A comparison of automated scanning electron microscopy (ASEM) and acoustic attenuation spectroscopy (AAS) instruments for particle sizing

By: Clint P. Aichele, Deepika Venkataramani, James E. Smay, Melinda H. McCann, Scott Richter, Mehdi Khanzadeh-Moradllo, Mohammed Aboustait, M. Tyler Ley

This document is the Accepted Manuscript version of a Published Work that appeared in final form in *Colloids and Surfaces A: Physiochemical and Engineering Aspects*, copyright ©Elsevier B.V. after peer review and technical editing by the publisher. To access the final edited and published work see http://dx.doi.org/10.1016/j.colsurfa.2015.03.052.

Abstract:

The objective of this work is to compare particle size distributions (PSD) measured by automated scanning electron microscopy (ASEM) and acoustic attenuation spectroscopy (AAS) instruments at Oklahoma State University (OSU). The techniques provide quantitative PSD measurements of dispersed systems of both hard and soft particles. Glass spheres, fly ash, and latex spheres are chosen to illustrate the range of materials that can be analyzed with these techniques. The variety of length scales and material property attributes investigated provides some insight about the consistency of the ASEM and AAS measurements for these types of particles. Although both instruments yield internally consistent PSD, rigorous statistical analysis shows that the two instruments can yield different results when measuring samples from the same batch of particles. The difference is not surprising given the different physics of each instrument. Nevertheless, the similarity in PSD hints at the validity of comparing absolute PSD and PSD changes.
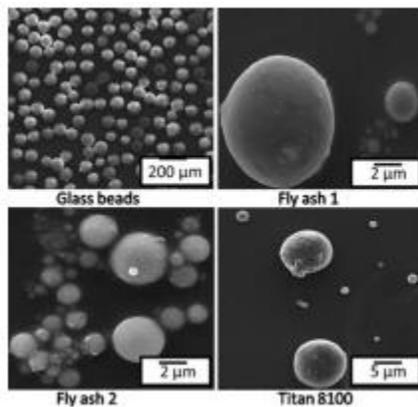
Article:

HIGHLIGHTS

- Two independent particle sizing instruments yield similar particle size distributions.
- Particle size distributions in the vendor specified range for standard materials.
- Statistical comparisons demonstrate importance of comparing statistical significance.

1. INTRODUCTION

Particle size distribution (PSD) measurements are important to understand the repeatability and efficiency of different industrial processes and products. Techniques to measure PSD rapidly and accurately are critical tools for these industries. A wide variety of techniques such as dynamic light scattering (DLS), X-ray scattering (XAS), transmission electron microscopy (TEM), and scanning electron microscopy (SEM) are available for particle characterization [1]. Attenuation acoustic spectroscopy (AAS) and automated scanning electron microscopy (ASEM) are relatively new techniques to quantitatively characterize polydisperse particulate systems [1] and [2].

AAS can characterize concentrated disperse systems in situ, is tolerant of contaminant species, is non-destructive, and uses industrially relevant liquid to solids ratios [1] and [3]. Therefore, AAS has good potential to be an inline, process monitoring tool. AAS measures the attenuation of ultrasound waves as they pass through a medium. The attenuation is a function of frequency, dispersion medium properties and the dispersed phase particle size distribution. Hence, upon measuring the attenuation spectra, the particle size is calculated based on an attenuation model [1]. Therefore, particle sizing by AAS might be considered an indirect measurement as it depends on fitting of model parameters. Since AAS requires a suspending medium, it may be the wrong technique to select if the research question involves the behavior of dry powders.

ASEM observes a field of particles deposited on a conductive substrate in a scanning electron microscope. A computer algorithm is used to recognize individual particles during scanning. Measurements of the diameter, shape factor and chemistry are made. The algorithm quantifies the percentage of particles counted in each size range and assembles a probability density function. While ASEM provides a wealth of knowledge about individual particle chemistry, size, shape, and aspect ratio, it is not amenable to inline process monitoring for particles in suspension. ASEM has proven particularly useful for understanding fly ash size distribution and phase distribution and local chemistry [2], [4], [5], [6], [7] and [8]. When performed accurately for dry particles, ASEM may be considered a direct measurement technique, but may be considered an indirect technique if the research question is to understand the particle size distribution in suspension.

