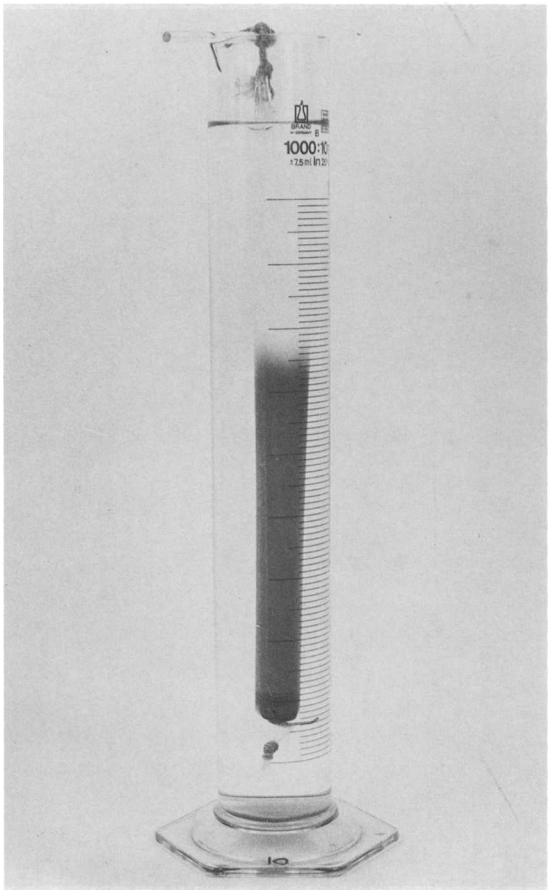


Figure 1. The effects of exposure to water for 34 days on a Na-treated 5 g sample of a Prairie Soil (Oxic Argiudoll). The dispersed material has diffused to a distance of 260 mm above the original surface in 34 days. The procedure for demonstration purposes has been employed.

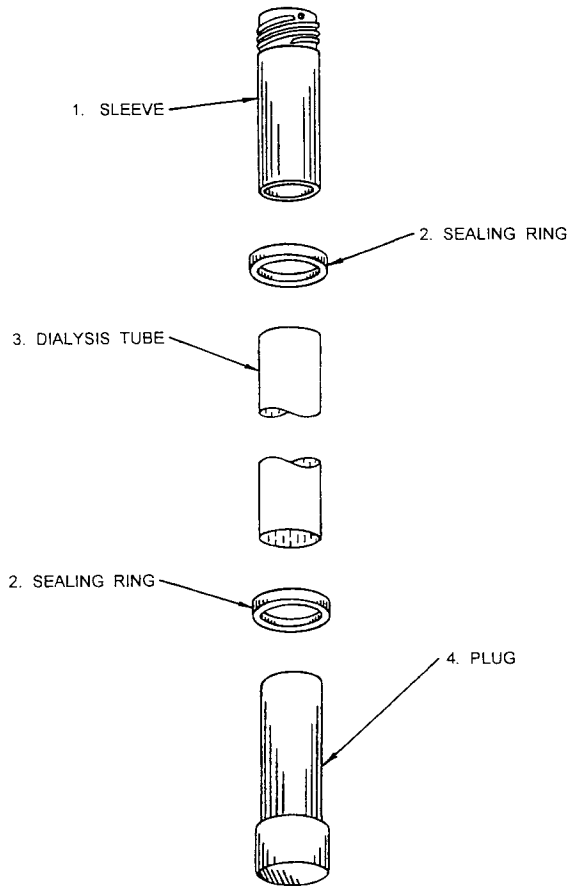


TABLE OF COMPONENTS	
1. SLEEVE	5 ml. sample tube; 'Disposable Products' 1988 cat. no. 21523 without lid; base removed; 3 holes around top of wall to suspend outfit by.
2. SEALING RING	Silicone rubber tubing 12 mm. I. D. 16 mm. O. D. 3 mm. long
3. DIALYSIS TUBE	'Union Carbide' no. 20; 25 mm. flat width, transparent cellulose.
4. PLUG	5 ml. sample tube; 'Disposable Products' 1988 cat. no. 21523 with lid.

