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Measuring suspended particle size with high accuracy

Abstract

The average particle size and its distribution are major characteristics of a powder. Instrumental techniques, i.e. particle size analysers, are frequently used and span a wide size range. The selection of the most suitable measurement procedure for a particular application may present a problem, aggravated by the complexity of comparing results produced by different methods.

The laser diffraction method is the most popular method of analysis. The authors therefore investigated the conditions required to improve the accuracy of the measurement. These conditions include the sample preparation (solvent, dispersant), the dispersion itself, and the optical characteristics of the particles. The paper summarizes the findings and defines general rules to be respected.

Keywords: powders, particle measuring methods, particle size analysis, particle size distribution analysis, laser diffraction

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Huili Zhang,[1] Jan Baeyens,[1,2] Qian Kang[3]
[1]Beijing University of Chemical Technology, College of Life Science and Technology, China
[2]University of Warwick, School of Engineering, Coventry, UK
[3]KU Leuven, Department of Chemical Engineering, Belgium

Correspondence: Qian Kang, KU Leuven, Department of Chemical Engineering, Belgium, Email kang.qian@kuleuven.be

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Introduction

The average particle size and its distribution are undoubtedly major characteristics of a powder, and the average particle size occurs in all correlations related to the powder behaviour in, e.g., packed, moving or fluidized beds, in pneumatic conveying and in dust filtration. When traditional sieving can no longer be used for smaller particles, instrumental techniques, i.e. particle size analysers, are used. These analysers extended the measured particle size to the micron or sub-micron sizes, and span a fairly wide size range. Among these instrumental techniques, major developments have taken place, resulting in a wide range of equipment and including, e.g., sedimentation, electrical sensing methods, laser diffraction, dynamic light scattering (for molecules and sub-micron particles), X-ray scattering, acoustic methods, and/or focused beam techniques.[1–15] The common equipment and a non-exhaustive list of companies involved are given in Table 1.

Table 1 Common particle size analysers and some of the suppliers

Method	Company	
Laser diffraction & scattering	SYMPATEC	www.sympatec.com
	MALVERN	www.malvern.com
	COULTER	www.beckmancoulter.com
	HORIBA	www.horiba.com
	LEEDS & NORTHRUP	www.leedsandnorthrup.com
	CILAS	www.cilas.com
	SHIMADZU	www.shimadzu.com
Photo sedimentation	HORIBA	www.horiba.com
	SHIMADZU	www.shimadzu.com
	BROOKHAVEN	www.bic.com
X-ray sedimentation	MICROMERITICS	www.micromeritics.com
Light obscuration	GALAI INSTRUMENTS	www.cortera.com
Electrical sensing zone	COULTER	www.beckmancoulter.com
	MICROMERITICS	www.micromeritics.com
Dynamic light scattering	MALVERN	www.malvern.com
	BROOKHAVEN	www.bic.com
Image analysis	ALLIED VISION	www.alliedvisiontec.com
Acoustic methods	SYMPATEC	www.sympatec.com
Focused beam		

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The final objective of the particle size analysis technology is to accurately measure the particle size distribution in any size range. And yet, repeat determinations often lack consistency and accuracy. It is essential to respect a number of rules, related to the sample preparation, the measurement procedure and to the analysers themselves. Procedures should hence be standardized and this has been presented in the ISO standards for each of the measurement techniques.[16–21] Each standard describes at some degree the advantages and limitations of particular methods. Unfortunately, no information is given about comparing results produced by different methods, and such a comparison often creates the impression that results are very different.

These differences are due to several reasons, associated with:

i. Sample preparation and handling

ii. Breaching instrument specifications

iii. Differences in presenting the particle size distributions.

The laser diffraction and scattering method is one of the most popular particle size analysers. The authors therefore conducted a number of experiments on a Malvern laser analyser to establish conditions and associated operating conditions required to improve the accuracy of the measurement.

The accuracy of the particle size analysis is largely dependent on a number of parameters, including the sample preparation, the degree of dispersion and the analysis itself. In most common suspension analysers, particles are dispersed in a solvent, mostly using a dispersant to lower the surface tension.

The main parameters that are to be considered in particle size measurement are listed in Table 2. The effect of these parameters on the particle sizing results will be illustrated in Section 3 of the paper.

