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Comparison of Subsieving Techniques Based on the Relation of Particle Size and Specific Surface Area in Mineral Processing

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This investigation is concerned with the comparison of the advantages and disadvantages of subsieving techniques widely used in mineral processing throughout the world. With this purpose, the subsieving techniques of Andreasen pipette, Coulter counter, and laser diffraction methods were compared each other by determining the relationship between specific surface area and particle size distribution. In the tests, the two different standard quartz sand samples (A and B) from the Official Material Testing Institute for Nonmetallic Minerals, Clausthal-Zellerfeld with a particle density of $\rho = 2.65 \text{ g/cm}^3$ were used. As a result, the Coulter counter method had the most favorable results in terms of calculated specific surface areas of $3848 \text{ cm}^2/\text{g}$ for sample A and $2852 \text{ cm}^2/\text{g}$ for sample B, with standard deviations 41.29 and 32.48, respectively.

Keywords: Andreasen pipette, coulter counter, laser diffraction, specific surface area, subsieving

1. Introduction

Sieve analysis is one of the oldest methods of size analysis and is accomplished by passing a known weight of sample material successively through finer sieves and weighing the amount collected on each sieve to determine the percentage weight in each size fraction Wills (1985). It has been used since early Egyptian times for preparation of foodstuffs. The simplest sieves would be made of some woven material, but punched plate sieves are recorded in early Egyptian drawings. It is a particularly useful technique since particles are sorted into categories on the basis of size alone, independently of their other properties such as density, surface properties, etc. (Allen 1992). Moreover, it has an important role for the determination of particle size distribution, particle liberation degree, and designing of comminution, grinding, and drying units (Saklara et al. 2000). However, it is rarely carried out on a routine basis below about $38 \mu\text{m}$; below this size, the operation is referred to as subsieving. The subsieving techniques are also used in different industries such as chemistry, fertilizer, pharmacology, astronomy, meteorology, cement, blood analysis, etc. There are over 400 subsieving techniques in different industry for different purposes Bart and Flippin (1995). However, the most widely used procedures are sedimentation, electronic sensing, and laser diffraction methods in the mineral processing industry.

Therefore, Andreasen pipette, Coulter counter, and laser diffraction methods were chosen for the investigations in this paper and were compared in terms of their advantages and disadvantages. Typically, in particle size analysis, size is expressed as an “equivalent sphere diameter” based on the response of the particle to the particular measurement used. An important consequence is that, for irregular particles, different kinds of measurements (e.g., sieving, sedimentation, and light scattering) give different apparent sizes for the same particle. Ideally, particles of identical size and shape would give the same response to sieving or sedimentation or to volume-based methods such as the Coulter counter method (Hogg et al. 2004). Although each method has a different theory to determine particle size distribution, the differences of size distribution curves depend not only on the particular instrument and material tested but also on the shape of the size distribution of the sample being measured (Austin 1997).

This study aimed to investigate of advantages and disadvantages of these various methods by using standard quartz sand samples with certain specific surface areas. The subsieving techniques, widely used in mineral processing industry, are compared with each other in order to determine the relationship between particle size and specific surface area. For this purpose, the specific surface areas of samples were calculated by using different particle size distributions, (obtained from all subsieving methods) and compared with actual specific surface area values of samples. In the permeability method, the outer surface area of a particle collective is measured, which corresponds approximately with the geometric surface area. This allowed the samples to be tested with a Blaine apparatus and the results evaluated together.

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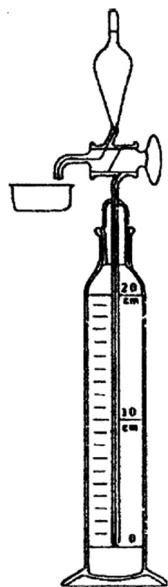


Fig. 1. Andreassen pipette technique.

1.1 Andreassen Pipette Technique

The Andreassen pipette method, which is one of the sedimentation methods, is among the most widely applied methods for subsieveing measurements. The method is simple, cheap, and readily available (Ulusoy et al. 2006). It has the advantage of producing a true fractional size analysis. The device is suitable for size measurement in the range of 1 to 50 μm . This method has same principle with decantation methods; however, it supplies to take experiments' conditions under control (Saklara et al. 2000). On the other hand, it is extremely tedious, as long settling times are required for very fine particles, and separate tests must be performed for each particle size.

