

A further comparison may be made by tabulating the averages from the various data made use of in the above computations.

TABLE VI.

	H From Curve	H From Ult. Anal.	H From Ind. Calories.
Average of first 20 (Table No. 2)	3.39	3.28	3.42
Average of 50 from Bulletin No. 261, U. S. Geol. Survey	3.51	3.46	3.69
Average of 50 from report of Lord and Haas	3.96	3.95	3.90
Average of 12 from Mich. Geol. Survey	4.09	4.06	4.16
Average of first 20, as in Table No. 4 . .	3.30	3.17	3.29
Average of 70 from Bulletin No. 290. U. S. Geol. Survey	3.54	3.41	3.61

It will be seen from the above that out of 180 coals the extreme of error by use of the curve is much within that resulting from the ultimate analysis; and that on the average the error represented by the former is less than that resulting from use of Dulong's formula. There is here opened up the possibility of making the essential factors as derived by ultimate analysis equally accessible with those of the proximate method, the real value of chemical work on coal being greatly enhanced thereby. Further interest in this connection would center in the derivation of the factor for total and fixed carbon, a discussion of which must be reserved for another time.

UNIVERSITY OF ILLINOIS, URBANA, ILL.

[CONTRIBUTION FROM THE OFFICE OF PUBLIC ROADS, U. S. DEPARTMENT OF AGRICULTURE¹.]

AIR ELUTRIATION OF FINE POWDERS.

BY ALLERTON S. CUSHMAN AND PREVOST HUBBARD.

Received February 4, 1907.

In recent years the production of extremely fine ground material for use in the various arts has made it highly desirable to obtain a standard method for separating and measuring particles which will readily pass through the finest meshed sieves. All the methods heretofore used depend upon the elutriation of the material with water flowing at different velocities, each velocity allowing the sedimentation of particles within the range of certain definite dimensions. Various forms of apparatus have been constructed on these principles, among which the best known are those of Schoene² and Hilgard³. Recently an improved form has been described by Binns⁴. In all the methods of water elutriation, however, it

¹ Published by permission of the Secretary of Agriculture.

² Bulletin de la société impériale des naturalistes de Moscou (1867), Part I, 324.

³ Bulletin 38, Bureau of Chemistry, Department of Agriculture, 60.

⁴ Trans. Am. Ceram. Soc., 8, 244 (1906).

is impossible to avoid the loss of the very finest portions of the material, which remain indefinitely in suspension. Krehbiel¹, working with the Binns form of apparatus, found that in samples of normal flint and spar as obtained from the miller, over 50 per cent. of the material was composed of particles under 0.01 mm. in diameter. These are inevitably lost in the final overflow from the apparatus. For the separation of comparatively coarse material from very fine, the water methods, however, undoubtedly possess certain advantages, provided that no change or decomposition of the particles takes place under the action of the water, and the coarser particles can be sized with considerable quantitative accuracy.

A number of objections to the elutriation of extremely fine particles with water have already been advanced by one of us². A brief summary of the difficulties which would be overcome by a laboratory method of air separation is as follows:

- (1) Decantation and evaporation of large quantities of water.
- (2) Varying settling effects produced by the presence of small quantities of electrolytes in the water used.
- (3) Difficulty of filtering fine particles.
- (4) The necessity of drying residues before weighing.
- (5) The decomposing action of the water on the surfaces of fine particles, affecting their flotation.
- (6) The loss of the finest particles.

In our investigations on the decomposition of various rock powders it has been found necessary to devise some means of separating and sizing the extremely fine particles without allowing them to come in contact with water, owing to the reactions which at once take place the moment the particles become wet.