Table 2 Major parameters, effects and actions required

Parameter	Effect	Comments
Cohesion	Agglomeration	Mostly Van der Waals forces; Counteract by promoting wetting and rupture
	(increase in effective d_p)	
Wettability	Cover particle surface with liquid; Replace air from external/internal surface	Liquid should be less polarisable than solids; Use surfactants
Rupture of agglomerates	Make particles individual	Induce shear on particles; Use mixing and/or ultrasound; Fibres and needles form strong bridges
		De-agglomeration requires higher energy input
Stabilisation	Agglomeration (increase in effective d_p)	Do not promote too heavy collision of particles;
		Use low particle concentration; Use de-flocculants (polyphosphates, organic polymers)

Assessment of relevant measurement parameters

Solvents

The solvent should satisfy the following conditions:

a. Be free of solid particles

b. Having a good affinity for the particles

c. Not dissolving the particles, or acting upon them by shrinking or swelling

d. Not being reactive with the particles

e. Having a different density from that of the sample in case of sedimentation

Usually deionised water is used for insoluble materials, although the pH of the deionised water could affect the zeta-potential of the particles. For materials which are insoluble but hardly wettable by water, adding an inorganic alcohol or a neutral detergent is effective, although detergents might cause foam formation. Organic solvents such as alcohols generally promote de-agglomeration of particles, whereas acetone or aromatics tend to promote particle adhesion on the cell wall, thus making measurements badly reproducible.

Dispersants

Dispersants have to be selected for each particle and solvent, but polyphosphates are usually applied for insoluble materials, with hexametaphosphate being most effective in most cases, although solutions cannot be stored for more than one day, since the dispersant loses its activity. The dispersant and its concentration should be selected on the basis of:

i. The zeta-potential, preferably below - 60 mV

ii. Avoiding visual obscuration and sedimentation

Suspension aids

Of course, particle samples should be de-agglomerated before the measurement. The dispersant is hence important, but also ultrasonic and mechanical mixing are essential. The effect of sonication and mixing is further debated using experimental results.

The energy input during sonication should be limited to avoid particle disintegration. Also, care has to be taken to use an inert sonication tip, since cavitation can erode the tip, and since particles from the tip contaminate the sample suspension. The time of sonication could influence the measurement, with the size distribution shifting to fine sizes. Sonication should therefore be limited and additional mechanical stirring could be necessary to avoid re-agglomeration.

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If this nevertheless occurs, either a different dispersant or its higher concentration are needed.

Particle refractive index

The laser diffraction and scattering method has many advantages such as wide dynamic range, good reproducibility, easy operation and quick measurement. This is the reason why the laser diffraction and scattering method is most widely used. However, this method has a disadvantage which is difficult to handle, i.e., the input value of particle refractive index. When sample particles are of μm order or smaller, the value of the particle refractive index considerably affects the results as shown in Section 3. Data on the particle refractive index values are listed in handbooks such as "Handbook of Chemistry and Physics".[22] If the index is unknown, its measurement is very difficult.

Review of the relevant sample preparation conditions

Some previously mentioned and additional effects are summarized in Table 3.

Table 3 Parameters affecting the instrumental particle size measurement

Sample
Particle and solvent density, particle refractive index
Solvent
Type, refractive index, density and viscosity
Dispersant
Type and concentration
Sample suspension
Concentration, and temperature
Dispersion
Beaker size, dispersion device (Ultrasonication bath or tip), suspension volume, power, frequency, duration of Ultrasonication, tip material and size, tip position
Treatment of suspension
Duration from preparation to measurement,
dilution ratio of the suspension for measurement

Results and discussion

Dispersants

Polyphosphate solutions are commonly used for insoluble powders, with hexameta-phosphate normally the most effective in many cases, although losing its activity in one day. As illustrated in the evolution of the zeta potential (ξ) with dosage of phosphates, the dispersing effect is active over a wide concentration range Figure 1. It is however clear that an over-dosage could lead to re-agglomeration rather than dispersion.

As alternative to polyphosphates, low-molecular weight organic dispersants are also widely used, including e.g. Daxad 11G,[23] a condensation (Na^+/K^+) product of naphthalene sulphonic acids. Higher concentrations do not improve the dispersion and concentrations around 1% are recommended. Illustration of its use is given in Figure 2.