Figure 2. An exploded view of the dialysis outfit used for the analytical particle separation procedure. The parts are described in the Table of Components.

Assembling the outfit. 1. Cut the desired length of dialysis tube (we generally use 160 mm), with square ends, and immerse it to soak in distilled water.

2. Fit a Sealing Ring over the Sleeve, with the lower face of the Sealing Ring about 10 mm above the lower end of the Sleeve, but, assuming the end of the Sleeve is straight and square, the same distance from the end all the way around.

3. Put about 2 g of glass beads or similar ballast into the vial to be used as a plug, add water to take up the rest of the space in the vial and fasten its lid.

4. Fit a Sealing Ring to the Plug, as to the Sleeve in step 2, taking similar care to leave the same distance between the upper face of the Sealing Ring and the upper end of the plug all the way around.

5. With the dialysis tube having soaked for at least 2 min, remove it from the water and open one of its ends by rubbing it between two fingers. Manipulate the opened end of the dialysis tube over the lower end of the Sleeve and slide it on to meet the lower face of the Sealing Ring all the way around.

6. Taking care not to upset the positioning of the dialysis tube on the Sleeve, blow through the top end of the Sleeve so as to open the rest of the dialysis tube. Manipulate the bottom end of the dialysis tube over the top end of the Plug and, having rewet the outside of the tube if necessary, slide it on to meet the upper face of the Sealing Ring all the way around. With two ends of the dialysis tube in position on the Sleeve and Plug, allow it to dry until the ends are taut and no longer slide on the sleeve and plug.

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7. Work the upper Sealing Ring over the end of the dialysis tube by stretching one part of it outwards and away from the Sleeve and edging it downwards, and then the same all around the Sealing Ring until its lower face is flush with the lower end of the sleeve. Work the lower Sealing Ring upwards in the same way until its upper face is flush with the upper end of the Plug.

8. Contrive a device, such as a short piece of flexible tube that will make an airtight fit with the top end of the Sleeve, with a Hoffmann clamp on it tightened only so far as to make it grip the tube without closing it, and attach it to the top end of the Sleeve.

9. Reimmerse the dialysis tube in distilled water, taking care not to let water spill into the outfit through the opening at the top. Allow the dialysis tube to soak for at least 2 min, then remove the outfit from the water and inflate it by blowing through the device attached to the Sleeve (step 8), then, while maintaining the pressure inside the outfit, close off the device to leave the dialysis tube inflated. Hang the outfit vertically for the dialysis tube to dry in air.

10. When the dialysis tube is dry and rigid, remove the sealing device from the top end of the Sleeve and tie the end of a roughly 150 mm length of sewing thread to each of the 3 holes in the top end of the Sleeve wall. Lower the outfit, plug first, into a 500 ml measuring cylinder, with the threads draping over its edge to the outside, and when the top of the Sleeve is almost level with the top of the cylinder, arrange the threads roughly equidistantly around the cylinder's circumference, and while holding them in position with one hand around the outside, fit a wide rubber band over the top to hold the threads with its top edge about 15 mm below the top of the cylinder. With a rod, push the outfit down until the top of the Sleeve is below the intended level of fluid in the cylinder (generally the 500 ml mark in our experiments) and ensure that the outfit hangs centrally. The bottom of the Plug should be far enough above the floor of the cylinder to ensure a gap remains when the dialysis tube wets up, whereupon its length will increase by about 5%. Fix the threads in position with pieces of adhesive tape close to the top of the outside of the cylinder and remove the rubber band.

Assembling the filling gear. The filling gear is illustrated in Figure 4. The Soil Tube is long enough that, when lowered into the outfit, it reaches within 10 mm of its floor, while protruding from its top far enough to accommodate the Fluid Tube restrainer (see Step 7 below), the clamp holding the Soil Tube and the Funnel Joiner. It also requires a Fluid Tube Tip holder, which is about 50 mm in length, and a Fluid Tube Tip. The latter is made by cutting a segment from a Pasteur pipette which when lodged in the Fluid Tube Tip Holder protrudes 5 mm above and 20 mm below the Holder. Its ends are smoothed with fine abrasive paper. The gear is then assembled as follows:

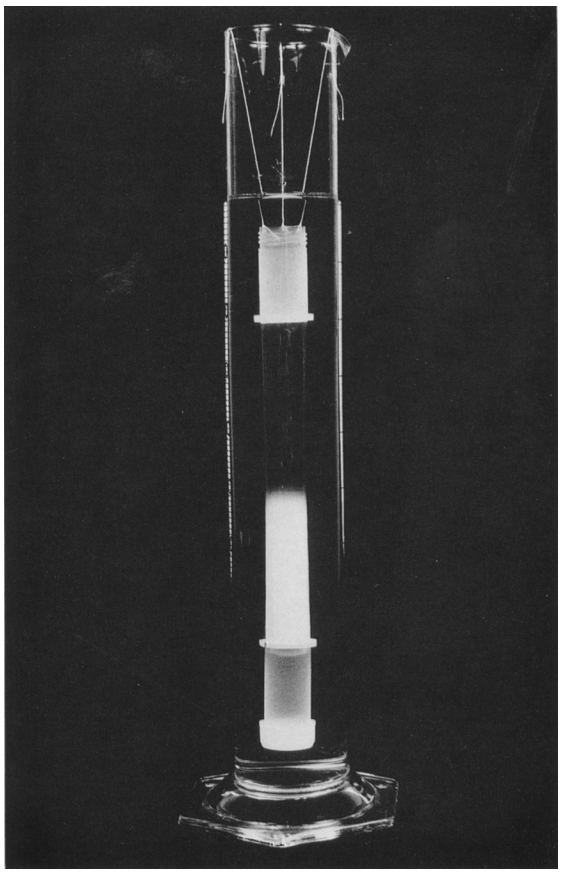


Figure 3. The effects of exposure to water for 12 days on a 1 g sample of a saline sediment. The procedure for analytical purposes has been employed.

1. Lash the Fluid Tube Tip Holder to the side of the Soil Tube using a strip of plastic food-wrapping film, at such a height that when the Fluid Tube Tip lodges in the holder, its narrower end protrudes about 10 mm beyond the end of the Soil Tube.

2. Attach the Funnel to the top end of the Soil Tube using a suitable length of flexible tube as a joiner.

3. Attach one end of the Fluid Tube to the tip of the Burette using a suitable joiner and its other end to the Fluid Tube Tip by pressing it firmly into the flared end of the Tip.

4. Suspend the Soil Tube vertically using a clamp attached to a stand whose height can be adjusted with steady motion, e.g., by rack-and-pinion or on a laboratory jack, so it can be lowered into the dialysis outfit in its cylinder.

5. Suspend the burette, preferably on the same stand as the Soil Tube, with its lowest intended fluid level above the bottom end of the Fluid Tube Tip.

6. Fill the burette with the fluid to be used (most commonly either 1 M NaCl or deionised water), allowing the fluid to fill the Fluid Tube also, and some

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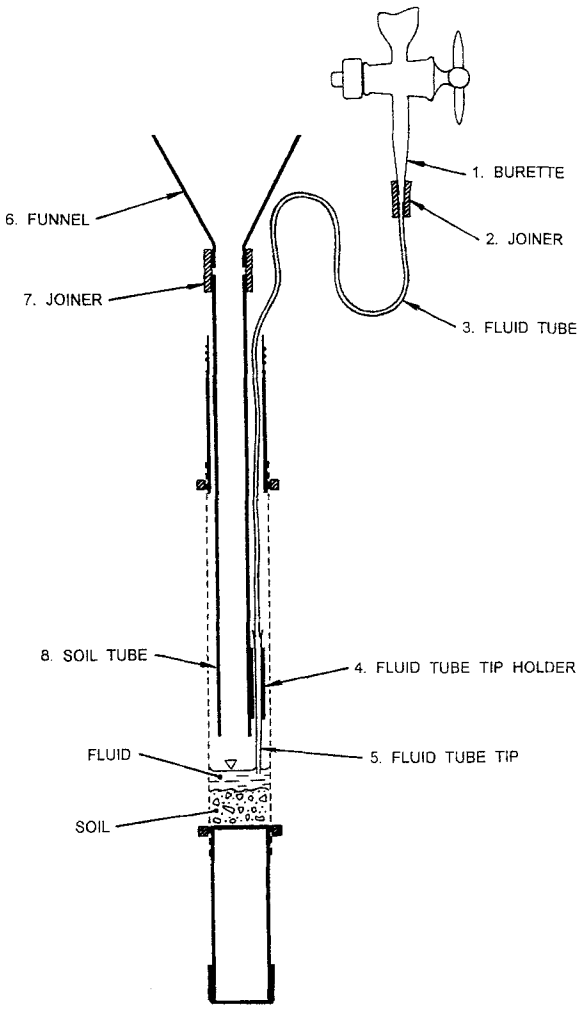


TABLE OF COMPONENTS	
1. BURETTE	Suitable fluid reservoir with regulated outflow.
2. JOINER	Flexible tube for joining fluid tube to burette tip.
3. FLUID TUBE	Thin walled flexible tubing; 1 mm. I. D.
4. FLUID TUBE TIP HOLDER	Glass tube; medium wall, 4 mm. O. D.
5. FLUID TUBE TIP	Glass; commonly broken from the end of a Pasteur Pipette.
6. FUNNEL	Throat diameter similar to I. D. of soil tube.
7. JOINER	Flexible tube for joining funnel to soil tube.
8. SOIL TUBE	Glass tube; medium wall, 9 mm. O. D.

Figure 4. The experimental arrangement for filling the dialysis outfit with solid sample and solution. The parts are described in the Table of Components.

to run out of its tip as a rinse. Close the burette tap, hold the Fluid Tube Tip above the fluid level in the burette and open the tap to allow fluid to run back to leave about 10 mm at the end of the tip free of fluid. Close the tap again, wipe the Tip dry, and slide it downwards into its holder on the Soil Tube until it comes to rest under its own weight, so that it can still be dislodged from its resting position by a gentle tug.

7. Restrain the Fluid Tube by fixing it to the Soil Tube near its upper end e.g., with a *c.* 10 mm length of flexible tube cut open along its wall and made to embrace the Soil and Fluid Tubes, holding them gently together, so that the Fluid Tube hangs alongside the Soil Tube and can be moved up and down.

Filling the outfit and cylinder with solid sample and fluid. The sample generally consists of air-dry <2 mm aggregates. The amount of soil or sediment to be used is governed by such factors as the amount required for analysis, which, in turn, is dictated by the sub-micron particle content of the sample. In our experiments, between 1 and 6 g of sample have been used, according to the following procedure:

1. Align the Soil Tube/Fluid Tube combination over the outfit, wind the tubes downwards so they enter the outfit, and stop when the Fluid Tube Tip is about 1 mm from its floor.

2. Open the burette tap far enough to allow fluid to flow slowly onto the floor of the outfit, to cover it without trapping any air bubbles. As the level of fluid in the outfit rises, use the winding mechanism to keep the fluid surface drawn up to the bottom of the Fluid Tube Tip to keep the fluid from wetting the sides of the Tip or forming a bridge between it and the dialysis tube. Halt the flow when the fluid level reaches about 2 mm, the fluid having touched the dialysis tube.

3. Wind the Fluid Tube Tip out of contact with the fluid. Dislodge the Tip from its resting position by pulling the Fluid Tube upwards and secure the Tube in such a position that the bottom end of the Tip is about 5 mm above the bottom end of the Soil Tube.

4. With the end of the Soil Tube about 10 mm above the surface of the fluid, pour the solid sample through the Soil Tube via the Funnel into the fluid, slowly so as to allow air entering the fluid with the sample to escape. If the sample piles up unevenly, it may be redistributed by tapping very gently with a glass rod on the lower Sealing Ring. Stop pouring before the sample juts above the fluid surface.