The apparatus is a glass cylinder with an Andreassen pipette and a suspension column of initial height 200 mm. It consists of a thin, capillary pipette with tip fixed at a known depth in a vertical cylinder containing an initially homogenous suspension (see Figure 1).

1.2 Coulter Counter Technique

The Coulter counter technique is a method for determining the number and size of particles suspended in an electrolyte by causing them to pass through a small orifice on either side of the orifice, which is immersed an electrode. A known volume of an extremely dilute suspension of particles in a conducting fluid flows through a standard interchangeable aperture having an immersed electrode on either side. The particles traverse the aperture substantially one at a time, causing an attendant change in electrical resistivity proportional to the volume of the particle passing through the aperture. The amplified signals pass through a gate circuit, which ensures that only pulses greater than a preselected threshold value are counted; thus, the pulse count records the number of particles greater than a certain size (Harris 1965).

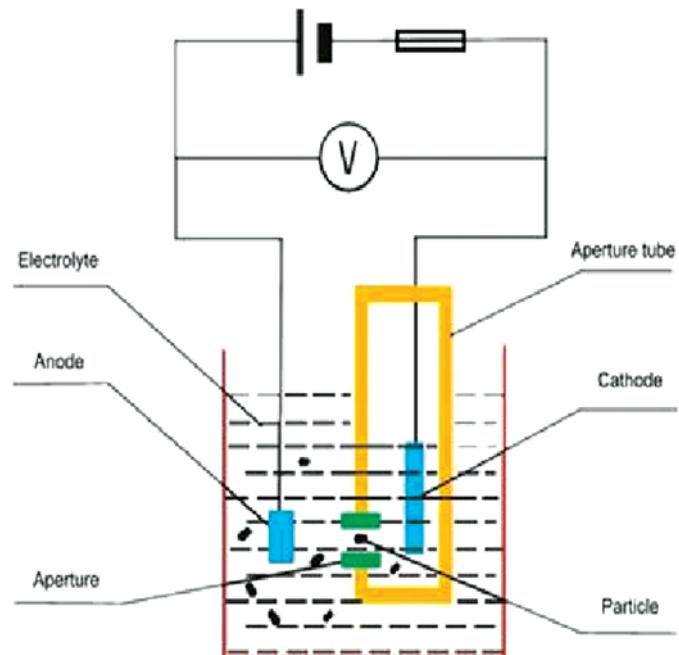


Fig. 2. Working sketch of Coulter–counter technique (<http://folk.uio.no>). (Figure available in color online.).

The basic assumption underlying the operation of the Coulter counter is that the response is directly proportional to particle volume. The reliability of the instrument depends on the accuracy of this assumption.

Since analyses may be carried out rapidly with good reproducibility using semi-skilled operators, the method has become popular in a wide range of industries.

The instrument response essentially is to particle volume. It has been claimed that particle shape, roughness, and the nature of the material have little effect on the analysis, but there is considerable evidence that the size parameter measured is, in fact, the envelope of the particle. Comparison with other techniques has been found to be good for spherical particles; for nonspherical particles, results may differ with porous materials such as nylon the measured volume may be several times skeletal volume (see Figure 2).

1.3 Laser Diffraction Technique

For particle size analysis, optical instruments based on the measurement of laser light scattered by the particles in the forward direction have now become a popular and standard technique in the laboratory. Since the light scattered in the forward direction can often be accurately approximated by diffraction theory, the class of instruments have become generally known as laser diffraction instruments. Compared with other particle sizing techniques, laser diffraction has the advantage of high speed, good reliability, and high reproducibility. It is increasingly being applied for direct online process and quality control of powder processes (Zhanhua et al. 2000).

