

THE SEPARATION OF THE FINER CONSTITUENTS OF SEDIMENTARY ROCKS.

BY PROFESSOR P. G. H. BOSWELL.

The geologist's interest in "powders" usually lies in one or both of two directions, the "pure" and the "applied". He may be concerned with the finer sedimentary rocks and problems regarding their origin, or with the products resulting from the artificial crushing of rocks for economic purposes. In the latter direction lie such practical applications of the fine grinding of geological materials as abrasives (quartz, felspar, flint, etc.), slimes (as in ore-dressing and especially tin recovery), dust for reducing mine explosions, pottery-slips, brick- and tile-making earths, soils, fillers for asphalts in road-making, and so forth.

On the pure side, the aim of the geologist is to abolish the vague use of general terms, and to establish the description of geological materials on a quantitative basis. The "powders" of geology are the silts, muds, and clays, and also rocks such as loam, loess, and brickearth constituted largely of such materials.

An outstanding example of the looseness of the old terminology is provided in the reports of the famous "Challenger" expedition, in which sands are called muds as often as not, and the particles in deposits to which the same name is given vary enormously in size.

The application of quantitative methods to the description and analysis of sedimentary rocks being in its infancy, time exists for a certain amount of standardisation in such matters as grade-sizes, methods of mechanical analysis, and the expression of results.

Fundamentals in Elutriation.

Grade Sizes.—The soil analysts were first in the field with definite but arbitrary limits to the various grades which were separated by subsidence methods. The scheme adopted is as follows:—

1 mm. to 0·2 mm.	gravel and coarse sand
0·2 mm. to 0·04 mm.	fine sand
0·04 mm. to 0·01 mm.	silt
0·01 mm. to 0·002 mm.	clay

For various reasons, chiefly concerned with matters of their field characters, behaviour in bulk and physical properties, the above limits are not entirely satisfactory to geologists. The scheme most favoured by geologists in this country and America is as follows:—

CONSTITUENTS OF SEDIMENTARY ROCKS

35

Approx. I.M.M. Mesh.	G.	Greater than 2.0 mm. diameter				Gravel grade (G)		
12-5	VCS	"	"	1.0	"	and less than 2.0 mm.	Very coarse sand	} Sand grade (S)
20-12	CS	"	"	0.5	"	"	Coarse sand	
50-20	MS	"	"	0.25	"	"	Medium sand	
120-50	FS	"	"	0.1	"	"	Fine sand	
180-120	cs	"	"	0.05	"	"	Superfine sand or coarse silt	} Silt grade (s)
—	fs	"	"	0.01	"	"	Silt	
—	c	"	"	"	"	"	Clay or mud	grade (c)

The limit of accurate screening so far as the usual purchasable sieves are concerned is considered by the writer to be 0.5 mm., *i.e.*, about 12 mesh to the inch. This may sound somewhat heterodox when it is remembered that industrial products are supplied as "passing 180-mesh" or even 200-mesh. Anyone, however, who has examined microscopically these products or who has attempted to screen water-separated materials, will fully realise the significance of the above use of the term accurate, although for that matter no screening is ever really accurate.

But it is with the mineral particles of diameter less than 0.25 mm. that the following notes deal mainly. Below the limit of accurate screening the method adopted of separating geological materials into various grades is that of elutriation in water. Soil analysts in Britain have been in the habit of using the reverse process, that of separation by subsidence, despite the fact that elutriation as a separatory process seems to have arisen in Germany for purposes of soil analysis.¹

In the schemes of grade-sizes outlined above, the limit between fine sand and silt is undoubtedly a difficult one to fix. A diameter of 0.1 mm. has the merit of being a handy one to remember, but it is found that there are actual sediments (for example, the sands of the Inferior Oolite-Lias) which consist largely of material of diameter from 0.1 to 0.05 mm. and which behave in the field as sands. The geologist has chosen 0.05 rather than 0.04 mm. as his limit in this connection for purposes of uniformity and convenience in plotting results. The most important difference in the two classifications rests on the definition of the clay grade. It is the geologist's contention that material of diameter less than 0.01 mm. behaves as a true clay and satisfies all the criteria expected of the sediment so-named. Moreover, elutriation methods are difficult to apply to separations below 0.01 mm. on account of the low velocities in operation. Subsidence methods must be resorted to, and they are accurate only after an infinite number of decantations. Even with only a few decantations the methods are cumbrous and tedious. The question is one, however, of the limits of experimental accuracy.

