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Original Research Paper

# Particle size analysis on wide size distribution powders; effect of sampling and characterization technique

G. Bahar Basim [a,*], Mohsen Khalili [b]

[a] Department of Mechanical Engineering, Ozyegin University, Cekmekoy, Istanbul, Turkey
[b] DuPont Central Research and Development, Wilmington, DE, USA

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ABSTRACT

Particle size distribution of powders plays a very important role in determining the critical chemical and physical properties of the particulate systems. Precise determination of particle size distribution depends on effective sampling of the powders, which is more pronounced for the particulate systems with a wide particle size distribution. Predominantly, the significant increase in the total surface area of the powders at nanometer scale particle sizes may lead to improper characterization of the bulk if the sampling technique fails to collect and represent them in the analyses. In this study, effects of sampling on the precision of particle size analysis are studied on a clay sample with a wide particle size distribution (particles ranging from nanometer to micrometer sizes) using light scattering technique in aqueous media. Three different sampling methods are applied to systematically analyze the effect of sampling on particle size measurements including; riffling the original sample into sixteen equal parts, sampling the powder after removing the very fine and very coarse size particles and riffling to sixteen parts and finally by riffling the powder to the exact feed amount of the particle size analyzer. The effectiveness of the applied methods were compared statistically by calculating the coefficient of variance (CV) values of the 10%, 50% and 90% passing particle size data of the sequential runs. The most effective sampling method was determined to be riffling the sample to the exact feed amount of the analyzer based on obtaining the minimum CV values of the measurements. In the second part of the study, results of size distribution analyses conducted by different particle size analyzers utilizing numerous characterization techniques are compared using the most effective sampling technique developed in the first part. It is observed that the use of different characterization equipment tend to result in variations in the particle size distributions of the same powder which presents another variability in classification of the wide particle size distribution powders.


## 1. Introduction

Particle size distribution of particulate systems is a critical variable for a number of industrial operations ranging from mining to pharmaceuticals. Precise measurement of particle size distribution of powders is important since the quality and performance of most powder-based products are closely related to the size distribution and particularly the concentration of the fine particles. Although the fine size fraction generally compromise of a small volume, the presence of fine particles leads to immense increase in the specific surface areas, creating a state of high surface energy that dominates properties of the selected system [1]. A good example is increasing moisture content in coal due to fine size content (−100 mesh) particles retaining more water molecules due to their large surface area that results in elevated filtration, drying and transportation costs [2]. Another critical example applies to microelectronics manufacturing, where the presence of slightly larger size particles at parts per million (ppm) levels in slurry made of nanometer size particles may result in significant surface deformation during the planarization of the wafers by the chemical mechanical planarization (CMP) process [3,4]. For pharmaceutical applications, where more than three-quarters of all tablets and capsules are manufactured using powder blends are more prone to sampling problems since the inaccuracy of sampling may directly affect the human health [5]. Food products, such as skim milk powder, are also hard to sample due to agglomeration during processing which skews the particle size distributions to larger ends and hence may not be representative [6]. Consequently, analysis of a representative powder sample is very necessary to obtain

* Corresponding author. Tel.: +90 216 5649344; fax: +90 2165649057.
*E-mail address:* Bahar.Basim@ozyegin.edu.tr (G.B. Basim).

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the correct size distribution of a particulate system in many industrial applications, which in turn helps in enhancing the process efficiency as well as the product performance.

The main objective of this study is to evaluate the effect of powder sampling on precision of the particle size distribution analyses and to delineate potential approaches for improving the sampling related variability in size distribution analysis of wide particle size distribution powders. The precision of particle size analysis strongly depends on the effectiveness of the selected sampling technique. In many industrial applications, as discussed above, sampling starts from a large amount of material, yet the collected sample powder must accurately represent this larger entity. Generally, gathering a representative sample with a very small volume is the primary challenge to particle sizing operations. It was reported that the quick and careless sampling of powder often results in an expensive study of an irrelevant or non-representative group of particles [7]. *Furthermore, it was also reported on a pharmaceutical application that not only the method of sampling but also the selection of proper sampling device is critical in sampling. Use of different sizes of the thieves for sampling the same drug powder was observed to make significant differences in the strengths of the pills produced* [8]. There are new technologies developed for the sampling of powders for delicate applications [9,10], methods that utilize digital imaging [11], in addition to methodologies for sampling large amount of materials (as in mining industry) [12] and many conventional instruments available for decades [13].