5. Wind the Soil Tube/Fluid Tube combination to a height at which the Fluid Tube Tip, when lodged in its holder, will not dip into the fluid, and lower the Fluid Tube until the Tip lodges in its holder. Wind downwards to bring the Fluid Tube Tip just into contact with fluid, then upwards so that the fluid surface is drawn up to the bottom end of the Tip. Open the burette tap only far enough to permit a flow of fluid

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slow enough not to disturb any fraction of the sample. Use the winding mechanism to keep the fluid surface drawn up to the Tip as the fluid level rises and halt the flow when the level is about 2 mm above the top of the sample. Wind upwards to take the Tip out of contact with the fluid again, lift the Fluid Tube to dislodge the Tip and secure the tube so that the bottom end of the Tip is about 5 mm above the end of the Soil Tube.

6. Repeat steps 4 and 5 until all of the solid sample has been poured into the outfit and levelled, then add fluid as in step 5, keeping the bottom end of the Fluid Tube just beneath the fluid surface. If the fluid is water, continue until the level is just below the holes at the upper end of the sleeve (Figure 2), increasing the flow rate when it becomes possible to do so without disturbing any fraction of the solid sample. The flow rate may be increased substantially if the Soil Tube/Fluid Tube combination is replaced by a glass tube of larger e.g., 3 mm bore when the fluid level is at a safe distance (*c.* 20 mm) above the sample, and the remainder of the water added through that tube. If the fluid is salt solution, add it until its level is about 10 mm above the sample.

7. Add fluid to the cylinder, outside the outfit, gradually enough that the outfit is not swayed by turbulence e.g., *via* a glass tube suspended with its bottom end just above the floor of the cylinder. If the fluid is water, continue until the outfit is wholly submerged and the desired fluid level is reached. It is desirable to have a reserve of at least 30 ml of fluid above the top end of the sleeve prior to sampling. If the fluid is salt solution, add it until its level is equal with the level of solution inside the outfit.

8. Seal the cylinder against evaporation using e.g., plastic food wrapping film.

Equilibration and fluid changes. Although the only requisite is that electrolyte surrounding the sample not inhibit dispersion, our aim has been to rid the fluid of electrolyte by replacing the fluid outside the dialysis outfit with deionised water until, when equilibrated with that inside, it has an electrical conductivity (E.C.) <10 $\mu S\ cm^{-1}$, or shows no precipitate with $AgNO_3$ solution, where the sample has been soaked in a solution of chloride salt.

Where the sample has been contacted with water only, usually only one replacement is necessary, if any at all. Where the sample has been contacted with a salt solution, remove the solution outside the dialysis outfit after a minimum of 24 hours, then add water to the outfit and cylinder as in steps 6 and 7 of the last section, taking care not to mix water with the salt solution in the outfit by turbulence. We have found that enough electrolyte is removed if the water is first added in the morning, changed at the end of the day and then again the next morning.

When soils contain considerable organic matter, microbial growth, which can destroy the dialysis tubing, can be prevented e.g., by the addition of *c.* 1 μg mercuric chloride/ml to the water in the cylinder outside the dialysis tubing assembly.

Sampling. Sampling of dispersed material is usually carried out after the rate of diffusion of material has slowed considerably. This occurs after about 30 days.

Clay which disperses from soil or sediment samples often forms layers which are discernible by eye because of differences in color and/or density. It may be sampled according to this layering, or sequentially by depth. In our experiments, 1 ml samples of even the most highly dispersed (and therefore the most dilute) material from solid samples of only 1 g generally yielded sufficient material (2–3 mg) for both XRD analysis of material sedimented on to a silicon wafer and particle size analysis by photon correlation spectroscopy. If a substance such as dissolved organic matter outstrips the clay in ascent, it may be periodically removed selectively while the clay is given time to migrate upwards, or finer fractions of clay may be treated the same way to allow coarser fractions to rise. Samples are taken before the front defined by the most dispersed material has reached the top of the outfit. Dispersed or dissolved material is removed as follows:

1. Mount a pipette with a diameter to comfortably fit inside the Sleeve on a stand of adjustable height above the outfit.