This technique is based on the fact that the grain diameter is inversely proportional with the refraction angle of the

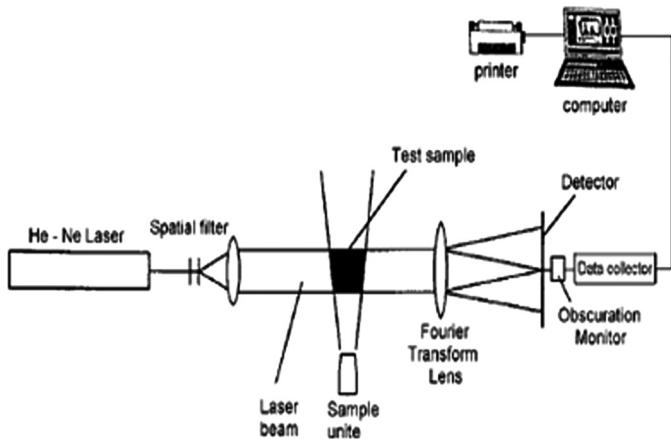


Fig. 3. Working scheme of laser diffraction technique.

laser light. In other words, larger diameter grains refract laser light with small angles whereas small diameter grains refract laser light with greater angle. As particle size decreases, the observed scattering angle increases logarithmically (Rawle 1995a; 1995b; Kippax 2005; Ulusoy et al. 2008).

The most important difference of the laser diffraction system is that it determines grain-size distribution according to volumetric basis. By using the density of scattered laser light, the volumes of grains are calculated and results are presented. The laser diffraction technique utilized equivalent sphere theory for the calculation of grain sizes. The most important advantage of this technique is that it does not require the specific gravity and weight of grains. By this way, mistakes during the calculation of weight is avoided (Orhan et al. 2004). On the other hand, the laser diffractometer uses a very small sample, usually about 4 g, and special care is required to ensure the sample is representative. The equipment has alternative configurations for predicting the light scattering pattern of particles, using the Fraunhofer approximation or the complete Mie theory (Schneider et al. 2006) (see Figure 3).

2. Materials and Methods

In the present study, the standard substance quartz sand from the Official Material Testing Institute for nonmetallic minerals, Clausthal-Zellerfeld, with a particle density of $\rho = 2.65 \text{ g/cm}^3$, was chosen as the test material. Two different test samples were used; sample A had specific surface area of $3970 \text{ cm}^2/\text{g}$ and sample B had specific surface area of $2930 \text{ cm}^2/\text{g}$. The samples were sieved at the same sieving sizes, but with different size distributions. (The quartz sands, used in our tests, were a special production for the calibration of the Blaine-specific surface area analyzer and had certain specific surface areas.) First, the samples were sieved to under $38 \mu\text{m}$ with the classical wet screening method; then our subsieving techniques (Andreasen pipette, Coulter counter, and laser diffraction) were applied for determination of finer size distributions; this was done repeatedly and then combined to obtain the sieving data. The combined particle size distributions were obtained by

multiplying the cumulative undersize value of $38 \mu\text{m}$ as determined by the sieving techniques.

In the Andreasen pipette experiments, the 2% solid contents by weight suspension were used. The samples were continuously taken to 10 ml cell in determined time intervals, which were doubled each time, starting from 60 s and ending at 1920 s in each experiment. The particle size distributions were calculated by using Stoke's law.

$$\nu = \frac{d^2 g (D_s - D_f)}{18 \mu} \quad (1)$$

where μ is viscosity, centipoises ($\text{g}/\text{cm} \cdot \text{sec}$); ν is velocity, cm/sec ; D_s is density of solid particle, g/cm^3 ; D_f is density of fluid, g/cm^3 ; d is diameter of a sphere, cm ; and g is acceleration due to gravity, cm/sec^2 .

In the Coulter counter experiments, the 1% solid contents by weight suspension were used. During the tests, the threshold values were changed to obtain different pulses, which identify different particle sizes.

In the laser diffraction experiments, the 10 g sample was used. The particle size distributions were calculated automatically by a computer program.

The specific surface areas of test materials were then determined with a Blaine apparatus, which is a test apparatus for determining the specific surface area by using the permeability of a fluid through a particle bed. In the permeability method, the experiments are performed repeatedly in order to compare actual values. In scope of this research, the average mean of measured specific surface areas from 10 measurements were $3959 \text{ cm}^2/\text{g}$ with the 5.49 standard deviation for sample A and $2892 \text{ cm}^2/\text{g}$ with the 6.80 standard deviation for sample B. The tests were repeated 10 times and standard deviations were calculated and conducted at room temperature.

