Dry Powder Particle Size Analyses for Dry Powder Processes (Restriction or Recommendation)

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Abstract

It is generally known that the results of particle size analyses done in liquid suspension are more reliable than those in a dry system. This stems from the fact that most principles are based on sedimentation or electric conductivity changes in liquids. With the development of laser diffraction instruments for particle size distribution, the dry powder measurement became quite significant. Although we know the difficulties of dispersion of very fine particles in gases, we have to take into account the great importance of sample preparation procedures for the wet technique. This involves the influence of suitable liquid, the proper dispersant and the time of dispersion. The topic of this work is comparison of the results obtained by dry and wet techniques on laser diffraction instruments evaluated by mass balance for air classification processes with a cut size under 10 micrometers. The comparison was carried out with different materials. Wet analyses were made with well-known instruments. The results are supplemented also by other principles of measurement. The accordance of analyses and mass balance for fractions of coarse and fine products with input material is greatly influenced by instrument quality. The differences in the results obtained with the same instrument by wet and dry techniques are relatively small even in the range of the low limit of the instrument. So it wouldn't always be necessary to make use of the wet technique, rather, more attention should be given to the quality and performance of the instruments.

1. Introduction

Particle size analyses are very important in process control and the determination of material properties. In the last decade, we could choose between several analyzers which function on the basis of laser diffraction. One of the main advantages of this instrument is the possibility of using the dry powder feeding system. The question arises at which lowest limit can a dry analysis be a good substitute for a wet one.

Data from the dry powder analyses are quite unfavorable, and some manufacturers do not have a dry system in their commercial programs although dry powder analyses are of great interest especially for on line measurements. The report by K. Leschonski, S. Roethele, and U. Menzel (lit. 1) seems promising and points in this direction. The main problem lies in good preparation of gas suspension without agglomerates in the sample.

In the range of about one micrometer, there are different principles for laser diffraction scattering (Fraunhofer,Mie). Of greatest interest for the dry power technique are analyzers using low angle laser light scattering, or Fraunhofer's law. The particle size analyzer can be tested on the basis of two different performances, accuracy and reproducibility. Reproducibility was determined in accordance with mass balance from size interval to size interval for the input material and for
both classification products, the fine and coarse fraction. For each test, all of the three samples have to be analyzed. The description relevant to the evaluation of results is given in the next section. In the second part of this work, the same evaluation procedure was used for the comparison of dry/wet particle size analyses using our equipment.

2. Evaluation procedure

It is quite difficult to achieve the mass balance for a classification process if the reproducibility of the analyzer used is low. Common practice for determination of the reproducibility is a degree of agreement between measurements repeated several times as described in lit.(2). In our case, a good reproducibility means that we have a high agreement between the calculated and measured mass balance data for each particle size interval of input, fine and coarse product of classification. As an example for size interval $i$:

$$m_{bi} = m_{fi} + m_{ci},$$

$m_{bi}$ mass fraction in input material
$m_{fi}$ mass fraction in fine product
$m_{ci}$ mass fraction in coarse product

or by analysis results:

$$100 \cdot \Delta D_i = X_f \cdot \Delta D_{fi} + X_c \cdot \Delta D_{ci},$$

$X_f$ per cent of fine product
$X_c$ per cent of coarse product
$\Delta D_{fi}, \Delta D_{bi}, \Delta D_{ci}$ per cent in interval by analysis

Standard deviation is defined as:

$$\text{STD.DEV.} = \sqrt{\frac{\sum_{i=1}^{n} ((X_f \cdot \Delta D_{fi} + X_c \cdot \Delta D_{ci} - 100 \cdot \Delta D_{bi})^2)/\Delta D_{bi}}{n}}$$

$n$: number of particle size intervals in which the sample is divided

An example of such an evaluation for reproducibility or detection is given in Table 1. However, the determination of accuracy with standard powders is not included in this work, because it is reported on in the report by Allen and Davis (lit.2). On the other hand, the differences in analysis between the wet and dry technique do not correlate with the algorithm for the calculation of particle size distribution which is the main particularity of each laser diffraction instrument. At this point, it can be mentioned that the results of our tests are comparable in terms of reproducibility with the statements in lit. 2.

3. Results of the analyses

The main purpose of our work was to test some particle size analyzers with samples obtained by air classification. We also wanted to verify the agreement that can be expected between the wet and dry techniques using the same samples. The classification process was performed with a laboratory Alpine centrifugal classifier.

3.1. First example

The first sample of classification was calcite (A) in the range from 0 to 100 micrometers and with a cut size of about 10 micrometers. The amount of fines (B) is 40 %, and the amount of coarse product (C) is 60 %. The samples A, B and C were delivered to the manufacturers of laser diffraction instruments. Recommendations for sample preparation procedures were enclosed. The recommendations prescribed water as a medium, Na-pyrophosphate as a dispersant, and they suggested 5 minutes as the time of dispersion in the ultrasonic bath. Particle size analyses are shown in Fig.1 to Fig.3. The curves in the diagrams are not specified by types of instruments or manufacturers, because not all of our requests for permission to publish the results, had been received yet. Besides curves from laser diffraction analyzers, there are also data from other principles, such as sedimentation and the electrical sensing zone method. Results obtained by using the well-known Sedigraph 5100 are represented by large black marks. The results obtained by dry powder analysis on Microtrac SRA are marked with triangular symbols. Analyses by Sedigraph and Microtrac were conducted at our institute. Differences among the curves by the wet technique are relatively great, which is understandable and expected. However, the position of the analysis obtained by the dry
### Table 1: Particle size analyses—Evaluation of results

| Size range | INPUT (A) | FINE (B) | COARSE (C) |
|------------|-----------|----------|------------|
| micrometer | ΔR        | ΣΔR      | ΔR         | ΣΔR      | ΔR         | ΣΔR      |
| +64.9      | 4.00      | 4.00     | 0.00       | 0.00     | 7.50       | 7.50     |
| +52.7 – 64.9 | 3.10      | 7.10     | 0.00       | 0.00     | 5.09       | 12.60    |
| +42.8 – 52.7 | 3.10      | 10.20    | 0.09       | 0.09     | 4.50       | 17.10    |
| +34.7 – 42.8 | 3.90      | 14.10    | 0.09       | 0.19     | 6.00       | 23.10    |
| +28.1 – 34.7 | 4.90      | 19.00    | 0.10       | 0.30     | 8.00       | 31.10    |
| +22.8 – 28.1 | 6.40      | 25.40    | 0.39       | 0.69     | 11.00      | 42.10    |
| +18.5 – 22.8 | 8.19      | 33.60    | 1.00       | 1.69     | 14.10      | 56.20    |
| +15 – 18.5  | 9.60      | 43.20    | 2.60       | 4.30     | 15.60      | 71.80    |
| +12.2 – 15  | 10.00     | 53.20    | 6.00       | 10.30    | 12.20      | 84.00    |
| +9.91 – 12.2 | 9.30      | 62.50    | 10.80      | 21.10    | 7.10       | 91.10    |
| +8.04 – 9.91 | 7.80      | 70.30    | 14.30      | 35.40    | 3.10       | 94.20    |
| +6.52 – 8.04 | 6.50      | 78.80    | 14.60      | 50.00    | 1.40       | 95.60    |
| +5.29 – 6.52 | 5.09      | 81.90    | 11.70      | 61.70    | 0.59       | 96.20    |
| +4.3 – 5.29  | 4.00      | 85.90    | 9.40       | 71.10    | 0.40       | 96.60    |
| +2.83 – 4.3  | 5.60      | 91.50    | 12.90      | 84.00    | 0.40       | 97.00    |
| +1.86 – 2.83 | 3.30      | 94.80    | 7.50       | 91.50    | 0.30       | 97.30    |
| +1 – 1.86   | 2.79      | 97.60    | 6.00       | 97.50    | 0.79       | 98.10    |
| +0 – 1      | 2.40      | 100.00   | 2.50       | 100.00   | 1.90       | 100.00   |

Mass Balance: Diff = A (From Analysis) – A (Calculated From B+C)