Dry versus wet observation of the particles aside, when comparing particle size distributions measured by ASEM and AAS machines, a statistical test can reveal the answer to the question:

"Are these measured distributions substantially different?" Equivalently, an appropriate confidence interval can be examined. Confidence intervals provide additional information and allow the researcher to not only assess statistical significance, but also practical significance. Consequently, for most situations, researchers rely on parametric tests and confidence intervals to assess results. However, because the distribution of the data is not always well known, a common parametric analysis is not always appropriate [9]. The situation in this study is one where the appropriate distribution of the test statistic is unknown, so we rely on permutation tests and associated confidence intervals for an appropriate analysis.

The quantitative difference between particle size distributions measured by different machines or techniques is often of little concern if one is operating in a quality control environment, where internal consistency and repeatability of the chosen technique is sufficient. However, when process and resultant size distribution changes occur, it is useful if the chosen technique can quantitatively measure how the distribution has changed. This paper asks a different research question: If the particles from a common batch are measured by a microscopy technique and an acoustic attenuation technique, at what level of certainty can one compare the quantitative description of particle size? The comparison presented here between ASEM and AAS results for a variety of particles seeks to investigate this question with statistical hypothesis testing of the measured distributions and to show the applicability of both techniques. A comprehensive comparison between the "techniques" of ASEM and AAS is beyond the scope of this research. Rather, this work is limited to a set of commercially available materials sampled from common batches. The study is further limited in scope to only one instrument for each technique. While the scope is limited, such inter-lab comparison of particle size data is informed by a careful initial study with seemingly simple materials.

2. MATERIALS AND METHODS

2.1 Materials

A range of particle morphologies including large (~40 μm) hard, small (~1 μm) hard, and small (~1 μm) soft were selected to evaluate the capabilities of the instruments. For large hard particles, glass beads (Polyscience Inc.) were selected. The manufacturer specified density is equal to 2.5 g/cm^3 and the vendor specified mean diameter is in the range 30–50 μm. The glass bead manufacturing process produces a low concentration of irregularly shaped particles and small debris; thereby, yielding primarily spherical geometries with the possibility of surface imperfections.

For small hard particles, two types of fly ash were investigated. Both fly ash types are categorized as class C according to ASTM C 618 standards. Class C fly ash is typically produced from burning sub-bituminous coal, and it is widely used in the concrete industry due to its self-cementing properties. Table 1 contains metal oxides in each of the fly ash samples according to the ASTM C 618 classification and the bulk density was equal to 2.6 g/cm^3.

Table 1.
Chemical composition of fly ashes.

Component	Fly Ash 1	Fly Ash 2
SiO_2 (%)	37.50	36.21
Al_2O_3 (%)	19.67	19.94
Fe_2O_3 (%)	6.28	6.67
CaO (%)	23.65	23.96
Na_2O (%)	1.63	1.67
K_2O (%)	0.60	0.52
TiO_2 (%)	1.46	1.44
MnO_2 (%)	0.02	0.03
P_2O_5 (%)	1.11	1.44
SrO (%)	0.37	0.38
BaO (%)	0.68	0.69
SO_3 (%)	1.20	1.44
MgO (%)	5.20	5.17
L.O.I (%)	0.64	0.15
Moisture (%)	0.03	0.04

For small soft particles, an acrylic latex particle (Elotex Titan 8100, Akzo Nobel Inc.) was selected. Titan 8100 is manufactured as a spray dried granule with manufacturer specified bulk density equal to 0.45–0.65 g/cm^3 and glass transition temperature $T_g = 10$ °C. The particle density was measured to be 1.1 g/cm^3 and the bulk density was equal to 2.6 g/cm^3. Titan 8100 is often used as a binder in the paint and coatings industries and is dispersible in water [10].

The dispersion mediums in this work were: deionized water with a resistivity of 18.2 MΩm and absolute ethanol (200 proof) with purity equal to 99 wt.%. The ethanol was produced by BDH and purchased from Sigma–Aldrich (CAS # = 64-17-5). The fly ash particles were suspended in ethanol while the Titan 8100 and glass beads were suspended in deionized water. Ethanol was used for the fly ash as they are reactive with water.