Added to this is the difficulty of securing adequate deflocculation (see page 38) down to 0.002 mm. or less.

Stokes' law for determining velocities of falling spheres has been the basis of both elutriation and subsidence methods. Stokes' well-known law is as follows:—

$$v = \frac{2}{9} \cdot \frac{g \cdot a^2}{\eta} (\sigma - \rho)$$

where a is the radius and σ the density of the sphere, ρ is the density of the medium in which it falls, and v is the velocity acquired.

The form of the particles of sediments and the conditions under which they subside are usually far from the ideal postulated by Stokes in his

theoretical discussion. Nevertheless, actual experimental work on the velocities of subsidence of various mineral fragments shows, for grains between certain size-limits, a close agreement with the calculated results.

The assumptions implied in Stokes' law (which deals with perfect spheres) have been detailed by Millikan thus: (1) That inhomogeneities in the medium are small in comparison with the size of the sphere, (2) that the sphere falls as it would in a medium of unlimited extent, (3) that the sphere is smooth and rigid, (4) that there is no slipping of the medium over the surface of the sphere, and (5) that the velocity with which the sphere is moving is so small that the resistance to the motion is entirely due to the viscosity of the medium and not at all due to the inertia of such portion of the medium as is being pushed forward by the motion of the sphere through it.¹

In considering the application to sediments of Stokes' Law and the above implied assumptions, the following points should be noted.

That only in exceptional cases are the grains which undergo separation smooth spheroids. They are even more rarely spheres. Their usual form is sub-rounded to sub-angular, and they may be angular or even flaky. In the case of quartz, which constitutes over 90 per cent. of coarser detrital deposits, the grains are usually sub-angular and almost, though not quite, equidimensional. In view of this departure from sphericity it is surprising that the theoretical and experimental results are as concordant as they prove to be.

As regards the five assumptions made above:—

(1) Water is usually, and air occasionally, the medium in which separation is effected. If temperature changes giving rise to convection are avoided, lack of homogeneity in, at any rate, water is likely only to give rise to errors smaller than those of the experimental method itself.

The upper limit of applicability of Stokes' Law is defined below on page 37. Also recent work by the physicists has given us the lower limit of validity. Arnold showed that the velocity of fall of spheres of rose-metal down to 0.02 mm. could be computed from Stokes' Law with an accuracy of a few tenths of 1 per cent. But Stokes' Law begins to fail in the case of gases only when the size of the falling particles is such that the medium ceases to be homogeneous with respect to them, that is, the radius of the particle is comparable with the mean free path of the molecules of the medium. In the case of water the mean free path is a quantity of far smaller order than the diameter of the least particle we are able to separate. Despite this fact, serious discrepancies between the observed and computed velocities required for separating particles of diameter less than about 0.02 mm. are noted in the table on page 37.

(2) In both elutriation and subsidence methods the diameter of the vessel used should be very large in proportion to the diameter of the grains undergoing separation. Dr. H. A. Baker, in dealing with the inaccuracies which arise from the use of the lower narrow tube of Crook's elutriator, points out that the velocity of the current in the centre of the narrow tube is nearly twice the average velocity as calculated from the outflow of the jet.² He arrives at this conclusion from the formula for the steady irrotational type of flow

$$v = \frac{2Q}{\pi R^4}(R^2 - r^2)$$

¹ R. A. Millikan, *The Electron*, Chicago, 1917, p. 93.

² H. A. Baker, *Geol. Mag.*, Vol. LVII., 1920, p. 321.

FINER CONSTITUENTS OF SEDIMENTARY ROCKS 37

where v is the velocity at a point distant r from the axis of the tube, Q is the volume passing a cross-section of the tube in unit time (that is, outflowing from the jet in unit time) and R is the radius of the tube.