Percentage of Fine (B) = 40% of Coarse (C) = 60%

| size range | A | B | C | B+C | DIFF | VAR |
|------------|---|---|---|-----|------|-----|
| micrometer | ΔR | ΣΔR | ΔR | ΣΔR | ΔR | ΣΔR |
| +64.9      | 4.00 | 4.00 | 4.50 | 4.50 | -0.12 | 0.01 |
| +52.7 – 64.9 | 3.10 | 0.00 | 3.06 | 3.06 | 0.01 | 0.00 |
| +42.8 – 52.7 | 3.10 | 0.03 | 2.70 | 2.74 | 0.11 | 0.01 |
| +34.7 – 42.8 | 3.90 | 0.03 | 3.60 | 3.64 | 0.06 | 0.00 |
| +28.1 – 34.7 | 4.90 | 0.04 | 4.80 | 4.84 | 0.01 | 0.00 |
| +22.8 – 28.1 | 6.40 | 0.15 | 6.60 | 6.76 | -0.05 | 0.00 |
| +18.5 – 22.8 | 8.19 | 0.40 | 8.46 | 8.86 | -0.08 | 0.00 |
| +15 – 18.5  | 9.60 | 1.04 | 9.36 | 10.40 | -0.08 | 0.00 |
| +12.2 – 15  | 10.00 | 2.40 | 7.32 | 9.72 | 0.02 | 0.00 |
| +9.91 – 12.2 | 9.30 | 4.31 | 4.26 | 8.58 | 0.07 | 0.00 |
| +8.04 – 9.91 | 7.80 | 5.72 | 1.86 | 7.58 | 0.02 | 0.00 |
| +6.52 – 8.04 | 6.50 | 5.84 | 0.84 | 6.68 | -0.02 | 0.00 |
| +5.29 – 6.52 | 5.09 | 4.68 | 0.35 | 5.04 | 0.01 | 0.00 |
| +4.3 – 5.29  | 4.00 | 3.76 | 0.24 | 4.00 | 0.00 | 0.00 |
| +2.83 – 4.3  | 5.60 | 5.16 | 0.24 | 5.40 | 0.03 | 0.00 |
| +1.86 – 2.83 | 3.30 | 3.00 | 0.18 | 3.18 | 0.03 | 0.00 |
| +1 – 1.86   | 2.79 | 2.40 | 0.47 | 2.87 | -0.02 | 0.00 |
| +0 – 1      | 2.40 | 1.00 | 1.14 | 2.14 | 0.10 | 0.01 |

TOT. VAR. = 0740874416
STD. DEV. = 1.51216713 (% Per Interval) Intervals = 18

ΔR = weight % in range
ΣΔR = cumulative % oversize
Method-Instrument: Malvern Mastersizer (Cinkarna-Celje)
technique on Microtrac SRA, for the finest sample (B) was surprising. Discrepancies among the results of using the wet technique increase when partition curves for interpretation of the classification process are used, as can be seen in Fig. 4. In this diagram, it is quite evident how great the differences can be in the description of the same process of classification. It is true that the differences arise from both types of instrument properties: accuracy and reproducibility, but it must be borne in mind that the same (laser diffraction) and wet techniques were used. Below the diagram, the values of the standard deviation in relationship to reproducibility are given. Items obtained by Sedigraph 5100 and by dry powder feeding on Microtrac SRA are shown in Fig. 4. The same diagram also shown in clearly indicates that the accuracy and reproducibility of the process description are not related.

3.2. Second example

The second example of classification process evaluation was also made with calcite,
but with finer particle size distribution and at a lower cut size, about 5 micrometers. Analyses were performed in our laboratory on Microtrac SPA and SRA with wet and dry techniques. As a part of Fig.5 which shows partition curves, the values for the standard deviation with respect to reproducibility are also given. Fig.6 shows curves of cumulative undersize, and Fig.7 shows the differential curves of those analyses.

![Fig. 5 Partition curves for the second example of classification](image)

![Fig. 6 Particle size analyses for the second example of classification](image)

**3.3. Third example**

The third example of classification was executed with Aluminium hydroxide powder. The effect of classification was poor, which is significant for materials which are prone to form agglomerates. Regardless of this, the differences between the partition curves (Fig.8) obtained by wet and dry techniques are not so great. This example is also interesting as a case in which the finest distributions for samples were attained by the dry technique (Fig.9 and Fig.10), although wet analyses were made in water and in the recommended organic medium.

![Fig. 8 Partition curves for the third example of classification](image)

**4. Conclusions**

The objective of this research project was to determine the possibility of adequately substituting the wet analysis with a dry one.
The wet analyses were carried out on different analyzers, and the dry ones are done on Microtrac SRA with some changes on the dry measurement cell. The results indicate that we can obtain the same information about the process with the dry as well as with the wet technique, even at the low limit range of the instrument. The differences between the partition curves under 5 micrometers are much greater in the analyses on instruments from various manufactures or in various models from the same producer. The reason lies in different algorithms for the determination of particle size distribution. In our case, the solid phase was dispersed in air flow with a velocity of about 30 meters per sec. This is a relatively undemanding condition, yet it is still sufficient for small discrepancies in the results obtained by the dry/wet technique.

The reproducibilities of the results obtained with dry analyses are the same as those obtained with wet analyses, but in comparison with other instruments and the wet technique, they are even better. We can expect to obtain equally small discrepancies in results with the dry/wet technique using other analyzers too, if the same conditions for gas suspension preparation are used. Comparisons were made only with a few samples, but we believe that similar results can be obtained for powders with similar distributions and properties. Extremely fine powders with a size distribution of under 2 or 3 micrometers are not the subject of this report. For such fine powders, good results can also be expected but only with careful and correct gas suspension preparation.

References
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