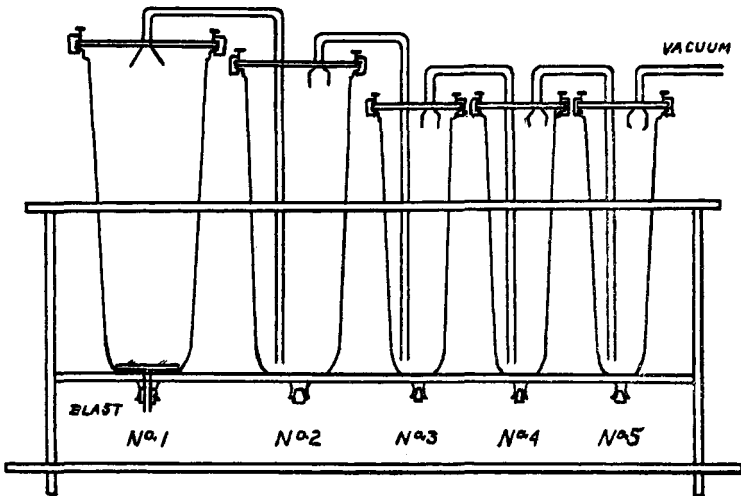
The relation between size of particle and rate of decomposition below the limits of the finest available sieves, *viz.*, 200 meshes to the linear inch has received very little attention. A powder which has passed a 200-mesh sieve contains particles which vary in size from almost 0.1 mm. down to those of an ultimate degree of fineness to which with our present knowledge of the subject, it would be difficult to put a definite limit. The coarsest particles passing the standard 200-mesh sieve have an approximate size of 0.065 mm. in diameter, while the finest which it is possible to measure with the micrometer attachment of a powerful microscope are of the order of 0.0001 mm. It is not generally realized that if we treat this ratio mathematically the finest measurable particles of the 200-mesh powder bear the same relation to the coarsest particles as these latter do to fragments 42.2 mm., or nearly 2 inches in diameter. The decompositions which take place under the action of water depend upon the surface area

¹Trans. Am. Ceram. Soc., 6, 173 (1904).

²Ibid., 8, 249 (1906).

of a powder, and it will be shown later on that in a unit weight of two powders in which the diameters of the particles vary, the surface area also varies enormously. It is a matter of great importance, not only from a scientific point of view, but also to the various industries which have to deal with finely comminuted powders, that the influence of size of particle beyond the mechanical limits of sieve separation, should be studied. In the cement and fertilizer industries, in which the value of the material is particularly affected by the decompositions which take place when water acts upon it, water elutriation methods are naturally out of the question.

For the purpose of our own investigations a pneumatic separator has been designed which has worked so satisfactorily that it is probable that, with slight modifications it will be found of general value. Our aim has been simply to secure sufficient amounts of different sized powders to carry on a study of the influence of size of particle on rate of decomposition. No attention has been paid to complete quantitative separations, but from the construction of our apparatus and from its general arrangement it is evident that by properly sizing the different chambers and regulating the air pressure such a separation could be readily made. The apparatus consists of five percolating jars set in a wooden frame and connected by tubes of glass passing through close-fitting caps tightly clamped to the jar tops, as shown in the figure.



AIR ELUTRIATOR

The first jar is of 3 gallons capacity, the second 2 gallons, and the third, fourth and fifth each 1 gallon. In the bottom of No. 1 is placed a flat spiral tube closed at one end but with a number of very small open-

ings through small jets soldered into the upper surface of the spiral at an angle of about 30° . The open end of the spiral passes through a tightly-fitting rubber stopper inserted in the neck of the jar and is connected to the source of air supply. An inverted funnel tube whose stem passes through another rubber stopper fitted in the cap at the top of the jar is connected to a glass tube which passes in a similar manner nearly to the bottom of No. 2. An inverted thistle tube connects No. 2 with No. 3 in like manner, and so on through Nos. 4 and 5, the exit tube of No. 5 being connected to a vacuum. It is possible to tie pieces of fine linen lawn over the mouths of the inverted thistle tubes, but in our own work this has not been done except in the final exit in No. 5. Rubber stoppers close the necks of the jars and are removed only when it is desired to draw off the charges of powder which have accumulated during a run. A charge of oven-dried powder not exceeding 1 kg. is placed in jar No. 1. Blast and vacuum are then turned on and adjusted so that a steady stream of air passes through the powder with sufficient force to raise a dense white cloud which assumes a vortex motion as it ascends, owing to the arrangement of the air jets. The heavier particles continually fall in a ring near the walls of the vessel where they build up until caved in by the air jets, while the lighter particles are carried into No. 2 through the funnel tube. Here the heavier portions are retained while the lighter pass into No. 3, and so on, the cloud in each succeeding jar becoming less dense. If properly adjusted, there is but little loss in No. 5, although the powder here is so fine that no trace of grit can be noticed when it is placed between the teeth. In fact the material (in our case orthoclase feldspar) seems almost to dissolve in the mouth. In our own work the blast of air is supplied from a Westinghouse compressor, and is dried by passing through four large towers containing lumps of pumice soaked with concentrated sulphuric acid.