2.2. Measurement techniques

2.2.1. Acoustic attenuation spectroscopy (AAS)

An acoustic spectrometer (Dispersion Technology Inc., DT-1202) was used to quantify the particle size distributions [1]. The DT-1200 produces monochromatic ultrasound over the frequency range 1–100 MHz. The intensity of the acoustic wave transmitted to the detector through the sample compared to reference of the dispersion medium is used to determine the attenuation associated with the dispersed phase [1]. The DT-1200 measures 18 discrete frequencies in the 1–100 MHz range. The distance between the transmitter and detector is varied in 21 steps at each frequency to compile an accurate measure of linear attenuation coefficient as

a function of frequency for the dispersion. The typical measurement time for one sample is approximately 10 min.

The experimentally measured attenuation spectrum is compared to a theoretical model using the particle size distribution as an adjustable parameter [1]. The model attenuation is calculated (Eq. (1)) as the sum of viscous, thermal, structural, scattering and intrinsic loss mechanisms [1].

$$\alpha_{total} = \alpha_{thermal} + \alpha_{scattering} + \alpha_{structural} + \alpha_{intrinsic} \tag{1}$$

The innate basis for the AAS technique is weight, so the particle size distributions calculated by AAS are presented on a weight basis. The technique has been developed to yield an overall weight of particles for a given size. The innate basis of the ASEM technique is number, so the particle size distributions calculated by ASEM are presented on a number basis. For the purposes of comparison to the ASEM instrument, the weight basis particle size distributions of AAS were converted to number basis particle size distributions using the algorithm provided by the Dispersion Technologies, Inc. software. For the AAS measurements, ISO standards 9276-1, 9276-2, and 9276-5 were employed by the Dispersion Technologies, Inc. software for calculating the particle size distribution on a weight basis and converting it to number basis [11], [12] and [13]. Due to this fundamental difference between the AAS and ASEM technologies, for polydisperse dispersions, there may be bias in the results. The AAS technology may be bias with regard to small particles as they will not attenuate as readily as the larger particles. However, the ASEM technology may be bias with regard to finding large particles that are present in very small number. This difference is critical to consider when choosing the correct particle sizing technique for a given application.

The attenuation of each sample depends on the individual material properties. Attenuation of soft, small particles (i.e., latex) is dominated by thermal losses. For small, hard particles, such as fly ash, viscous losses contribute most to attenuation. For large, hard particles, scattering becomes a significant contributor to attenuation [1]. The materials chosen in this study specifically target these three contributions of acoustic attenuation and probe the accuracy of the result relative to the material independent results of the ASEM.

For each AAS measurement, 200 mL of the sample flows through the sample chamber using a Masterflex peristaltic pump. The instrument is equipped with a zeta potential probe, pH probe, temperature probe, and aqueous/non-aqueous conductivity probes.

2.2.2. Automated scanning electron microscopy (ASEM)

An FEI-ASPEX PSEM Explorer with a tungsten filament and a SDD EDS detector was used in this study. The instrument works as a scanning electron microscope with an image processing based operating system. The system uses the contrast between the back scattered (BS) data from the particles of interest and the substrate to find and measure the particles. In addition to counting the number of particles and measuring their shape and size, this instrument is also capable of characterizing chemical composition of the particles through EDS. The instrument is automated to find, measure, and count the number of particles that are dispersed on carbon tape. The system can use either pre or post processing to examine particles with a given chemistry,

shape, or size. This is very useful for complex industrial materials and mixtures of materials as it can show the chemical complexity of the different particles and their size range. The ASEM takes roughly 5 s per particle to measure morphology and chemistry but less than 1 s per particle if only measuring morphology. More details can be found in other publications [2] and [7].

