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# Recent Advances in Particle Characterization †

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**Abstract:** Particle characterization is critical in industries that are influenced by particle size distribution. Understanding particle behavior is crucial for product quality control and manufacturing process optimization. Particle characteristics significantly affect material performance and properties. This review paper examines the importance of particle characterization in many industries and focuses on particle size and shape measurement. This paper begins by delving into particle size and size distribution analysis, emphasizing the impact of particle size on material properties and the many methodologies used for particle size analysis. This paper then examines particle shape characterization and its impact on material characteristics. It gives an overview of particle characterization techniques and the criteria for selecting the best technique for a given sample. Particle characterization in ceramics, food, cosmetics, medicines, and metallurgy are also thoroughly discussed. Overall, this work emphasizes the importance of particle characterization in numerous industries and provides insights into particle size and shape measurement.

**Keywords:** particle characterization; particle size distribution; particle shape



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## 1. Introduction

Particle characterization is highly utilized in industries with products affected by particle size distribution, such as food manufacturing, petrochemicals, pharmaceuticals, mineral science, etc. A particle is a discrete sub-portion of materials such as powders, granules, sprays, slurries, and more that can be solid, liquid, gas, or as molecular aggregations [1]. There are different sizes and shapes of particles. These particles can be differentiated by their diameters, ratio, solidity, sedimentation rate, etc., which describes the behavior of a particle. Given the difference, analysis of the material's measurable properties is significant in choosing the right instrument and technique of characterization.

Particle characterization is often employed in manufacturing and production industries as it allows for better product quality control, which benefits the competitive global economy. Furthermore, it gives a better understanding of the ingredients, appropriate processes, and the product that improves product performance, optimization of the process efficiency, and increased output, and helps troubleshoot manufacturing and supply issues. Analyzing the particle's behavior by characterization is critical for product quality control. It is essential to know which particle properties must be measured in particle characterization as a particulate material is dominated by the constituent particles' physical properties, which can influence a material's performance and properties, such as its chemical reactivity, dissolution rates, solubility, and abrasivity. In manufacturing industries, the significant properties often measured are the particle's size and shape, the surface, mechanical and charge properties, and the surface structure that can be seen microscopically. The easiest and most important two of the aforementioned properties are particle size and shape. Currently, numerous industries acknowledge the importance of measuring the particle's size and shape, especially in understanding the process that the material will undergo and the quality of the product depending on how its properties are affected or influenced by different factors.

## 2. Particle Size and Particle Shape

### *2.1. Particle Size and Size Distribution*

The sizes of materials differ based on the process or mechanism that generates particles. Classifying the range and distribution of particle sizes in plant and equipment design is essential to optimize the removal/recovery of particles or quality control. Particle sizes are usually measured in microns or nanometers and are designated as the particle's average diameter or average radius. Material qualities such as flow and conveying behavior, reactivity, abrasiveness, solubility, extraction and reaction behavior, taste, and compressibility are influenced by particle size distribution.

Particle size distribution analysis is a well-established practice in many laboratories. Various procedures are employed for this purpose, depending on the sample material and the extent of the examination. Optical methods, imaging, acoustic/ultrasonic, and electrical methods are some techniques used in particle size analysis. Typically, suspensions, emulsions, and bulk materials are evaluated.

Electrical techniques of particle size measuring, such as the Coulter Counter, have been in use for a long time, in which particles in a solution with conductivity pass separately through a small opening that separates each electrode of the sensor circuit [2]. The passage of each particle interrupts the electrical current running through the opening, and the resulting amplitude of the pulse provides a measure of the size of the particle. Another example of the particle size analysis technique is laser light scattering (LD), a widely used optical technique for determining particle size and distribution over a wide range of particle sizes. Also, dynamic light scattering (DLS) estimates particle size distributions using Brownian motion.

### *2.2. Classification of Particle Size*

Particle sizes are expressed as the average diameter of spheres of the same size. To calculate the molecular volume and size when the composition of molecular particles is known, we use the formula:

$$v_1 = \frac{M_1}{\rho_1 N_A} = \frac{\pi}{6} d_v^3 \Leftrightarrow \; d_v = 2r_v = 2\sqrt[3]{\frac{3v_1}{4\pi}} \tag{1}$$

where;

$d_v$ = particle volume diameter
$r_v$ = particle volume radius
$M$ = molar mass
$\rho_1$= particle density
$N_A$ = Avogadro's number

Represented in Equation (2) is the formula for calculating the average monomer equivalent surface area,

$$a_1 = 4\pi r_v^2 = \pi d_a^2 \; \Leftrightarrow \; d_a = \sqrt{\frac{a_1}{\pi}} \tag{2}$$

Also, the area per unit volume and projected 2-dimensional area is given as follows:

$$a_v = \frac{a_1}{v_1} = \frac{6d_a^2}{d_v^3} \tag{3}$$

$$a_{2D} = \pi r_{2D}^2 = \frac{\pi}{4} d_{2D}^2 = k_{2D} d_{2D}^2 \Leftrightarrow d_{2D} = \sqrt{\frac{4a_{2D}}{\pi}} \tag{4}$$

where $k_{2D}$ = shape coefficient, $d_{2D}$ = $d_a$ for convex particles.

The particle size properties of liquid, polymers, and minerals are based on the monomer's diameter that can be observed in Equation (1). By taking into account the available experimental techniques, it is possible to experimentally measure the volumes for gravimetry, area for gas adsorption, and the particles' parameters, and then use the Equations (1)–(4) to derive individual property dependent non-equal sizes. Due to broken bonds that cause monomers in the outermost layer to move further away from their equilibrium positions, these molecules have extraordinary electrical characteristics. The sum of the Gibbs free energy of a bulk particle and the surface energy can be used to calculate a unit volume's total energy, represented as Equation (5).

$$G_p = G_p^b + G_p^s = G_p^b + A_p \sigma_s \tag{5}$$

When the particle size is decreased, the contribution of irregular surface electrical and catalytic characteristics is increased [3].

### 2.3. Particle Shape

Particle shape is one of the factors that is easily considered in particle characterization, as it can impact the material's reactivity, solubility, efficient abrasivity, texture, powder flow, dispersion state, and handling. It is measured using different imaging techniques wherein the data projected is a 2-dimensional particle profile. From the projected image, the particle shape parameters can be determined. Some of the parameters are the particle form, which can be characterized by computing the particle's aspect ratio, dividing the width by the length, and the particle outline, where the roughness of the particle surface can be evaluated. Other changes in the parameters can be observed using the particle form and outline, as these two can affect the behavior of a particle being measured.

Circularity is a commonly used shape parameter that is measured to determine the closeness of a particle's shape to a perfect sphere. Understanding this can help in monitoring the abrasivity property of the particle. Today, no specific particle shape parameter applies to every industry. While circularity is the most common, it is not suitable for some applications, and careful considerations and further understanding of the particle sample being examined are required to properly assign the appropriate parameter that must be used. