

Micronization

The Analysis and Standardization of Micron Materials

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This article focuses on the methodologies of fine particle grading or as known in the industry as Micronization. The topics covered include sedimentation, elutriation and centrifugation.

While each of these topics is an extension of the methods used in size separation, the purpose is not to teach or to become elutriation operators, but to obtain a better understanding of the science behind exact sizing techniques using the hydraulic principles.

The article outlines the standardization of finely graded materials describing one methodology borrowed from the automotive industry, and will describe the different types of modern analyzers that are considered industry standards by their wide use and application in the area of fine particle measurement.

The validation of these pieces of equipment is through standard statistical techniques that are easier to understand in the context of the end result of micron and sub-micron measurements and not the means of obtaining these measurements.

Elutriation

Elutriation is the process of grading micron and sub-micron sizes (usually less than 60 microns) into equal distributions that are normally distributed. Normally distributed meaning that there is a mean, mode and median to the number of particles in the population of material being graded. The population of particles will follow the gaussian form of $f(x) = 1/s * \sqrt{2\pi} \exp[-(X-\mu)^2/2s^2]$. Since the population follows a normal distribution the control (or standardization) of micron material lends itself very well to Statistical Process Control. This will be discussed later in this paper.

Decantation

Simple methodology using Stokes principles that, Particles falling under the influence of gravity will be acted upon by drag. Constant velocity will be reached when these two are in equilibrium. $\{\mu \text{ ft./sec.} = 2gmp(pp - p)/pprArC\}$.

Where g = acceleration due to gravity, mp = mass of the particle, pp = density of the particle, Ap = apparent density, C = drag coefficient.

So by varying the material (density) or the mass of the particle we can calculate the time that a certain fraction of the distribution of particles will fall through a medium and extract that distribution by simple decantation. Micronizers are still employing this method of decantation, although mostly in the Far East. Automation of this methodology is with the use of mixers and computerized drawtubes. The machines are known as auto graders.

For proper Elutriation it is important to have the following in control:

- Complete dispersion of the material in the fluid.
- Constant fluid velocities.
- Short treatment times for a given weight of material to be separated.
- Sharp separation as measured by a minimum overlap between grades.
- Production of grades that are standardized with customers requirements.
- Minimum attrition of the material being graded.

Dispersion

It is of the utmost importance in micron and especially sub-micron separation that the population of particles be dispersed. If not properly dispersed fine particles will adhere to other particles forming agglomerates, or they will adhere to larger particles thus providing a false distribution or loss of yield.

In this regard the use of dispersants are invaluable. The question here is how much dispersant to use? If we add too much chemical dispersant to the system we can reverse the effect creating a flocculated colloid state of fine particles. The use of Zeta Potential measurements greatly aid in the minimum use of chemicals in the elutriation process. *Zeta Potential-Methodology is for determining electrical activity at the liquid/solid interface between the stern layer and shear layer.* This interface is important and is depicted.

Some of the popular dispersing agents are saponin, sodium hydroxide, agar-agar, ammonia, tannic acid, etc. The choice of dispersant is dependant on zeta potential and the amount of ionic surface activity that will remain on the particle surface after final processing.

Constant fluid velocities are necessary to avoid fluctuations in the desired cut of material and to minimize the overlap between sizes. Even a very small increase in velocity will cause an errant coarse particle to become entrained in the velocity jet and become an unwanted particle in the overall distribution. The use of constant pressure systems with multiple vessels minimizes the effect of varying fluid velocities. The control of these systems is generally through capillary flow meters. The use of nozzles or spreaders at the base of the cutting vessels also helps to mitigate parabolic velocity fronts that can occur in the center of the cutting vessel. This is particularly true in single point elutriation systems.

Attrition is generally non-existent in water-based systems even for very friable materials as proteins and some clays. In air elutriation systems because of the mechanical action of high-speed rotors, particle impingement on rotor blades could cause attrition to very friable materials.

Quality of the Separation this is a function of elutriation time, end point constancy of fluid flow, completeness of dispersion, and control of the velocity front in the separator.

Elutriation rates should be constant with sufficient time to yield sharp distributions. Frequent samples should be taken to ensure that minor adjustments are made to keep the system in balance. Because of the stochastic nature of elutriation the control and subsequent quality of the product lends it self to Statistical Process Controls.

We will not detail the methods of devising \bar{X} and \bar{R} charts other than to say most production control books on this subject will provide this information. It is sufficient to say that the use of SPC techniques will allow us to determine the quality of the separation using a limited number of measurements. The methodology usually employed to deal with random fluctuations is as follows. (Slide 2)

The null hypothesis is used and followed for control of the particle size distribution.

> If one point is out of UCL / LCL, it is noted and nothing is done.

> If one data point per shift is out, for four consecutive shifts (all charts) a process change is made.

> If one data point per shift is out, for four consecutive shifts (one chart) a population change calculation is made.