A sample of ground feldspar, as obtained in bulk from the miller, was used and a battery consisting of four jars. Three grades of powder were obtained. Small samples of each were mounted in glycerol and examined under the microscope. Considerable difference in the relative sizes of the grains of the different samples was at once evident, and measurements were made in the following manner: The particles constituting each sample were arbitrarily classified as large, medium and small. The average diameter of each class was then determined by a series of measurements made with a screw micrometer and the relative proportions of each class estimated by means of a grating in the microscope, similar to that employed for counting microorganisms in bacteriological work. The results were as follows:

TABLE I.

Relative size.	Grade No. 1		Grade No. 2		Grade No. 3	
	Diameter mm.	Proportion.	Diameter mm.	Proportion.	Diameter mm.	Proportion.
Large	0.0122	8	0.0063	3	0.0037	3
Medium	0.0061	15	0.0025	10	0.0007	14
Small	0.0009	77	0.0002	87	0.0001	83
		100		100		100

Although the method is not exact, it was carried out as accurately as the conditions under which we were working permitted. It was approximately determined that in the case of particles ranging from 0.0122 mm. to 0.0009 mm. diameter, nearly 75 per cent. were of the latter size. Over 87 per cent. of the particles ranging from 0.0009 mm. to 0.0002 mm. diameter were 0.0002 mm. diameter, and over 83 per cent. of the particles from 0.0002 mm. to 0.0001 mm. were 0.0001 mm. diameter. These figures show that it is manifestly incorrect to adopt a mean diameter of 0.005 mm. for particles ranging from 0.01 mm. to those of submicroscopic size. We shall see a little later, when discussing the relations of surface area to diameter, what an important bearing this will have upon the total surface factor of a powder, especially when 50 per cent. or more consists of grains smaller than 0.01 mm. diameter, as has been previously noted.

Comparing the three samples according to the diameters of their largest and smallest particles we find that to some extent the figures overlap, but when we consider that ground spar as obtained in bulk contains a certain amount of quartz and a little mica, both having slightly different specific gravities from that of feldspar, it is evident that all methods of elutriation will present the same difficulty. With any method of elutriation, in order to obtain strictly uniform results the velocity of the carrying medium should be adjusted to the specific gravity of the material, unless, as in our case, microscopic measurements of the different sizes are made. As this would be too tedious and complicated a method for general use, however, and as the materials ordinarily compared, such as quartz and feldspar, are of nearly the same specific gravity, this error may be overlooked.

Assuming that all of the particles are spheres or cubes it follows, as has been pointed out by Heath¹ and again by Jackson², that the areas presented by unit volumes broken down into particles of smaller but uniform diameters are in inverse ratio to their diameters. Thus if a unit volume is broken down into particles all having $\frac{1}{10}$ of the unit diameter, and another unit volume is broken down into particles all having $\frac{1}{100}$ of the unit diameter, their respective areas will be in the ratio of 1 : 10 : 100.

Now if the unit volume is broken down into particles having different diameters it will be found that the law does not hold when an average

¹Article read at Victoria Institute, Tunstall, December 13, 1902.

²Trans. English Ceram. Assoc., 3, 16.

diameter is taken. Thus if a unit volume is broken down into particles of $1/10$ and $1/100$ the unit diameter, the proportions of the two sizes being respectively as 1:2, the average diameter would be $4/100$ and the ratio of the surface of the broken unit to that of the unit, calculated on the average diameter, would be as the reciprocal of these diameters, or as 25:1.

