Method for Particle-size Fractionation of Clays

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This method is the one developed by M. L. Jackson (Soil Chemical Analysis: Advanced Course, Second Edition, published by the author, 1969), as used in our laboratory. The method uses centrifugation to separate the particles, with the centrifuge-tube geometry as illustrated in the figure below.

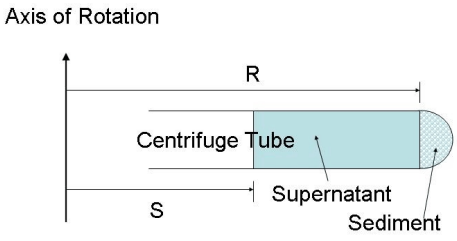

Illustration for Equation 1

With reference to this illustration, the relevant mathematical relationships for centrifuge washing are given by

$$T_m = \frac{63.0 \cdot 10^8 \, \eta \log_{10}\left[\frac{R}{S}\right]}{N^2 D^2 \Delta s} \qquad \textbf{(1)}$$

where T_m is the time for sedimentation in minutes, η is the viscosity in poise at the existing temperature, R is the radius in cm from the axis of rotation to the top of the sediment in the centrifuge tube or bottle, S is the radius in cm from the axis of rotation to the surface of the suspension in the centrifuge tube or bottle, N is the revolutions per minute (rpm), D is the particle diameter in microns, and Δs is the difference in specific gravity between the solvated particle (2.65 g/cm^3) and the suspension liquid (nominally 0.999 g/cm^3).

Proceed according to the following steps:

Step 1. Stir the clay (30 to 50 g) and shake in about 500 mL of 1 *M* NaCl solution for 24 hr.

Step 2. Using the SH3000 rotor (Swing Bucket) for the Dupont Sorvall Model RC5C Plus refrigerated centrifuge and a 250-mL centrifuge bottle.

Step 3. Place a mark on the bottle 7 cm from the bottom and place a mark near the bottom corresponding to the top of the sediment (it will be about 0.2 cm from the bottom). These marks correspond to S =10.5 cm and R = 17.3 cm for the rotor mentioned. If a

different rotor is used, the corresponding values for that geometry should be used. The viscosity in poise is 0.01111 at 16 ˚C and the difference in specific gravity between the clay (2.65) and water (0.999) is 1.651. The centrifuging time depends on the amount of sediment, temperature, rpm, and particle size. The time for sedimentation is 9.2 min when you use 500 rpm and wish to separate the <2 μm size fraction.

Step 4. To collect the clay particles which are less than 2 μm, decant the supernatant. This supernatant contains the particles (less than 2 μm).

Step 5. When fractionating for particles < 0.5 μm, centrifuge the supernatant at 2000 rpm for 9.2 min (set to10 min, sometimes the temperature of centrifuge is below 16 even when you set to 16 ˚C). The particle size of the sediment is between 0.5 to 2 μm. The supernatant has the particles which are less than 0.5 μm. Remember that these parameters are dependent upon your equipment and laboratory conditions.

Step 6. Wash the clay with 1 *M* NaCl solution 4 times and rinse with 0.001 *M* NaCl solution 4 times (or rinse it 2 times and dialyze it). See Lab book for Na saturation.

If you want to fractionate for a purified clay fraction without quartz and iron oxides, after step 5 (skip step 6), follow the procedures beginning with step 7 below:

Step 7. Centrifuge the fractionated sample (step 5) at high speed, such as 12,000 x *g* (Dupont Model Sorvall RC 5C plus centrifuge with SS-34 rotor, 10,000 rpm) and save the supernatant.

Step 8. Replace the supernatant liquid with fresh 1 *M* NaCl solution, then shake well again.

Step 9. Repeat Steps 7 and 8 six or seven times; and wash the sample several times with pure H_2O to eliminate excess NaCl and to disperse the layers. The first washings in this step consist of a rapid, short agitation followed by short centrifugation (12,000 x *g*), which removes the largest Fe oxide particles that are settled to the bottom of the centrifuge tubes. As the number of washing cycles increases, the layers become more dispersed, resulting in a suspension or stable gel which still contains some impurities. Any white or brownish-yellow deposit should be removed, then the gel is separated, diluted in pure H_2O and/or 0.5 m*M* NaCl, and shake vigorously to redisperse the layers. The centrifugation speed is increased progressively with the number of dispersion-centrifugation cycles, beginning at 10,000 rpm and ending at 15,000 rpm. The dispersion-centrifugation cycles are continued until no change in the color of the deposit is observed.

Step 10. The gel is then diluted with 1 *M* NaCl to obtain a concentrated suspension, then excess salts are removed by centrifuge washing with 0.5 m*M* NaCl (see step 6) and the resulting suspension (15 to 25 mg/mL) is kept in a plastic bottle for storage.