The relationship between particle size distribution and specific surface area have investigated in many other studies and are presented by Equations (2)–(4) (Hewlett 1998; Summer et al. 1989; Zhang et al. 1995; Kalkert 2000; Gultepe and Ergun 2009).

$$SSA = \frac{6}{\rho} \cdot \sum_{i=1}^n \left(\frac{W_i}{X_{mi}} \right) \quad (2)$$

$$SSA = \frac{6}{\rho} \cdot \varphi \cdot \sum_{i=1}^n \left(\frac{W_i}{X_{mi}} \right) \quad (3)$$

$$\varphi = \frac{\text{Actual Value of SSA}}{\text{Calculated Value of SSA}} \quad (4)$$

Equation (2) is used for spherical shape particles and Equation (3) is usually used for nonspherical shape particles. Much work has been done to study the effect of particle shape on size distribution as measured by different techniques. It has been shown that particle shape has a strong influence on size distribution measured by light diffraction, electrical sensing zone, x-ray sedimentation, photo-sedimentation, light-obscuration, and image analysis (Barreiros et al. 1996; Kaye

et al. 1997, 1999; Endoh et al. 1998; Xu and Guida 2003; Li et al. 2005; Taşdemir et al. 2011).

In this case, the samples, used in our tests, are special a production of standard quartz sands (samples A and B) from the Official Material Testing Institute for Nonmetallic Minerals, Clausthal-Zellerfeld, with certain specific surface areas and their shapes are rounded specially. The shapes of the samples have high sphericity (S) values and the shapes of samples are highly close to sphere.

Thus, Equation (2) was used for all calculations and then the adjusting coefficients were defined in place of shape factors for each sample by using Equations (3) and (4), where SSA is specific surface area (cm^2/g), ρ is density of particles (g/cm^3), w_i is the weight percentage of fractions, X_{mi} is mean particle sizes of x_i and x_{i+1} fractions, φ is the adjusting coefficient (i.e., it is rounded in shape, so it is called as adjusting coefficient).

Moreover, there are some different approaches in order to find the mean particle sizes of x_i and x_{i+1} fraction such as arithmetic mean, geometric mean, harmonic mean, and quadratic mean (see Equations (5)–(8)). The specific surface area values were calculated by using all approaches in terms of different mean calculations for each subsieving technique. They then were compared with actual specific surface area of samples.

$$x_{mi} = \frac{x_i + \dots + x_n}{n} \quad \text{arithmetic mean} \quad (5)$$

$$x_{mi} = n \sqrt{x_i \dots x_n} \quad \text{geometric mean} \quad (6)$$

$$x_{mi} = \frac{n}{\frac{1}{x_i} + \dots + \frac{1}{x_n}} \quad \text{harmonic mean} \quad (7)$$

$$x_{mi} = \sqrt{\frac{x_i^2 + \dots + x_n^2}{n}} \quad \text{quadratic mean} \quad (8)$$

3. Results and Discussions

According to theory difference between methods, the different particle size distributions were obtained and combined with wet screening results (Figure 4).

The combined particle size distributions, obtained from each method, were used for calculations of specific surface area in the terms of different equations (see Equations (5)–(8)). The calculated specific surface areas and their standard deviations by using the above equations are given in Tables 1 and 2 and the adjusting coefficients were calculated for each approach are given in Tables 3 and 4. Figure 5 shows the relationship between adjusting coefficients and equations, which were used for calculating of particle size mean.

This study mainly focused on comparing the subsieving techniques based on different physical properties and theories. Thus, no specific inferences about which technique is best for particle size distribution can be made.

There are some advantages and disadvantages of all techniques. Thus, it is not surprising that the consistent comparison of size distributions measured by one technique with those measured by another has proved difficult. According to calculated specific surface areas and adjusting coefficient values, the best reliable results were obtained from Coulter counter technique, then the Andreasen pipette and laser