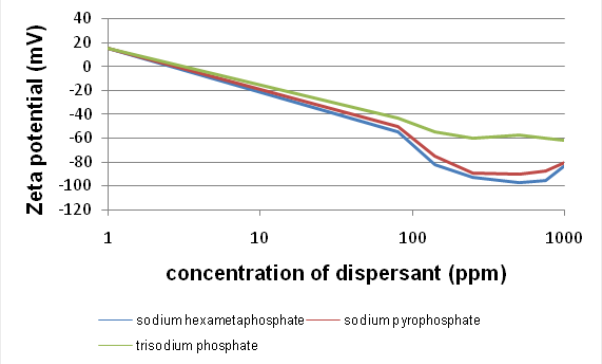

Figure 1 ξ -potential of Al_2O_3 versus dispersants and their concentration.

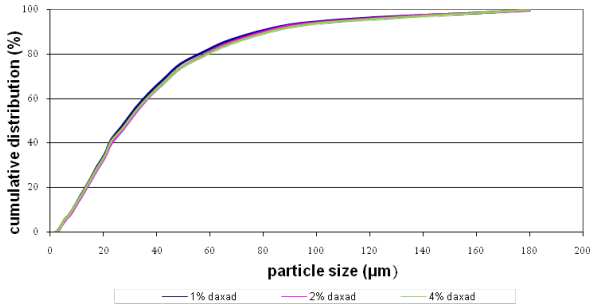

Figure 2 Effect of Daxad-concentration on measured particle sizes of SiO_2.

Dispersion

Solvents: As stated in *Section 2,* the use of organic solvents can be necessary for water soluble powders. Alcohols, acetone or even aromatics can be used (as a function of the density of the powders). Figures 3-6 provide examples of organic solvents, with the poor performance of acetone probably due to particle adhesion on the cell wall.

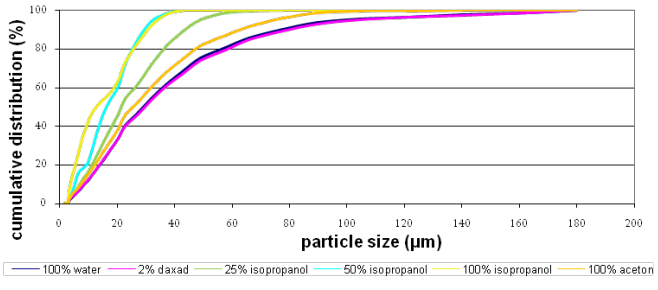

Figure 3 Effect of solvents on the measured particle sizes of SiO_2.

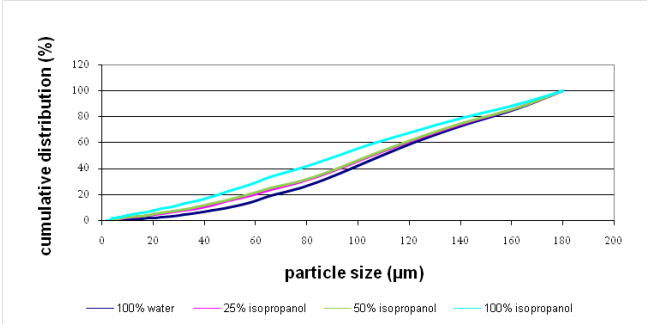

Figure 4 Effect of solvents on the measured particle sizes of saw dust.

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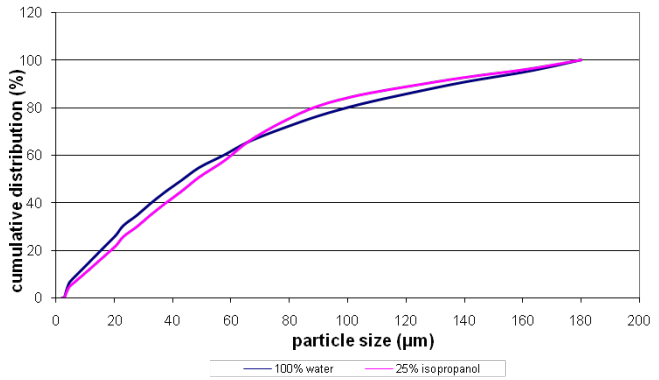

Figure 5 Effect of solvents on the measured particle size of incinerator fly-ash.

