

Clay Fractionation Method

$$T_m = \frac{63.0 \times 10^8 \times \eta \times \log_{10} \frac{R}{S}}{N^2 \times D^2 \times \Delta s} \quad (1)$$

$$T_m = \frac{63.0 \times 10^8 \times 0.01111 \times \log_{10} \frac{17.3}{10.5}}{500^2 \times 2^2 \times 1.651} = 9.2 \quad (2)$$

In the equation (1) (ref. M.L. Jackson (1973) Soil chemical analysis-advanced course, ed.2), T_m is the time for sedimentation in minutes, η is the viscosity in poises at the existing temperature, R is the radius in cm of rotation of the top of the sediment in the bottle, S is the radius in cm of rotation of the surface of the suspension in the bottle, N is the rpm, D is the particle diameter in microns, and Δs is the difference in specific gravity between the solvated particle and the suspension liquid.

1. Clay (30 ~ 50 gram) was stirred and shaken in ~500 mL of 1 M NaCl solution for 24 hr.
2. Use the rotor SH3000 (Swinging Bucket) with 250-mL centrifuge bottle.
3. Mark 7 cm from the bottom of bottle and mark the top of the sediment (here, 0.2 cm) in the bottle. So, S is 10.5 cm and R is 17.3 cm. The viscosity in poises 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 spin down over 2 μm size (equation 2).
4. To collect the clay particles which is less than 2 μm , decant the supernatant. This supernatant contains the particles (less than 2 μm).
5. When you fractionate 0.5 μm clay particles, centrifuge the supernatant at 2000 rpm for 9.2 min (set to 10 min, sometimes the temperature of centrifuge is below 16°C 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 . Remind that these parameters are dependent upon your experiment.
6. Then washing the clays with 1 M NaCl solution 4 times and rinse with 1000 times diluted solution 4 times (or rinse it 2 times and dialyze it)- See Lab book for Na saturation.

If you want to fractionate a purified clay fraction without quartz and iron oxides, after step 5 (skip step 6), follow the procedures of step 7 ~

7. The fractionated sample (step 5) is centrifuged at high speed such as 12,000 g (Dupont Model Sorvall RC 5C plus centrifuge with SS-34 rotor, 10,000 rpm) and save the supernatant.
8. The supernatant liquid replaces with fresh 1 M NaCl solution, then shaken again.
9. Steps 7 and 8 are repeated 6 to 7 times; and the sample is washed several times with pure H_2O to eliminate excess NaCl and to disperse layers. The first washings in this step consist of a rapid, short agitation followed by short centrifugation (12,000 g),

which removes the largest Fe oxide particles that are settled to the bottom of the centrifuge tubes. As the number of washing cycles increase, 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 H₂O and/or 0.5 mM NaCl, and shaken 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 any different color deposit is observed.

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 mM NaCl (see step 6) and the resulting suspension (15 ~ 25 mg/mL) is kept in plastic bottle for storage.