In the centre of the tube, r is zero, consequently $v = \frac{2Q}{\pi R^2}$, and since $\frac{Q}{\pi R^2}$ is the average velocity of the current, as calculated from the outflow, the velocity of the current in the centre of the tube is twice the average.

The retarding effect of the tube-wall upon the current is thus considerable, but since the ratio of the circumference of the tube to its cross-section ($2\pi R/\pi R^2$) varies inversely as the radius, it is obviously advantageous to increase the latter as far as practicable. Moreover, Osborne Reynolds has stated that the critical mean velocity at which the steady type of flow breaks up and is replaced by eddying flow varies inversely as the diameter of the tube.

Considering the problem next from the point of view of the falling grain in still water, rather than that of the ascending current of water which holds it in suspension, we find that Ladenberg has worked out a modification of Stokes' Law for particles descending in a cylinder of circular cross-section, radius R and length L , thus

$$v = \frac{2}{9} \cdot \frac{g \cdot a^2(\sigma - \rho)}{\eta \left(1 + 2.4 \cdot \frac{a}{R}\right) \left(1 + 3.1 \cdot \frac{a}{L}\right)}$$

In elutriation, the ratio a/L is usually a small one, but the ratio a/R must be taken into account in the separation of the coarser grades. In the case of the Crook elutriator the lower tube, which retains grains from 0.1 to 1 mm. in diameter, is about 15 mm. in diameter. For accurate separation of 0.1 mm. particles, the velocity is therefore approximately 0.95 and for 1.0 mm. particles is only 0.8 of that given by Stokes' Law.

The upper limiting size for separation by elutriation is taken by the writer at 0.25 mm. diameter. For a 1 per cent. error or less, the diameter of the separating vessel would require to be 20 cm. if the velocity calculated from Stokes' Law were adopted. The diameter of the vessel actually in use for this grade is generally 5 or 6 cm. Thus the velocity as determined by outflow must either be calculated from Ladenberg's equation, or determined from actual experiments based on counting and weighing the grains separated (see page 39).

Actual figures obtained by the latter (experimental) method are given in the following table, together with the final velocities as calculated from Stokes' Law:—

Size of Grain. Diameter in mm.	Velocity Determined by Experiment (Elutriation) in mm. per sec.	Velocity Involved in Assumptions in Soil Subsidence Methods in mm. per sec.	Velocities Calculated from Stokes' Law in mm. per sec.
0.4	47	—	126.8
0.3	32	—	71.3
0.25	25	—	49.5
0.2	20	separated by screening	31.7
0.1	6.7	—	7.9
0.05	1.78	—	1.98
0.04	1.14	1.0	1.27
0.01	0.12	0.1	.08
0.005	0.087	—	.02
0.002	?	0.01	.003

Temperature 15° C.
Viscosity (η) of water at 15° C. = 0.1142.

Density of water at $15^{\circ}\text{C.} = 0.99872$, but is taken as unity since the assumed density of the material separated is taken as that of quartz, 2.65, and the error is small compared with that introduced by the proximity of the tube walls.

It is evident from the table that the Law of Viscous Resistance ($v = k \cdot a^2$) as expressed by Stokes' Law does not hold for grains of diameter greater than 0.02 mm. In fact, the velocities then more nearly correspond to those given by Newton's Law of Eddying Resistance ($v = k \sqrt{a}$) although the agreement is not close. For grains of diameter from 0.1 to about 0.02 mm. there is a fair agreement with Stokes' Law, but for those of diameter less than 0.02 mm. there are serious and at present unexplained departures from it.

Reverting to the implied assumptions (p. 36):—

(3) Mineral substances are usually rigid but are occasionally far from smooth. Quartz grains are usually smooth when cleaned free from adherent iron oxides and clay.

(4) The assumption that there is no slipping of the medium over the surface of the sphere seems to be justified by Millikan's work.

(5) Millikan and Arnold have shown that with slow motion the error due to the inertia of the medium is minutely small.

Procedure in Elutriation.