That this ratio is in error may be shown as follows: As $1/3$ of the unit is broken into particles of $1/10$ the unit diameter, it is plain that the area presented by this fraction must be one-third of that presented by the whole unit broken to particles of the same size. This would give it an area of $10/3$. In like manner the area of two-thirds of the unit broken into particles $1/100$ of its diameter would be $200/3$. The total area then of the unit volume broken to the two sizes in the given proportions would be $10/3 + 200/3 = 210/3 = 70$, and the ratio of this area to that of the unit volume would be as 70:1, which is much larger than that calculated from the average diameter.

The great necessity of obtaining more knowledge as to the size of the finest particles, therefore, becomes at once evident when we attempt to compare different powders by their relative surface areas. If we consider a cube of unit dimensions to be crushed into smaller cubes of uniform dimensions, the total area of these cubes would be represented by the following formula, where a = area sought and l = length of edge of the component cubes:

$$(1) \quad a = 6 \times \frac{1}{l} = \frac{6}{l}$$

Or, if we wish to compute the area of any given weight of material broken down in the same manner, we can do so by multiplying this formula by $\frac{W}{S}$ where W = weight and S = specific gravity. This would give

$$(2) \quad a = \frac{6W}{l \times S}$$

Neglecting the relative proportions of the different sizes constituting each of our three samples and taking their average diameter to five decimal places expressed in centimeters, the area presented by an equivalent of one cubic centimeter of the original material would be calculated by formula (1) as follows:

TABLE II.

Sample No.	Mean diameter cm.	Extreme diameter cm.	Average diameter cm.	Formula.	Surface presented by 1 c.c. original material, sq. cm.
1	0.00122	0.00009	0.00066	$\frac{6}{0.00066}$	9091
2	0.00063	0.00002	0.00033	$\frac{6}{0.00033}$	18182
3	0.00037	0.00001	0.00019	$\frac{6}{0.00019}$	31579

The true surface areas would be represented more nearly, however, if we considered the particles of different diameters and their relative proportions, and for each sample could be calculated as follows where M_1, M_2, M_3 = the per cent. of the large, medium and small size particles, and l_1, l_2, l_3 = their respective diameters:

$$a = + \frac{6M}{l_1} + \frac{6M_2}{l_2} + \frac{6M_3}{l_3} = 6 \left[\frac{M}{l_1} + \frac{M_2}{l_2} + \frac{M_3}{l_3} \right]$$

Applying this formula to the three samples we obtain the following areas:

No. 1, $a = 6 \left(\frac{0.08}{0.00122} + \frac{0.15}{0.00061} + \frac{0.77}{0.00009} \right) = 53,202$ sq. cm.
 No. 2, $a = 6 \left(\frac{0.03}{0.00063} + \frac{0.10}{0.00025} + \frac{0.87}{0.00002} \right) = 263,688$ sq. cm.
 No. 3, $a = 6 \left(\frac{0.03}{0.00037} + \frac{0.14}{0.00007} + \frac{0.83}{0.00001} \right) = 510,486$ sq. cm.

The difference in these methods is certainly very strikingly illustrated when we compare the two sets of figures.

As the calculation of the actual areas presented by definite weights of material ground or sifted to different degrees of fineness is difficult and tedious, the use of a surface factor has been proposed. Jackson's¹ factor modified by Purdy² is very generally used in the ceramic industry. This factor is based upon the assumption that the surface areas of two or more powders derived from a unit volume are in inverse ratio to the average diameter of their grains. The reciprocal of the average diameter is therefore taken as the factor. Although this assumption is in error, as has been shown, the factor is a convenient approximation and has been found of considerable practical value for comparing material ground or elutriated to different degrees of fineness. The Purdy factor is obtained in the following manner. Suppose by a method of water elutriation a powder has been separated into four sizes as follows: No. 1, having diameters 0.12 to 0.04 mm.; No. 2, 0.04 to 0.025 mm.; No. 3, 0.025 to 0.01 mm.; No. 4, 0.01 to 0.00. Taking the average diameters, the surface factor of each group is as follows:

TABLE III.

Group.	Average diameter.	Surface factor		Per cent. present.
1	0.0800	$\frac{100}{8}$	= 12.50	10
2	0.0325	$\frac{10000}{325}$	= 30.77	20
3	0.0175	$\frac{10000}{175}$	= 57.14	20
4	0.0050	$\frac{10000}{50}$	= 200.00	50

¹Trans. English Ceram. Assoc., 3, 16.