2.3. Sample preparation

For AAS measurements that involved Titan 8100, the dispersions were formed by suspending 4.8 vol.% Titan 8100 (5 wt.%) in deionized water using a sonic dismembrator (Fisher Scientific Sonic Dismembrator Model No. 500) for 5 min at 40% power. For AAS measurements that involved fly ash, 5.0 vol.% fly ash was suspended in ethanol (200 proof) using a Ultraturrax T25 homogenizer at a speed equal to 5000 rpm for 5 min. For AAS measurements that involved hard glass beads, 10 wt.% glass beads were suspended in deionized water, and the sample was measured in situ in the acoustic spectrometer.

For ASEM measurements, 50 mL of the dispersion was prepared using a sonicator (GB-928 Ultrasonic Cleaner). A drop of the sample from the 50 mL batch was placed on carbon tape and dried at room temperature and atmospheric pressure. This procedure is commonly utilized for ASEM measurements, and it was used for all materials.

3. RESULTS AND DISCUSSION

Fig. 1 provides comparisons between the AAS and ASEM techniques for one representative distribution for each sample type. Note that only one representative distribution is shown so that the general behavior of the methods can be easily observed with a relatively smooth figure. The particle size distributions shown in Fig. 1 are on a number basis. The ASEM technique directly yields a particle size distribution based on the number of particles measured. The number basis particle size distribution as calculated by AAS is plotted in comparison to the ASEM results in Fig. 1a for the hard, glass bead system. ASEM yielded a larger mean value than AAS and reveals a bi-modal PSD. The effect of the residual debris on the particle surface from the manufacturing process (observable in Fig. 2a) may cause an artificially large diameter reading on ASEM; although a rigorous investigation of this was not performed. We hypothesize that this surface debris may sometimes detach and be measured as the small peak in the ASEM PSD. Likewise, in AAS, detachment of the surface debris may cause a smaller average particle size when the attenuation data is fit with a uni-modal model as done here. The AAS offers a bi-modal fitting option, but the uni-modal fit produced less error. Despite the differences exhibited in Fig. 1b, the mean sizes measured by both ASEM and AAS are within the vendor specified range (30–50 μm).
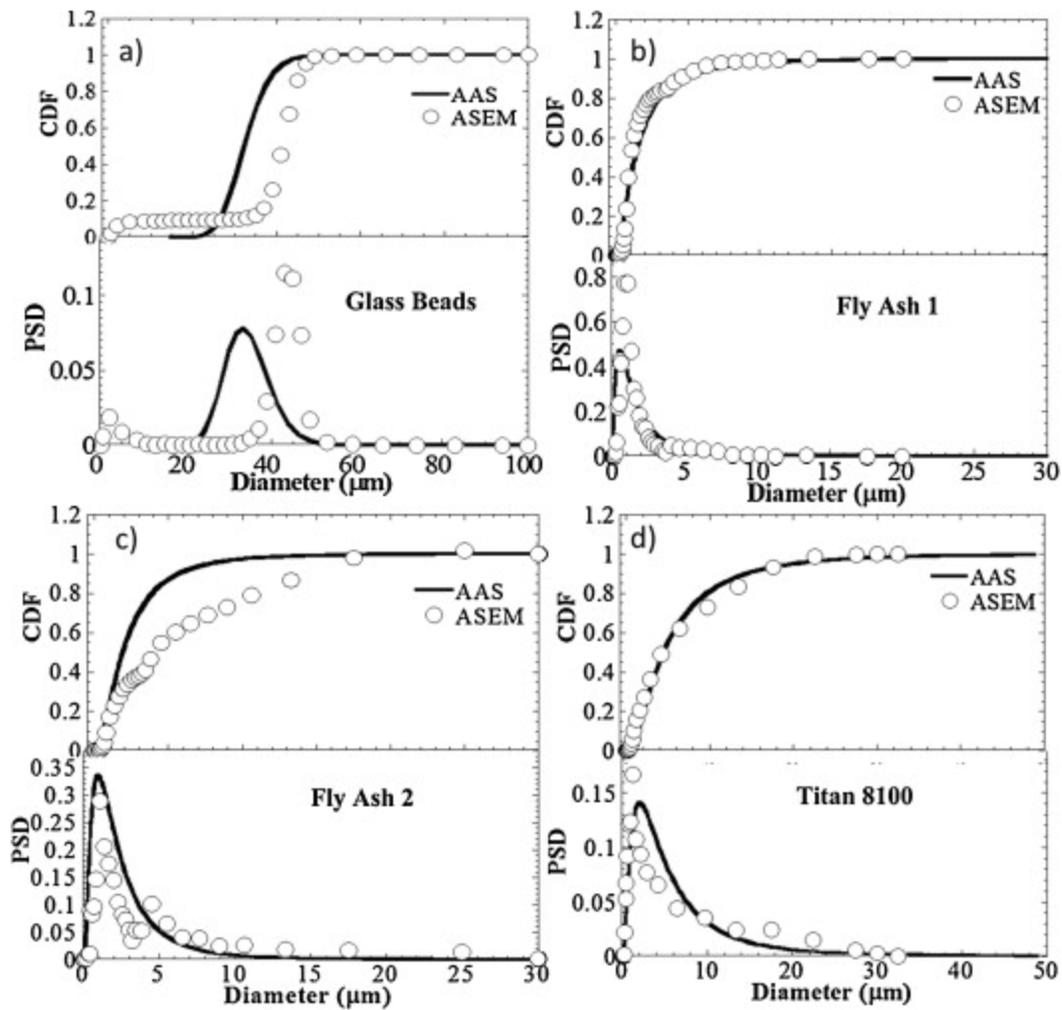
Fig. 1.
Comparison of representative particle size distributions (PSD) and cumulative distribution functions (CDF) on a number basis for (a) glass beads, (b) Fly Ash 1, (c) Fly Ash 2 and (d) Titan 8100.
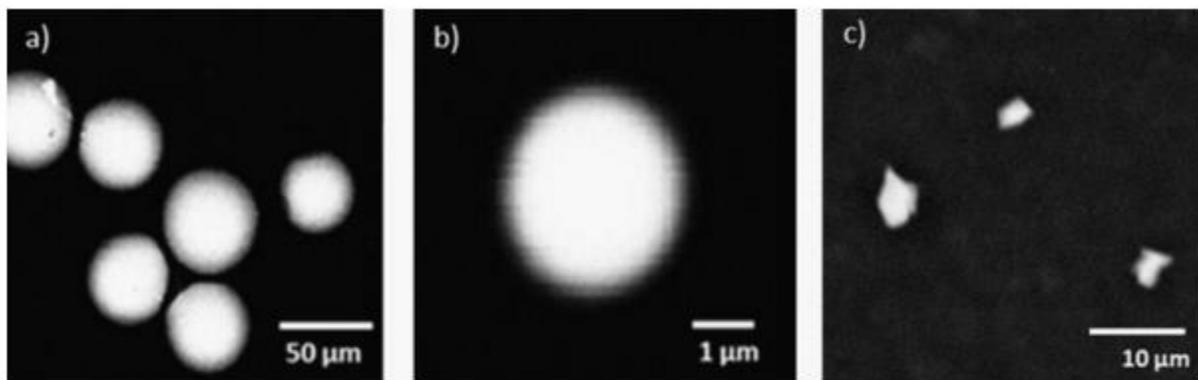

