Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Frechen, M., Schweitzer, U. and Zander, A., 1996. *Improvements in sample preparation for the fine grain technique.* Ancient TL 14(2): 15-17. https://doi.org/10.26034/la.atl.1996.257

Improvements in sample preparation for the fine grain technique

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(Received 26 June 1996; in final form 29 August 1996)

Fine grain sample preparation was originally developed by Zimmerman (1971). A more convenient method using water flotation was subsequently proposed by Weida & Junding (1989).
The standard preparation technique we have followed in our laboratory is based on Zimmerman (1971) with modifications as proposed by Frechen (1991). We prefer the 4-11m µm grain size range, because the far-travelled material (in case of loess) is mostly finer than 11 µm; by rejecting grains less than 4 µm, the clay content is effectively eliminated.
As part of an empirical testing of the validity and reliability of luminescence dating for loess/paleosol sequences of the last glacial/interglacial cycle, about 950 fine grain samples have been prepared and investigated during the last 5 years. In order to survive such an "impact", the fine grain preparation technique had to be turned into a more convenient and more rapid procedure. Labour intensive steps, like the enrichment of the right grain size and the pipetting procedure, had to be improved. Especially the need to wash the deposition tubes had to be avoided. We also substitute demineralized water for acetone for most of the working steps as suggested by Wintle & Huntley (1980).

Preparation technique

For sediments like loess or fine grained lake deposits crushing is not necessary, unless the minerals hold together by carbonates or other cements. Cemented sediments or volcanics have to be gently crushed with mortar and pestle.
Coarse grained particles (>2 mm diameter) are removed by dry sieving and the remaining material is treated with 0.1 N hydrochloric acid, 0.01 N sodium oxalate and hydrogen peroxide (30%) in order to remove carbonates and organics. After every step the material is washed 3-4 times with demineralized water and is centrifuged.
Then the grain sizes are separated in order to obtain polymineral grains in the grain size range of 4-11 µm. The separation of the fraction < 11 µm is done under the gravitation field as described by Weida and Junding (1989). The settling time of the grains takes about 11 minutes for a water column of 7 cm. The grain size fraction >11 µm falls to the bottom of the beaker. This step is repeated several times to enrich the fraction <11 µm in the solution above the bottom.
The enrichment of the grains > 4 µm is carried out in a centrifuge using a method developed for the separation of the clay fraction for x-ray diffraction analysis of soils (Schweitzer 1992).
The physical background and the formula for calculating the settling of the grains and the centrifugation time for other grain sizes or other room temperatures than 20°C have been determined following Tributh and Lagaly (1986) and Schweitzer (in prep.).

Physical background of grain size separation

Stokes' law describes the behaviour of an idealized sphere in a liquid subject to the force of gravity.
The velocity of descent (v) increases until the frictional resistance is equal to the difference between the gravitation force and the force of lift; hence:

$$3\,\pi\,\eta_0\,d\,v = V\,g\,\rho - V\,g\,\rho_0 \qquad (1)$$

V = Volume of sphere, ρ = density of sphere, ρ_0 = density of liquid, g = acceleration by gravity, η_0 = viscosity of liquid (see table 1), d = diameter of sphere.

temp[°C]	$10^3\,\eta_0$ [kg m^{-1} s^{-1}]	temp [°C]	$10^3\,\eta_0$ [kg m^{-1} s^{-1}]
16	1.109	21	0.9779
17	1.081	22	0.9548
18	1.053	23	0.9325
19	1.027	24	0.9111
20	1.002	25	0.8904

Table 1: Viscosity coefficients of water in relation to different temperatures

In a centrifuge the acceleration by gravity (g) is unimportant and is replaced by the centrifugal acceleration (a_c):

$$3\,\pi\,\eta_0\,d\,v = V\,a_c\,\rho - V\,a_c\,\rho_0 \qquad (2)$$

$$3\,\pi\,\eta_0\,d\,v = V\,4\,\pi^2\left(\frac{U}{60}\right)^2 r\,(\rho-\rho_0)$$

for U = revolution per minute, r = distance between rotation axis and sphere.

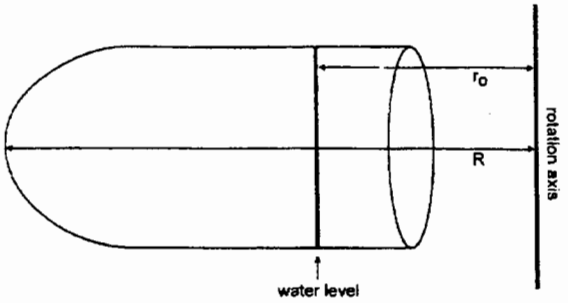

Figure 1
Standard glass tube used for centrifugation (r_0: distance between rotation axis and sphere; R: distance between rotation axis and bottom of the glass tube).

The velocity of each single grain depends on its distance from the rotation axis. Thus equation (2) is only valid for a moment, a very short time/distance interval. For our purpose the formula has to be integrated within the range through which the sphere of defined size might fall (from water surface to bottom of the glass tube ($R - r_0$), see figure 1), hence we get the centrifugation time (t):

$$t = \frac{18\eta_0 60^2}{4\pi^2(\rho-\rho_0)d^2U^2}\int_{r_0}^{R}\frac{dr}{r} \qquad (3a)$$

$$t = \frac{\eta_0}{\pi^2(\rho-\rho_0)\,d^2\,U^2}\,ln\left(\frac{R}{r_0}\right)\,1.62\;10^4 \qquad (3b)$$

There is a small error due to the acceleration and the deceleration of the centrifuge. The error is minimized, when the time of acceleration and the time of deceleration is equal.