2. Attach a bulb-type pipette filler to the top of the pipette and lower the pipette into the outfit by adjusting its stand, until its tip is about 1 mm above the highest suspended or dissolved material which is distinct from the fluid outside the outfit.

3. Draw the first sample of suspension or solution into the pipette, gently so the sample is drawn from either side of the pipette rather than from below, one hand operating the pipette filler, the other working the stand to lower the pipette, as the outfit is cleared of suspension or solution and it is replaced by fluid from above. A slow but steady rate of movement of sample into the pipette can be achieved through control of the pipette filler by operating the valve with a Hoffmann clamp.

4. When the chosen sample has been taken into the pipette, stop winding the pipette downwards but continue to draw fluid gently into the pipette so some (*c.* 0.1 ml) clear fluid is drawn in below the sample as a buffer.

5. Wind the stand upwards to withdraw the pipette from the outfit and the cylinder. While withdrawing the pipette, use the pipette filler to draw clear fluid slowly into it, to at least counteract the resultant change in head between the fluid inside and outside the pipette and thereby prevent the sample from escaping from the pipette.

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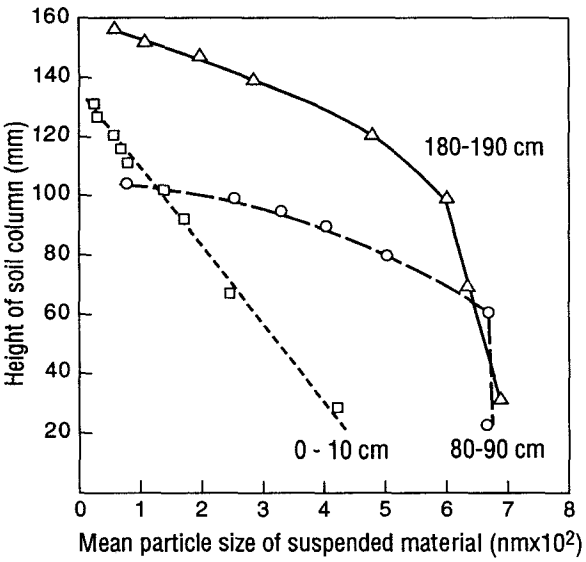


Figure 5. The mean sizes of particles in successive aliquots of suspended material taken from the dispersed columns of samples of three horizons of a Humic Gley Soil (Umbraquult) which had been Na-treated then exposed to water.

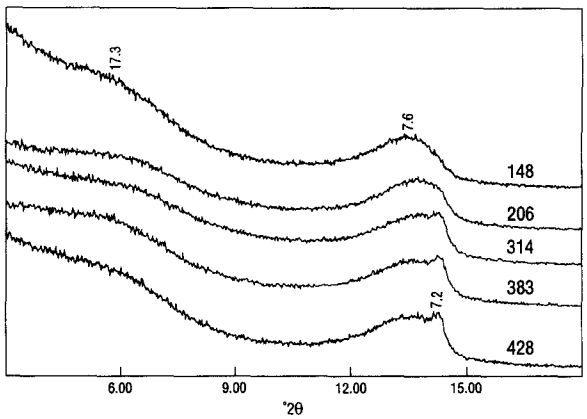


Figure 6. X-ray diffraction patterns following treatment with ethylene glycol of the top 5 successive 5 ml samples taken from the column of suspended dispersed material from the Prairie Soil shown in Figure 1 after 105 days of reaction. Patterns are offset from one another. Spacings of major peaks are given in Å. The mean size in nm of particles in each sample is written horizontally.

6. Bottle the sample and take subsequent samples, keeping the level of fluid in the cylinder above the top of the Sleeve by replenishing the fluid in the cylinder if necessary.

EXAMPLES

Treatment by the demonstration procedure of the Mollisol (Figure 1) has left the bulk of the soil remaining at the base of the dialysis tubing bag. Fine material has dispersed from the main soil mass to form a vertical column in suspension.