Before mechanical analysis by elutriation or subsidence is proceeded with, it is necessary to ensure that the material undergoing separation shall be in the condition of individual particles, that is, be thoroughly deflocculated. Most clayey sediments have, in the course of their formation been thrown down as flocculated mud by the action of the dissolved salts in sea—or river—water. These aggregates must be broken up, and among the deflocculants most commonly used are sodium carbonate, liquid ammonia, sodium silicate, and soap. Of these the most effective seem to be sodium silicate and ammonia. The concentration of these electrolytes adopted usually is about 0.05 per cent. It is probable that as regards different kinds of clays the various substances are selective in their action.

If the sample be simply a crushed mineral or a clay consisting largely of fine crystalline particles such as kaolinite and micas, only simple boiling is necessary for deflocculation.

Division into separate particles having as far as possible been effected, the sample is washed into some form of elutriator. Here again it is desirable that the method and apparatus should conform to standard. The types of elutriators used in France, Germany, America, and the United Kingdom are varied. As an outcome of experience with most types and with full realisation of the impossibility of comparing results obtained in different types of apparatus, the writer has returned to the simplest form of all, what may be called the "single-vessel" type of elutriator, a modification of one of the earliest forms of the apparatus. Brief descriptions of this and other forms of apparatus have already been published,¹ with comparative results obtained by using different types of elutriators.²

A few notes on the limits of accuracy and possible experimental difficulties may be of service to other workers.

¹ P. G. H. Boswell, "A Memoir on British Resources of Sands and Rocks used in Glass-making," 2nd edn., 1918. T. Crook, *Econ. Proc. Roy. Dublin Soc.*, Vol. 1., Part V., 1904, p. 367; Appendix to Hatch and Rastall, "Sedimentary Rocks," 1913. E. Schoene, *Ueber Schlämmanalyse*, Berlin, 1876. H. Ries, "Clays, their Occurrences, Properties and Uses," New York, 1914, p. 138.

² H. A. Baker, *op. cit.*, p. 321.

FINER CONSTITUENTS OF SEDIMENTARY ROCKS 39

In the separation of the finer grades the only method of checking the accuracy of the process, so far as size is concerned, is by direct measurement under the microscope. Such a measurement is, however, always greater than the average diameter because the grains lie in their flattest position.

The accuracy of the separation of the various sand grades may be tested by counting out many thousands of sand grains of quartz so separated, weighing the crop, and calculating the average diameter, the density being determined or taken for example as 2.65.

The mechanical analysis of sediments rests on the assumption that all the material has the same density. If there are grains of different minerals present, the accuracy of the separation depends upon their size. If they are of diameter greater than 0.05 mm., the separation is inaccurate. As, however, the particles become smaller and smaller, it is clear from Stokes' Law that the surface area plays an increasingly greater part in determining their final velocity. Hence although the density of clay-substance is less than that of quartz, the error due to elutriation on the assumption of an average density of 2.65 is less than those inherent in the experimental method. If it is desired to make an accurate mechanical analysis of a coarse sandy sediment containing a larger proportion than 1 per cent. of different heavy minerals, separation into crops of various densities by means of heavy liquids or otherwise must be adopted.

The experimental condition which is of utmost importance in elutriation is that of maintaining a constant temperature. The viscosity of water decreases rapidly with small increase of temperature, and the density also becomes less. The final velocities corresponding to particular diameters have been calculated or observed at 15° C., a temperature which can be maintained for days in most laboratories without difficulty. If the temperature of the water varies, one of two courses may be adopted. Either separation may be made at the observed temperatures and a correction applied, or between the inflow and the elutriator may be inserted a coil of tubing immersed in a bath of water heated or cooled so that elutriation proceeds at 15° C. Unless some method of this kind is adopted, the difference in the summer and winter temperatures in some laboratories may introduce a large error.

Strictly speaking, tap water should not be used in elutriation on account of the flocculating power of its dissolved salts. If distilled water is used, there are obvious laboratory difficulties when the total outflow of a series of elutriators amounts to 100 c.c. or more per second (as in the writer's laboratories). For clays alone, the consumption of water is very much smaller and it becomes practicable therefore to use distilled water, as Dr. J. W. Mellor has done. The error involved depends on the proportion and character of the salts present in the particular tap-water used. Elutriation in districts with hard waters may be more troublesome.