²Trans. Am. Ceram. Soc., 7, Part III., 441.

The total surface factor is now obtained by multiplying the surface factor of each size by the percentage weight of each group, and finding the sum of the products thus obtained. Supposing the percentage amounts are as indicated in the last column, the total surface factor would then be

$$\begin{array}{r}
 12.50 \times .10 = 1.25 \\
 30.77 \times .20 = 6.15 \\
 57.14 \times .20 = 11.43 \\
 200.00 \times .50 = 100.00 \\
 \hline
 118.83 \text{ or } 119
 \end{array}$$

The decimal points as used by Purdy seem unnecessary in such an approximation and the factors are improved by stating them as the nearest whole number. If the total surface factor had been calculated as it is for each of the groups, *i. e.*, on the basis of the average diameter, it would be as follows:

$$\begin{array}{r}
 0.0800 \times 0.10 = 0.0080 \\
 0.0325 \times 0.20 = 0.0065 \\
 0.0175 \times 0.20 = 0.0035 \\
 0.0050 \times 0.50 = 0.0025 \\
 \hline
 0.0205 = \frac{205}{10000} \\
 \text{Reciprocal} = 48.8 \text{ or } 49.
 \end{array}$$

The difference between these two factors 119 and 49 is certainly very marked, but it is not so large as it would be if we neglected the percentage amounts and took the average diameter 0.12 to 0.01 mm. = 0.065 mm., which would give us a surface factor of about 15. And yet this is what is commonly done with the usual 50 per cent. of the material ranging from 0.01 to 0.00 mm. diameter. As the difference between the true surface factor and that calculated from the average diameter increases as the diameter decreases, the immense importance of knowing more about this finest material, if we wish to obtain a close approximation of the total surface factor, needs no further comment. While it seems unavoidable to take averages of the last groups into which we divide our material, it would certainly be an improvement if we could further subdivide our groups and especially the finest which, when constituting a large proportion of the original material, exerts so great an influence upon the surface factor.

It is for this reason that the air elutriator is suggested as a means of sizing the extremely fine particles, and while the limitations of the method are apparent, there seems to be at present no other way of accomplishing the work which it is capable of doing.

In discussing Jackson's formula, Purdy has shown that in the development of the surface factor, specific gravity may be neglected and the factor based upon volume rather than weight. As, however, in general practice it is customary to state quantities of solids in units of weight rather than in units of volume, it would seem better and more logical to compare total surface factors on the basis of weight, especially when the materials compared differ in specific gravities. This factor would then be the volume factor divided by the specific gravity, and for a material composed of

particles of uniform size would be $\frac{1}{D \times \text{Sp. gr.}}$ where D = the diameter of

the particles. The total surface factors of any two materials would then express the relative surface areas presented by any unit weight of the substance. It might be urged that this factor does not represent the fineness of the material, but it is evident that the same is true of the volume factor, for the fineness of the material would be represented by the average diameter of its particles.

A method of dry air elutriation offers an interesting field for study in connection with Portland cement and other hydraulic cements, in which the impossibility of using water elutriation has prevented any large scale investigations as to the influence of a variable preponderance of very fine particles. In ceramic investigations air elutriation should be of great value, as it provides an easy method of obtaining information as to the effect of the very finest powders in body and glaze mixtures. In the analysis of dry fertilizers the influence of the fineness of grain on the yield of available plant foods is a most important consideration worthy of more study than it has heretofore received.

The method for the preparation of the sample as adopted by the Association of Official Agricultural Chemists, directs that the sample should be well intermixed, finely ground, and passed through a sieve having perforations 1 mm. in diameter. A sample having 90 per cent. of its particles exceeding 0.9 mm. in diameter would conform to this direction just as well as one in which 90 per cent. were below 0.0009 mm. in diameter. As has been shown above, the difference in surface area of unit weights of these two samples, and the consequent variation in the determined availability of the plant foods, is very large.

The study of the effect of fineness of grain on the rate of decomposition of rock powders enters into a number of the problems which are being studied by the authors, and these investigations will be carried further.