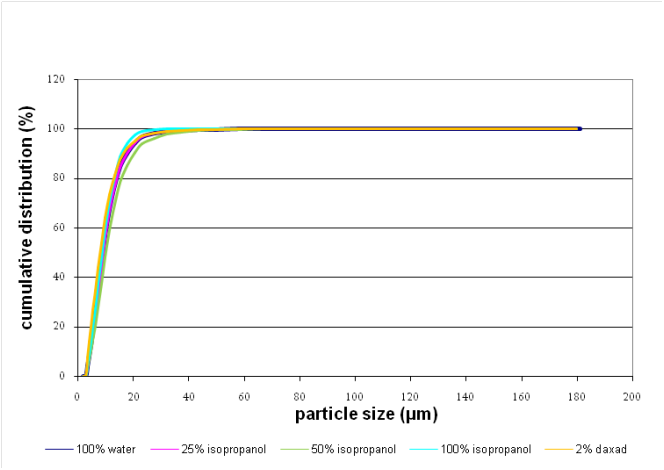

Figure 6 Effect of solvents on the measured particle sizes of Sb_2O_3.

Amount of sample: As shown in Table 4, the amount of sample is critical, with excess sample leading to agglomeration.

Table 4 Effect of sample amount on the effect of agglomeration of TiO_2

Sample amount (g)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
0.5	(0.17)	0.25	0.73
1	(0.19)	0.30	0.80
2	(0.22)	0.36	0.85
4	(0.22)	0.43	0.90

Mixing: Common laser diffraction equipment provides mechanical and ultrasonic mixing. The problems of non-appropriate use of the ultrasonic tip have already been described in Section 2. It is very important to maintain the amount of solvent in the bath at a required level, since mechanical and ultrasound mixing are affected by the liquid volume. The effect of sonication output power and duration are illustrated in Figure 7, Figure 8. Neither output power, nor duration of sonication has a significant effect within the applied range.

Particle refractive index: A very important parameter when using laser diffraction is the particle refractive index. For samples of µm order or below, the refractive index influences the results considerably as depicted in Figure 9. It is therefore very important to make sure that the refractive index of the finer samples does not alter significantly.

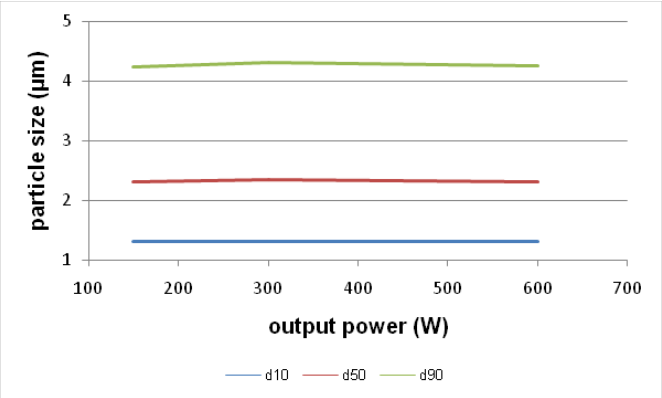

Figure 7 Influence of sonication output power (laser diffraction of Al_2O_3).

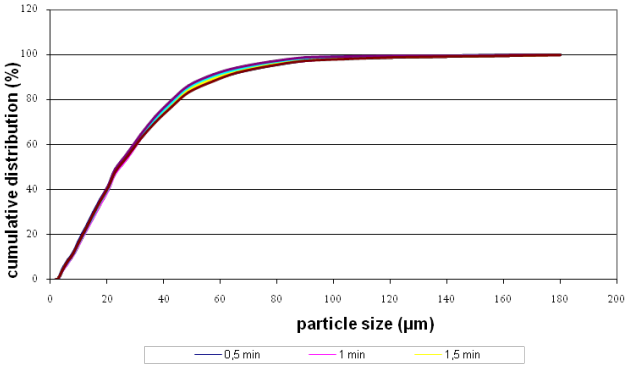

Figure 8 Influence of duration of sonication on the measured particle size.

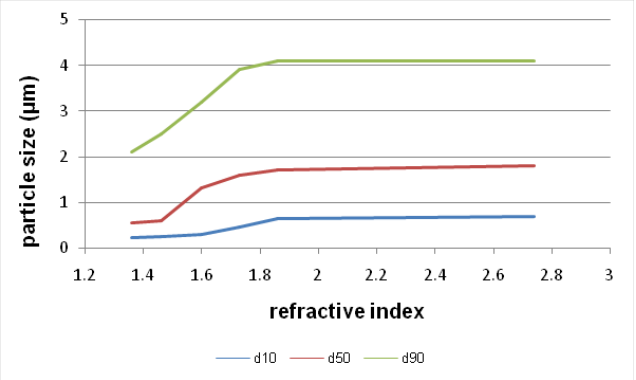

Figure 9 Effect of the refractive index on the size distribution of Al_2O_3 (laser diffraction).