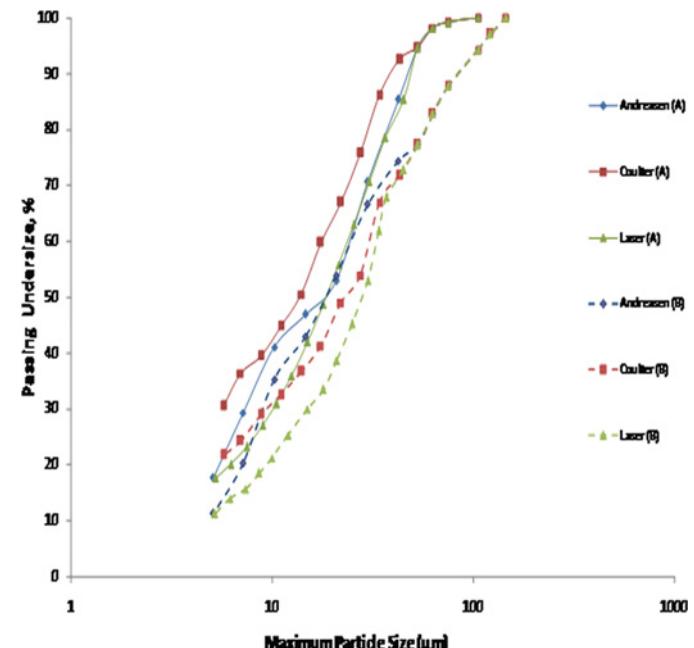


Fig. 4. Combined particle size distributions. (Figure available in color online.).

Table 1. Calculated specific surface areas and their standard deviations for sample A

Number of equation	Andreasen pipette		Coulter counter		Laser diffraction	
	SSA (cm ² /g)	STD	SSA (cm ² /g)	STD	SSA (cm ² /g)	STD
Equation (5)	2537.48	26.22	3010.18	37.41	2326.52	14.25
Equation (6)	3015.27	31.05	3848.18	41.29	2794.93	19.67
Equation (7)	3654.36	35.55	5023.85	55.84	3432.28	30.06
Equation (8)	2297.02	24.27	2631.47	24.21	2101.59	13.92

The minimum particle size is assumed as 1 μm.

SSA = specific surface area.

STD: standard deviation.

SSA = specific surface area.

STD: standard deviation.

Table 2. Calculated specific surface areas and their standard deviations for sample B

Number of equation	Andreasen pipette		Coulter counter		Laser diffraction	
	SSA (cm ² /g)	STD	SSA (cm ² /g)	STD	SSA (cm ² /g)	STD
Equation (5)	2537.48	26.22	3010.18	37.41	2326.52	14.25
Equation (6)	3015.27	31.05	3848.18	41.29	2794.93	19.67
Equation (7)	3654.36	35.55	5023.85	55.84	3432.28	30.06
Equation (8)	2297.02	24.27	2631.47	24.21	2101.59	13.92

The minimum particle size is assumed as 1 μm.

SSA = specific surface area.

STD: standard deviation.

Table 3. Calculated adjusting coefficient and their standard deviations for sample A

Number of equation	Andreasen pipette		Coulter counter		Laser diffraction	
	Adj. coeff.	STD	Adj. coeff.	STD	Adj. coeff.	STD
Equation (5)	1.56	0.0066	1.32	0.0094	1.71	0.0036
Equation (6)	1.32	0.0078	1.03	0.0104	1.42	0.0050
Equation (7)	1.09	0.0090	0.79	0.0141	1.16	0.0076
Equation (8)	1.73	0.0061	1.51	0.0061	1.89	0.0035

3970 cm²/g was used as actual value of specific surface area according to data of Clausthal-Zellerfeld Institute.

SSA = specific surface area.

STD: standard deviation.

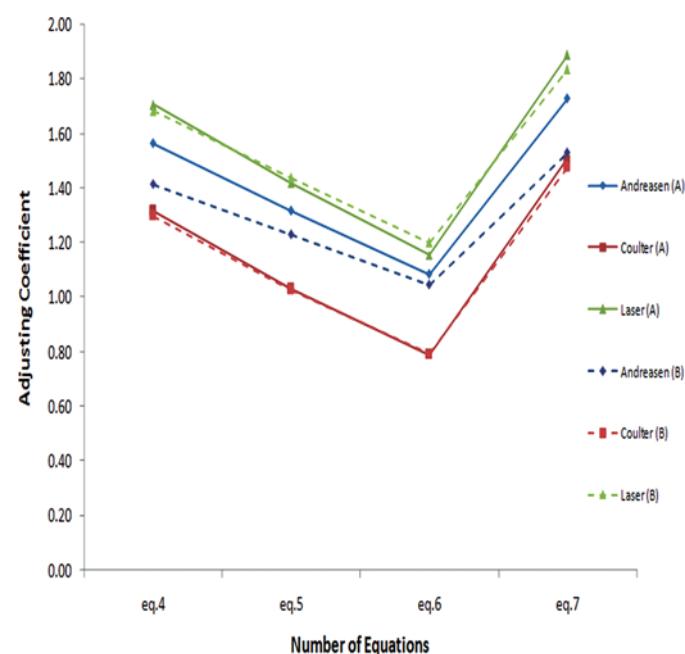
Table 4. Calculated adjusting coefficient and their standard deviations for sample B