Samples of various horizons of an Ultisol (Umbraquult) or Humic Gley soil ("UNE 7") from New South Wales (Stace *et al* 1968) which were Na^+-treated and dialysed against water by the new method showed different behavior with depth in the profile. These different effects in the different horizons are shown in photographs to appear elsewhere (Churchman and Oades 1994). The two uppermost horizons show dispersion but no swelling while lower horizons show substantial swelling as well as dispersion. This behavior is consistent with the trend of clay mineralogy in the profile. All horizons contain kaolinite and a kaolin-smectite interstratified phase with upper horizons being more kaolinitic and smectite content increasing with depth (Churchman *et al* 1994). Mean particle sizes, as determined using a Nicomp 370 sub-micron analyser based on photon correlation spectroscopy, increase at different rates with depth in the column (Figure 5).

In Figure 5, there is a steady linear increase in size with column depth for the topsoil horizon. This is contrasted in the columns for the two lower horizons with a more rapid increase through their upper portions and then a nearly constant particle size within their lower portions. From Figure 5, it appears that particles have a uniform size within the swollen portions of the soils.

The composition of the mixture can change as mean particle size changes with height in the column of suspended dispersed material. Figure 6 illustrates this effect for the Mollisol shown in Figure 1. The uppermost sample, with the finest particles (mean size = 148 nm), gives two broad peaks in X-ray diffraction patterns obtained with CoKα radiation following ethylene glycol treatment that correspond to spacings of 17 and 7.7 Å. These peaks are characteristic of kaolin-smectites (Cradwick and Wilson 1972). The lowest, coarsest sample shown (mean size = 428 nm) gives a sharp peak for a 7.2 Å spacing, which is typical of kaolinite, in addition to two broad peaks at lower spacings than those for the uppermost sample. Spacings in kaolin-smectites decrease as their kaolin contents increase at the expense of smectite (Cradwick and Wilson 1972). The three other samples each give patterns that indicate a gradual variation between the two extremes. They indicate a gradual decrease in kaolinite content and a concurrent transition from kaolin-rich kaolin-smectite to a more smectitic variety of this interstratified mineral as particle size decreases.

As already noted, prior treatment with Na^+ by the addition of NaCl is not necessary to achieve the separation of fine particles when soils or sediments are naturally sodic and hence disperse spontaneously in water. This may be the case even if as little as 6% of the exchangeable cations are sodium i.e., ESP = 6% (Northcote and Skene 1972). The sediment shown in Figure 3 is sodic (with an ESP of *c.* 36—Churchman

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and Weissmann 1995) and has dispersed upon exposure to water.

FURTHER APPLICATIONS

This new technique has other uses besides the extraction of fine particles from soils and sediments affected by Na^+. It also makes it possible to determine the particle size, amount and nature of material dispersed from soils affected by such factors as: 1. different proportions of Na^+ ions on exchange sites; 2. presence of other dispersive ions e.g., K^+; 3. presence of different kinds and amounts of organic matter; 4. chemical treatments e.g., the oxidative removal of organic matter; and 5. mechanical treatments. Some soils e.g., kaolinitic soils at low pH do not disperse when treated with Na^+ alone (Churchman and Foster 1994). These may be dispersed following a suitable pretreatment e.g., by raising their pH, and the new technique then applied to separate their fine material.

For naturally dispersive soils, the method described provides a direct measure of the relative mobilities of soil particles. The relative mobilities of fine particles determine their tendencies to form crusts on soil surfaces and to block pores in the profile (Shainberg and Letey 1984). Blocked pores lower soil permeability, so that water tends to run off the surface and cause erosion, instead of reaching the subsoil where it can be useful to plants. High relative mobilities also lead to transport of eroded soil material as pollutants into watercourses and reservoirs. This method therefore enables assessment of the likely contributions of soils to land degradation. Some of the factors affecting mobilities of sodic soils are examined in a forthcoming publication (Churchman and Weissmann 1995).

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