Presenting particle size distributions: The particle size distribution is usually presented as a histogram with the particle size on the linear or log-normal x-axis, and the fraction of each particle size range on the y-axis. This log-normal x-axis is used for the wide size-range, poly-disperse distributions. This y-axis fraction can be expresses either as a weight of particles (weight basis) or as a surface area of the particles (area basis) or as number of particles (number basis). Statistically defined particle size distributions are usually normalized and the total area of the histogram equals 1.

Each technique has its own basis where it yields the most reliable data. Most popular macroscopic methods (light scattering, acoustics) and fractionation methods (sedimentation, sieving, centrifugation) present data usually on a weight (same as volume) basis, on a

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logarithmic scale and as normalized distribution. If particle size distributions measured by different instruments, even by different methods, are expressed on the same basis and scale, then results are quite comparable, even for very broad particle size distributions.

Comparing common particle size analysers

Different particle analysers were compared for 2 powders, i.e. Al_2O_3 and SiC. The properties of both powders are given in Table 5.

The average values of 10 particle size measurements and the coefficients of variation are summarized in Table 6. As shown, the coefficients of variation of d_{50} measured by X-ray sedimentation, light obscuration and electrical sensing zone method are less than 10%. Photo-sedimentation has the worst relative accuracy. The coefficients of variation of d_{50} of the laser diffraction method are less than 20%.

Table 5 Sample properties and optimum dispersants

	SiC	Al_2O_3
Absolute density (kg/m³)	3130	3960
Specific surface area (m²/g)	20.5	1.9
Refractive index (-)	2.65	1.76
Dispersant and concentration (wt %)	Tri-sodium phosphate 0.025	Sodium Hexametaphosphate 0.05
ξ -potential (mV)	-64	-97.5

Table 6 Comparison of results for Al_20_3 and SiC

Al_20_3	X-Ray sedimentation	Photo-sedimentation	Light obscuration	Electrical sensing zone	Malvern
d_{10} (µm)	(0.95)	(0.95)	1.16	1.16	0.71
C_V (%)	2.8	14.2	5.8	8.3	35.9
d_{50} (µm)	1.81	1.69	2.88	2.16	2.1
C_V (%)	3	12.6	7.2	4.8	12.7
d_{90} (µm)	3.68	4.13	4.89	4.07	4.69
C_V (%)	5.2	41.8	3.2	4.6	9.6
SiC	**X-Ray sedimentation**	**Photo-sedimentation**	**Light obscuration**	**Electrical sensing zone**	**Malvern**
d_{10} (µm)	(0.11)	(0.16)	0.63	(0.20)	(0.24)
C_V (%)	(15.20)	(27.20)	3.5	(21.30)	34.5
d_{50} (µm)	0.47	0.47	1.02	0.68	0.64
C_V (%)	21.7	39.4	6.9	10.4	18
d_{90} (µm)	1.92	1.6	3.12	2.71	1.96
C_V (%)	10.8	34.7	17.7	14.5	31.2

Conclusion

The accuracy of the particle size analysis is largely dependent on a number of parameters, including the sample preparation, the degree of dispersion and the analysis itself. In most common suspension analysers, particles are dispersed in a solvent, mostly using a dispersant to lower the surface tension.

The use of organic solvents can be necessary for water soluble powders. Alcohols, acetone or even aromatics can be used (as function of the density of the powders). The results demonstrate that the type of solvent used affects the experimental results.

The amount of sample is critical, with excess sample leading to agglomeration. Common laser diffraction equipment provides mechanical and ultrasonic mixing. It is very important to maintain the amount of solvent in the bath at a required level, since mechanical and ultrasound mixing are affected. The effect of sonication output power and duration is negligible, within the applied test range.

A very important parameter when using laser diffraction is the particle refractive index, certainly for samples of µm order or below. It is therefore very important to make sure that the refractive index of the finer samples does not alter significantly.

If particle size distributions measured by different instruments, even by different methods, are expressed on the same basis and scale, then results are quite comparable, even for very wide particle size distributions.

The average values of 10 particle size measurements and the coefficients of variation demonstrate that the coefficients of variation of d_{50} measured are fair and mostly acceptable. Photo-sedimentation has the worst relative accuracy.

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Conflict of interest

The author declares no conflict of interest.

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