Number of equation	Andreasen pipette		Coulter counter		Laser diffraction	
	Adj. coeff.	STD	Adj. coeff.	STD	Adj. coeff.	STD
Equation (5)	1.56	0.0162	1.32	0.0164	1.71	0.0105
Equation (6)	1.32	0.0136	1.03	0.0111	1.42	0.0100
Equation (7)	1.09	0.0106	0.79	0.0088	1.16	0.0101
Equation (8)	1.73	0.0183	1.51	0.0139	1.89	0.0125

2930 cm²/g was used as actual value of specific surface area according to data of Clausthal-Zellerfeld Institute.

SSA = specific surface area.

STD: standard deviation.

**Fig. 5.** The variation of adjusting coefficients in order to different mean approaches. (Figure available in color online.).

diffraction, respectively. In a similar study, Xu and Guida (2003) investigated the comparison of laser diffraction, electrical sensing zone (Coulter counter principal), and image analysis methods by using unimodal sphere glass bead particles; they indicated that particles with mean values obtained from the three technologies agree well. The trend in the deviation of laser diffraction results from those of electrical sensing zone and dynamic image analysis is very clear as the shape of particles departs from that of a sphere. On the other hand, electrical sensing zone and dynamic image analysis are more appropriate in obtaining equivalent spherical diameters.

However, the Coulter counter technique has disadvantages such as having narrow analysis intervals, low analysis speed, operational difficulties, and only being able to be used in wet applications. Advantages to the laser diffraction technique include analysis speed, ability to be used in dry and wet application, wide analysis intervals, easy usage, and adaptation of online analysis systems. The Andreasen pipette technique generally shares disadvantages of the Coulter counter technique, but it has important advantages such as being a cheap and easy method for analysis under 38 µm particles.

4. Conclusion

In this study, the methods were evaluated by means of their relationship between particle size distributions and specific surface areas. In this manner, the specific surface areas were calculated by using different approaches for each method with two different fractions of quartz sand samples.

The results show that the Coulter counter method was the most favorable technique in order to obtain a ratio between calculated specific surface area and actual surface area values (Figure 5). The most favorable results were found as 3848.18 cm²/g for sample A, with the actual specific surface area of 3970 cm²/g, and 2852.78 cm²/g for sample B, with the actual specific area of 2930 cm²/g.

The second most favorable technique was the Andreasen pipette, giving the calculated specific surface area of 3654.36 cm²/g for sample A, and 2796.13 cm²/g for sample B. The laser diffraction technique gave specific surface of 3432.28 cm²/g and 2439.72 cm²/g for samples A and B, respectively. The Coulter counter technique had the minimum adjusting coefficient value of 1.03. Normally, it can be described as shape factor of sample. In this case, the quartz samples, as used in our tests, were a special production from the Clausthal-Zellerfeld Institute. Thus, the shapes of sample were rounded specially. Therefore, the shape factor was ignored and it was defined as adjusting coefficient as discussed in this article.

In specific surface area calculations, the mean of fractions were calculated by four different approaches: arithmetic mean, geometric mean, harmonic mean, and quadratic mean (see Equations (5)–(8)). The best results were obtain from geometric mean for Counter coulter technique, whereas the harmonic mean was found to be best for others. The adjusting coefficients in order for each equation are shown in Figure 5.

Finally, in the evaluation of all methods, the most favorable sizing method was determined to be the Coulter counter under 38 µm particles. However, the laser diffraction method has very important advantages (as discussed above).

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