Fig. 2.
ASEM images of (a) glass beads, (b) Fly Ash 1, and (c) Titan 8100 using backscattered electron imaging.

Fly Ash 1 and 2 are shown in Fig. 1b and c, respectively. The figures show that there is general agreement between the two data sets and the means are similar. One major difference is that the shape of the PSD is different near the mean, particularly for the polydisperse Fly Ash 2. The ASEM data suggests that the PSD is more complex than the unimodal model assumed by the AAS technique. For Fly Ash 2, the ASEM showed a bimodal distribution at a size of 2 and 5 μm; however, a single peak provided the best fit for AAS. Despite this, the means and size distributions for the measurements were similar. For the Titan 8100 investigated in Fig. 1d, both methods yielded similar results for the mean.

The CDF and PSD measured by our ASEM and AAS instruments for each material type is quantified and compared based on estimated means, variances, and distribution shapes. When considering the comparison of the estimated means because there are only 3 or 6 repeat measurements for the two methods, we do not have enough data to justify performing a t-test. However, the t-test would also be valid if the observations themselves were normally distributed. Note that the estimated mean of each material is based on many observations (i.e., measurement of 2000 particles for ASEM and the attenuation of an even larger number of particles for AAS), but these are not strictly independent. Consequently, normality of the sample mean is not assured. Thus, to be conservative, we performed permutation t-tests [9] to compare the estimated means of the two instruments separately for each of the four materials. The permutation t-test does not require any distributional assumptions on the data, unlike the t-test, and it is also easily invertible to obtain confidence intervals. However, note that the observations contributing to each sample mean are likely to be nearly independent. Consequently, the t-test results are also provided for comparison, and the results are given in Table 2.

Table 2.
Comparison of the estimated means of the four materials.

Sample	Instrument	Average mean (μm)	Permutation t-test p-value	t-test p-value
Glass beads	AAS	32.41	0.012	<0.0001
	ASEM	40.80	(−13.45, −3.33)*	(−9.71, −7.07)*
Fly Ash 1	AAS	0.91	0.024	0.0085
	ASEM	1.28	(−0.73, −0.02)*	(−0.60, −0.14)*
Fly Ash 2	AAS	1.38	0.012	<0.0001
	ASEM	2.76	(−2.17, −0.61)*	(−1.56, −1.22)*
Titan 8100	AAS	2.71	0.786	0.7003
	ASEM	2.57	(−0.73, 1.02)*	(−0.71, 1.01)*

* Respective 95% confidence intervals for the mean difference, AAS-ASEM (μm).

Note that the means for the two instruments are not statistically significantly different for Titan 8100. Thus disagreement between the two instruments for this material with respect to the mean is not indicated. However, the means are significantly different for the other three materials. However, statistical significance does not always result in practical significance, as with enough data even extremely small differences from the null hypothesis (i.e., the estimated means are different) will generally be assessed as statistically significant. In this case, many of the

estimated means are very similar, and while statistically significant, this difference may not be of practical significance to many. However, if a question revolves around the production or measurement of narrowly separate particle populations between samples, the difference between these two techniques may be vital. Here, we simply state the differences between these two specific instruments in a rigorous statistical treatment of the limited measurements to understand their magnitude.

To allow individuals to assess the practical significance of the differences, 95% confidence intervals for the mean differences were computed (arbitrarily AAS versus ASEM), and these are provided under the respective p-value in parentheses in Table 2. Note that the mean difference for Fly Ash 1 is relatively small, while the mean difference for glass beads is quite large. The 95% confidence intervals in Table 2 illustrate that the range of the difference of the means between the two techniques can be large, but still within the range of practical applicability as both techniques yielded mean diameters in the vendor specified range. Depending on the application, practical applicability can mean operating within a broad range consistently, or it can mean operating within a narrow range consistently. The results from each technique show the variability that can exist when using two different techniques to characterize the same system. The practitioner must decide on the most appropriate technique for their application based on several factors including accuracy, repeatability, throughput, and reliability, among others.

The variances were also compared via statistical analysis. As variance estimates are not normally distributed (even under independence), and the number of estimated variances is again either 3 or 6, we have the same issues performing a t-test as for the mean comparisons. Consequently, permutation t-tests were again utilized, although the standard t-test results are again provided for comparison. Both the test results and the respective confidence intervals for differences are provided in Table 3.

Table 3.
Comparisons of the standard deviations for the four samples considered in this work.

Material	Instrument	Average std. dev. (µm)	Permutation t-test p-value	t-Test p-value
Glass beads	AAS	4.93	0.012	<0.0001
	ASEM	11.67	(−10.88, −2.58)[*]	(−7.99, −5.47)[*]
Fly Ash 1	AAS	1.14	0.75	0.66
	ASEM	1.1	(−0.15, 0.23)[*]	(−0.17, 0.25)[*]
Fly Ash 2	AAS	1.49	0.012	<0.0001
	ASEM	3.25	(−2.71, −0.8)[*]	(−1.87, −1.64)[*]
Titan 8100	AAS	3.12	0.036	0.0002
	ASEM	4.18	(−2.01, −0.10)[*]	(−2.02, −0.09)[*]

[*] Respective 95% confidence intervals for the mean difference, AAS-ASEM.