Practical grain size separation

It is important to use the grain size <63 µm. Therefore wet sieving is suggested before using the centrifuge. Larger grains and a too large volume of grains would press the finer material to the bottom during the settling process. It is necessary to repeat the centrifugation of the sample several times.

In order to enrich the grain size fraction <11 µm settling is carried out in demineralized water under the gravitation field. The sediment is filled into a beaker (with a max. content of at least 600 ml).

Equation (1), formula of Stokes, results in the settling time (t):

$$t = \frac{18\;\eta_0\;h}{(\rho-\rho_0)\,g\,d^2} \qquad (4)$$

h = height of water in the beaker, ρ and ρ_0 = density of sphere and water, g = acceleration by gravity, η_0 = viscosity coefficient of water, d = diameter of the grain.

We work under the following conditions:

h = 0.06 m, g = 9.81 m/s^2, d = 11 * 10^{-6} m and a water and room temperature of 20°C, resulting in a viscosity coefficient of η_0 = 1.002 * 10^{-3} kg m^{-1} s^{-1}.

For sediments (ρ = 2.65 * 10^3 kg/m^3) a settling time of 9 min 13 s is necessary to optimize the grain size separation; for volcanic glass (ρ = 2.45 * 10^3 kg/m^3) a settling time of 10 min 29 s is needed due to the lower density of volcanic glass. Afterwards the suspension containing the fraction <11 µm is tipped into a beaker. This operation is repeated about five times. It is important to keep about 2 cm of the liquid in the centrifuge (beaker), because coarser material can be stirred up from the bottom. Coagulation of single grains is avoided by a large water volume.

The next operation is necessary to remove the grain size <4 µm by centrifugation (Fig. 1).

According to the conditions of our laboratory and by use of equation (3b) with R = 0.154 m, h = R-r_0 = 0.07 m, η_0 = 1.002 * 10^{-3} kg m^{-1} s^{-1} (the demineralized water has to be tempered at 20 °C), ρ = 2.65 * 10^3 kg/m^3, U = 793 rpm (for volcanic glass with ρ = 2.45 * 10^3 kg/m^3: 846 rpm), ρ_0 = 1.0 * 10^3 kg/m^3, d = 11 * 10^{-6} m we have determined the centrifugation time to t ~ 60 sec.

This results in the grain size fraction 4-11 µm being enriched at the bottom of the tube and the supernatant liquid is poured away. The operation of stirring and centrifugation is repeated until the solution is clear. The calculation of grain size behaviour is based on idealized round shaped spheres. The shape of the grains will determine

whether the grain size range is near to the expected value or not. Empirical verification of whether the suitable grain size based on the centrifugation time and velocity was enriched, has to be done under the microscope. This is even more important for the separation of glass shards, because of the varying shapes of volcanic glass fragments.
The results obtained by this technique are certainly more precise than those achieved by the use of acetone as described by Frechen (1991) and Zimmerman (1971), and the most striking goal, the separation by the centrifuge takes essentially less time than the standard procedure. A suitable centrifuge is the *Heraeus Sepatech* (Omnifuge 2.0 RS), which is used in our laboratory.

Preparation of fine grain discs
After having prepared the right grain size, 1 ml suspension of the 4-11 µm polymineral fraction (200-250 mg/120 ml acetone) is pipetted onto each aluminium disc and allowed to evaporate in a dry box at 40° C. A theoretical monolayer of grains of 4-11 µm diameter is achieved by 100 mg per 100 ml of acetone. We use about the double concentration because a part of the material settles under the discs. Batches of 90-100 discs are prepared for each sample since additive dose and regeneration methods are used for each sample. Fading experiments are carried out for an extra batch of 50 discs in order to allow five different irradiation steps.

To avoid the labour intensive cleaning of glass tubes we constructed and built an acetal (=polyoxymethylen) block containing 14 * 14 holes. The block is screwed together with a base plastic sheet. In order to make the acetal block water tight and acetone tight, different seals have been tested (see Fig. 2). The best results have been obtained by using different seals of foam rubber and india rubber which are placed between the upper and lower part of the acetal block. Aluminium foil is used to avoid acetone attacking the india rubber. The upper part consisting of acetal has not been corroded by the acetone even after 12 months.
After all the components are assembled, an aluminium disc is placed in each hole. As the discs are 10 mm in diameter, a drill width of 11 mm was chosen for each hole. Altogether 196 discs can be prepared at the same time. After unscrewing the acetal block the aluminium discs can be picked up from the aluminium foil. Later on the acetal block is cleaned by demineralized water and the aluminium foil has to be replaced. The time needed for cleaning the glass tubes is avoided and the results of the sedimentation process is excellent. For the reproducibility from disc to disc a typical range of values is 5-10% for eolian loess samples.

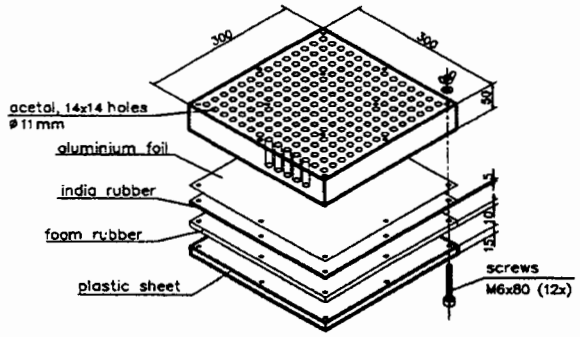

Figure 2
*Acetal (= polyoxymethylen) block containing 14*14 holes for peparation of fine grain discs.*

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Reviewer
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