The degree of aeration of the tap-water in use sometimes upsets elutriation work. Bubbles of air collect in the connection tubing, in the elutriator vessel or on the grains, and by their coalescence cause a large bubble to rise through the vessel carrying over with it coarser grains than the water current is arranged to do. Not only is the separation spoiled but the outflow jet is often blocked by a grain, thus causing much trouble and annoyance. The simplest remedy for excessive aeration is to insert some vessel such as a 3-necked Wolff's bottle between the tap and the elutriator so that most of the air bubbles are trapped before entering the latter, and the air as it accumulates can be tapped off.

Further trouble may be caused by bacterial or algal growth in the tubes, when the strands choke the passage and reduce the velocity. Frequent cleaning is then the only remedy, for if the strands are disrupted and enter the elutriator tube, they block the outflow jet.

If a Wolff's bottle is put into the "circuit," or tubes are shortened or lengthened, or rubber replaced by glass, all velocities must be checked anew, for a variation in flow due to the difference in friction is the inevitable result:

Graphical Expression of Results.

Finally, it is desirable that the method of representation of the results of mechanical analyses should be agreed upon. Both for the proper appreciation of the results and for the purpose of extending the investigation, graphical methods are desirable. The form adopted by the writer is similar to that used by those interested in ore-separation. It is a curve method in which cumulative percentages by weight of material above certain grade sizes are plotted at ordinates, and the logarithms of the grade sizes as abscissæ.¹ The large grade sizes are marked off in decreasing values from the origin, their logarithms being taken to keep the graph within the compass of the page.

Somewhat similar curves were independently plotted by the officers of the United States Geological Survey in connection with coarse deposits for water filtration, etc.,² and by the officers of the Danish Geological Survey³ to express the composition of moulding sands. The Danish curves, however, were plotted on the basis of marking off the finest grades nearest the origin, and the cumulative percentages less than each respective grade size were plotted as ordinates. Thus the curves were of the same form as those of the writer, but were differently situated with regard to the axis of reference.

A notable advance was made by Dr. H. A. Baker⁴ who evolved a similar graphical method of expression by plotting as abscissæ the cumulative percentage weights above each grade size, against grade sizes as ordinates, beginning with an assumed zero diameter at the origin. By this means he has been able to measure the areas bounded by curve, abscissa, and ordinate, and calculate the "equivalent grade" (area below curve line/length of base line). Such a figure yields a representative average of the diameters of the particles, and increases with increasing coarseness of the sediment. The perfection, or otherwise, of the grading is expressed by the "grading factor," which is a measure of the departure of the curve from the equivalent grade.

The grading factor (expressed as a fraction) is high for well-graded samples and increases to unity for sediments with all the grains of the same size.

Thus each sediment may be expressed by two figures, the equivalent grade and the grading factor. But for some reasons the expression of the mechanical analysis of a sediment by a single figure would be advantageous. No satisfactory method of plotting the composition of a sediment and computing such a figure (which should be unique for each different sediment) has yet been evolved.

¹ P. G. H. Boswell, *op. cit.*, p. 28.

² C. S. Schlichter, "Motions of Underground Waters," Water Supply Paper 67, U.S. Geol. Survey, 1902.

³ *Dan. Geol. Undersøg.*, Ser. ii., No. 16, 1905.

⁴ H. A. Baker, *op. cit.*, p. 367.

FINER CONSTITUENTS OF SEDIMENTARY ROCKS 41

Dr. Sven Odén in 1916 described an entirely different and novel method of plotting the composition of deep-sea deposits.¹ He first obtained a curve called the "accumulation curve" by plotting the time of accumulation of weights of the deposit as it subsided in water against the percentage weights. From the accumulation curve, by mathematical analysis and transformation, he constructed a "distribution curve" showing how the amount of particles of a certain size varied with that size. The method was ingenious, but the labour of the mathematical operations involved will, it is feared, prevent its wide application in geology. Perhaps the time is early for a general agreement upon a standard method of graphical representation of sediments.

¹Sven Odén, "On the Size of the Particles in Deep-Sea Deposits," *Proc. Roy. Soc., Edinburgh*, Vol. XXXVI., Part. III., No. 13, 1916.