• *Type 1 error (Deciding from sampled data that an undesired change has occurred when it has not.)*

• *Type 2 error (Deciding from sampled data that no change has occurred when there has.)*

> After process changes are made, the measuring system, as outlined, is resumed.

Types of Elutriators

Schonene Apparatus — Rising current methodology, this system is constructed using varied funneled sections followed by long straight sections. A number of funneled sections are followed by straight section forming chambers that could be sealed off by stopcocks. The current velocities are measured by piezometric analysis from the overflow section of each chamber. After the appropriate velocity calculations are made for a given feed, a graded size could be collected while the system is in operation. This was aided by knowing the average rising velocities that were given by the piezometer.

Mt. Morgan Mines Multi-tube — Other than in single point elutriation this is probably the most widely used type of elutriation vessel system. They permit optimal charging, particle spacing and as close to continuous flow processing as possible. The Mt. Morgan Mine and variations of this type of system have been used successfully for generations. The heart of the system is that with the use of proper dispersants and with the proper dilution vessel very sharp distributions (as evidenced by small standard distributions and small spans) and large amounts

of material can be processed. In large-scale production this type of system become the one of choice because of near continuous flow shop conditions.

Andrews Kinetic Elutriator — A modification to the Schoene apparatus, is essentially the same funnel and chamber design as noted above with the exception of an injector nozzle located in the lowest chamber. The purpose of this nozzle is to provide energy to the system to aid in de-agglomeration of particles in the hindered settling section of the elutriator. At the time (1940's) superior sizing results were reported by the Australian Institute of Mining and Metallurgy.

In summary, the two main systems that are in use today by micronizers are the single point and Multi-tube systems. Most likely because of their simplicity in design, operation and reproducibility. There are of course proprietary design modifications that are made on the basis of good engineering and experience. It is doubtful that any two micronizers will agree that one system is better than the other.

Sub-Micron Separation

As noted in the base equation of Stokes. The falling rate of a particle is influenced by its mass and density relative to gravitational acceleration and the density of the medium the particle is in.

Extremely fine separations in the order of 0.05 microns are obtainable by traditional elutriation systems, the time however to accomplish this (on a production basis) would be several months if not years. Additionally lighter or less dense materials would not be able to be elutriated.

So we turn to the last phase of micron grading that is known as centrifugation. Centrifugation uses mechanical energy to overcome the weaker forces of gravity. Classification by this methodology is accomplished by rotational speeds imparting high gravitational forces that run perpendicular to the general direction of sedimentation.

Centrifugation — applied mechanical force to overcome random Brownian movement. The base calculation is given by the form $F_c = 0.000142n^2Db$.

The magnitude of the force (F_c) exerted is given in terms of multiples of the standard force of gravity. This is where n = rotational speed, D = bowl or container diameter. By substituting relatively high rotation speed even for moderately sized bowls or containers very high g forces are obtained.

Care should be taken in the materials of design and in centrifuge operation. Critical speeds as well as materials of construction should be carefully engineered to match the requirements of the materials to be processed.

For centrifuge selection several methods are described by Moyers (Chem. Eng., vol.73,182 1966) and would prove invaluable in centrifuge selection.

Particle Analyzers — There are a number of particle size analyzers that are available on the market. There are electro zone, disk centrifuge and laser, laser and visible light analyzers. While all of these analyzers work well on spherical products. Most materials are not truly spherical. The problem becomes one of understanding the optics of the analyzer its capability to measure non-spherical particles and a program of using the best attributes of each analyzer to produce a standardized product.

One methodology that is suggested is to establish a standard of the material to be measured. This is accomplished by image analysis of micron and sub-micron materials. For micron sizes of greater than 5 microns standard light microscopy with image analysis capability could be used to establish the true attributes of the material to be analyzed. For sub-micron sizes Scanning Electron Microscopy with image analysis capability should be used.

Aspect ratios, circularity index, roundness are but a few the parameters to study. These same samples (standards) are then analyzed by the particle size analyzers of choice and a database is established. Once a database is established using standard statistical techniques (described earlier) a check of reproducibility and repeatability is accomplished by use of gage studies.

Gage Analysis — Method of analysis to verify the measurement error in measurement systems. The two components to look at are repeatability and reproducibility. The 10% rule does not discriminate the tolerance level. The measurement instrument has no feasibility to measure the process variation if the percent tolerance by total R&R does not exceed 10%. If it does not exceed 10% the measurement is excellent. Between 10 and 30% the process is said to be in control with acceptable results. Over 30% the process or the operators need examination to discern where errors may lie in the system.

Please remember the three elements of gage accuracy are linearity, accuracy and stability. We believe the instrumentation and process of standardization depicted in this paper satisfies this precept.

In summary; the precepts stated in this paper outline the background and current techniques available to produce micron and sub-micron particles to an acceptable standard that is industry wide acceptable.

Editor's note: *There is a wealth of data in Perry's Chemical Engineering Handbook, Taggart Mineral Processing Handbook, and Handbook of Mixing Technology by Oldshue and on the Web. It is a matter of research and application.*