Note that the standard deviations for the instruments are not statistically significantly different for Fly Ash 1. Thus, disagreement between these two instruments is not indicated. However, the standard deviations are significantly different for the other three materials. To assess practical

significance, the confidence intervals are provided. All of the variances of the materials are different except for Fly Ash 1. The likely explanation for the statistical agreement for Fly Ash 1 is that this particular system did not exhibit significant surface debris or irregularities; therefore, ASEM and AAS yielded similar results. Likewise, fly ash is easily dispersed in water for the AAS measurement and prepared as a dry powder for ASEM observation; and, fly ash is known to have a fairly narrow PSD.

Finally, the difference in the estimated density functions was assessed via a statistical test. Unfortunately, the standard statistical tests to compare density functions compare two independent density functions instead of two independent samples of density functions. Additionally, we do not have strictly independent observations within each estimated density function. Consequently, we rely on a permutation test for a difference in the estimated distribution functions, as permutation tests require no additional assumptions for validity. The Kolomogorov–Smirnov test statistic is often used to test for a difference between two distributions, the null hypothesis being that the two distributions are the same. The test statistic is given by Eq. (2):

$$KS = \max_z |\hat{F}_1(x) - \hat{F}_2(x)| \qquad (2)$$

where the two estimated cumulative distribution functions are given by $\hat{F}_1(x)$ and $\hat{F}_2(x)$, respectively. Unfortunately, this test does not accommodate multiple estimated cumulative distribution functions. Consequently, we computed this test statistic for each possible pair of observations where the first estimated distribution function is from the AAS technique and the second estimated distribution function is from the ASEM technique. The final test statistic value is then the average of these values for all possible AAS-ASEM pairs. A permutation test was then performed. This was accomplished by permuting the observed estimated distribution functions to the two respective groups randomly and calculating the value of the test statistic for all possible permutations. These values were then combined to obtain a reference distribution which was utilized to determine the p-value for the appropriate test. The results are given in Table 4.

Table 4.
Estimated p-values for the permutation test for the distribution functions (H_0: the distribution functions are equal).

	Material			
	Glass beads	Fly Ash 1	Fly Ash 2	Titan 8100
p-Value	0.0001	0.7152	0.0001	0.0001

Statistically, these results indicate that the distribution functions are significantly different for all but Fly Ash 1. This corresponds with the visual observations made about the graphs given in Fig. 1. Note that the two estimated distribution functions for Fly Ash 1 are almost identical. For the other three systems, the estimated distribution functions have differences, and even though for some of the systems these differences are minimal, they are still statistically significant.

Based on the totality of the three statistical tests employed, it appears that the two instruments evaluated are performing relatively the same for Fly Ash 1, although the means are statistically significantly different. However, the scale of the difference is not large. For the other materials, the three statistical tests indicate that the means, standard deviations, and distribution functions of the instruments are statistically significantly different, with the exception of the means for Titan 8100. Despite these differences, both instruments yielded mean diameter values for glass beads that were within the vendor specified range.

4. CONCLUSIONS

Calculated particle size distributions based on measured AAS and fitting of an attenuation model for a variety of materials was investigated and compared to the PSD for particles from the same batch as measured by ASEM. Internally, each particle sizing technique provides a repeatable measure of PSD for both hard and soft particles over a range of sizes. In the case of narrow, unimodal particle size distributions (Fly Ash 1 and Titan 8100), both instruments yielded similar distributions. Fly Ash 1 exhibited the best agreement among all the materials. This particular fly ash is known to consist of nearly spherical, hard particles with fairly narrow, unimodal particle size distributions that are ideal for both ASEM and AAS. This agreement underscores the utility of both instruments, as the two instruments use innately different physics to arrive at the particle size distributions.