One of the most important factors in sampling powders for particle size analysis is whether the characterization will be conducted offline or online [14]. For most of the industrial applications, particle size analyses are conducted offline in the laboratories. After the powder sampled from the production line reaches the laboratory for particle size analysis, further sampling is required to decrease the amount of material to the suitable quantity for the selected particle size analyzer. In the case of online sampling of powders (which is conducted during processing), on the other hand, it is also important to check whether the collection of particles is required, or the information can be obtained by indirect methods. The amount of sample that must be collected should not be disturbing the production process while the cost of the sampling method in money and time, calibration and operational characteristics of the sampling system are additional critical factors to be considered [14]. Optical particle sizers provide robust characterization ability for online monitoring through their ability of covering a wide size range by changing the technique that they utilize through changing the optical configuration [15]. There are standards developed for sampling of powders for particle size analysis, which further verifies the importance of sampling to conduct accurate particle sizing analyses [16].

Determination of the most suitable sampling technique is driven by a number of different considerations including the total mass, number concentration, size distribution and chemical composition of the particles to be characterized in addition to the size distribution technique to be used [17–19]. When sampling powders, there are two types of sampling errors possible that can affect the particle size analysis [20]; (i) errors due to segregation of the bulk powder which can be prevented by suitable mixing and (ii) statistical errors that are due to random fluctuations and cannot be prevented. Both errors are more noticeable when the particle size distribution of the studied powder is wide and variable (changes due to agglomeration or due to breakage of the large size particles by attrition). While the first type of sampling errors can be minimized by suitable mixing and collecting a representative sample, statistical errors cannot be prevented since the quantitative distribution in samples of a given magnitude is not constant even for an ideal random mixture [20]. Yet, increasing the sample size helps estimate the statistical errors and reduce them. Hence it is important to statistically evaluate the results of particle sizing to understand the effect of sampling on precision of the characterization.

In this study, a clay sample with a significant variability in particle size was selected as a representative wide size range particulate system for offline characterization at the laboratory. Clays are well characterized for their colloidal properties in water based systems and are used in many industrial applications [21]. A detailed statistical approach was followed to evaluate the variations that sampling may create in particle size analysis. Three different sampling methodologies were implemented by riffling the main powder (i) to sixteen parts and preparing suspensions of these samples in water (ii) using the same procedure after the very fine and very coarse fraction of the powder is removed by screening to tighten the particle size distribution and, (iii) riffling the powder to the exact feed amount of the particle characterization equipment and preparing aliquots of the exact feed amount. The sampled powders were analyzed for particle size distributions at 10%, 50% and 90% passing values to determine the best sampling technique, which produces the most repeatable results in particle sizing. The particle size analyses were conducted using conventional sieve analysis, light scattering and number counting techniques. Initially, Coulter LS 230 (wet) and Microtrac Full Range Analyzer (FRA) instruments were employed to evaluate the best sampling technique. Next the most accurate sampling technique was used to compare the variability in the results of different particle size analyzers including, Coulter LS 230 (both dry and wet), Microtrac FRA (wet), Coulter Multisizer and Sympatec Helos (both dry and wet).

## 2. Materials and methods

### 2.1. Materials

A broad size distribution bentonite clay sample was selected to study the effect of sampling on the precision of the particle size analysis. The main components of the clay sample were $Al_3Mg_2$, $CaAl_2Si_2$, AlMg, $Al_3Mg_2$, $Al_2Mg$, $CaAl_2Si_{1.5}$, $Al_{0.58}$ $MgO_{0.42}$, Si, K and $Mg_2Si$ according to X-ray analysis which was consistent with calcium bentonite composition. Fig. 1 illustrates a scanning electron microscope (SEM) micrograph of the clay powder at 200× magnification on which the broad size distribution of the sample was detected clearly. The sieve analyses were also conducted on the powder, confirming the broadness of the size distribution of selected clay as demonstrated in Fig. 2. The fifty percent passing particle size ($d_{50}$) was calculated as 137.6 μm based on sieve sizing.

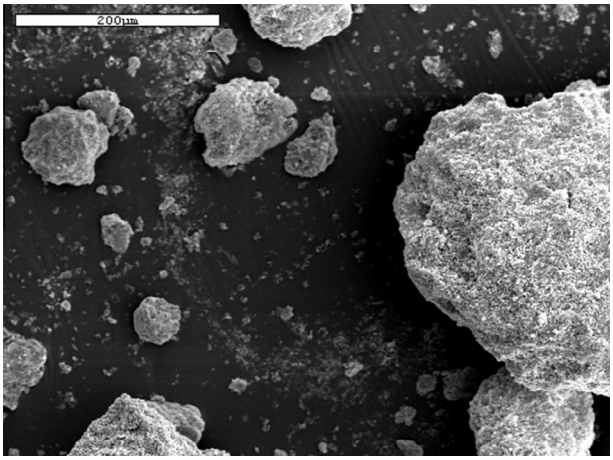


**Fig. 1.** SEM micrograph of the clay powder (Magnification = 200×).