For the broad, polydisperse systems, the mean diameters of the distributions are comparable, but the shapes of the particle size distributions for each instrument were different. Despite these differences, both instruments yielded mean diameters that were within the vendor specified range for the reference material (glass beads) that was used in this work. The ASEM measured subtle variations in the PSD that were not apparent in the AAS PSD. Since the AAS instrument uses a unimodal fit to the measured acoustic attenuation data, such subtleties may be hidden in the calculated PSD. Further investigation is required to determine if these effects can be observed in the raw attenuation data. For the scope of our study, the ASEM and AAS can be considered as complimentary measures of our particle systems. Questions of suspension behavior and PSD in suspension seem to be well-suited to AAS while detailed analysis of particle shape, chemistry, and PSD in the dry state are well-suited to the ASEM.

No statistical agreement was achieved for Fly Ash 2, likely because of the complex shape of the size distribution for that particular fly ash. The latex spheres (Titan 8100) exhibited statistical agreement in the mean diameters, but the standard deviations and distributions were statistically different.

AAS is an attractive technique for online process monitoring and quantitative PSD measurement under realistic process conditions. However, this paper highlights the challenges of using indirect methods for characterizing PSD of complex particles where imperfections or non-idealized distributions may impact the results. Through rigorous statistical comparisons, this paper also highlights that over a range of material types and properties, the two independent instruments can yield similar size information for each system, though not always statistically identical.

While there were statistical differences in many of the measurements, it does not mean that the inter-comparisons between AAS and ASEM results are not practically useful. For many industrial applications, it is critical to rapidly measure changes that occur with particles. Since AAS was shown to be repeatable, the method should be capable of measuring this. Also, if measurements must be made in solution, then AAS is a powerful tool for quantifying the particle size distributions. However, potential users should be cautious of staking critical process decisions on the absolute value of the PSD measured by AAS without first understanding the differences that might be present when comparing to a direct technique such as ASEM.

ACKNOWLEDGEMENTS

REFERENCES

[1] A.S. Dukhin, P.J. Goetz
Characterization of Liquids, Nano- and Microparticulates, and Porous Bodies Using Ultrasound
(2 ed.)Elsevier (2010)

[2] M. Aboustait, Q. Hu, B. Tabb, R. Frazier, Y. Zhang, T. Ley, J. Hanan
Innovative prediction of Fly Ash performance in concrete
Oklahoma Transportation Center Report (2013)

[3] A.S. Dukhin, P.J. Goetz
Characterization of aggregation phenomena by means of acoustic and electroacoustic spectroscopy
Colloids Surf. A: Physicochem. Eng. Aspects, 144 (1998), pp. 49–58

[4] S. Ghosal, J.L. Ebert, S.A. Self
Chemical composition and size distributions for fly ashes
Fuel Process. Technol., 44 (1995), pp. 81–94

[5] C.F. Ferraris, V.A. Hackley, A.I. Aviles
Measurement of particle size distribution in Portland cement powder: analysis of ASTM round robin studies
Cement Concr. Aggreg., 26 (2004)

[6] A. Sarkar, R. Rano, K.K. Mishra, I.N. Sinha
Particle size distribution profile of some Indian fly ash—a comparative study to assess their possible uses

Fuel Process. Technol., 86 (2005), pp. 1221–1238

[7] W.P. Linak, C.A. Miller, W.S. Seames, O.L. Wendt, T. Ishinomori, Y. Endo, A.S. Miyamae
On trimodal particle size distribution in fly-ash from pulverized coal combustion
Proc. Combust. Inst., 29 (2002), pp. 441–447

[8] M. Aboustait, T. Kim, M.T. Ley, J. Davis
Physical and chemical characteristics of fly ash using automated scanning electron microscopy
Fuel (2015) (Submitted for publication)

[9] P.I. Good
Permutation Tests: A Practical Guide to Resampling Methods for Testing Hypotheses
(2nd ed.)Springer (2000)

[10] K. Kendall, J.C. Padget
Latex coalescence
Adhes. Adhes., 2 (1982), pp. 149–154

[11] ISO 9276-1: 1998 (E) Representation of results of particle size analysis Part 1: Graphical representation.

[12] ISO 9276-2: 2014 (E), Representation of results of particle size analysis Part 2: Calculation of average particle sizes/diameters and moments from particle size distribution.

[13] ISO 9276-5: 2005 (E), Representation of results of particle size analysis Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution.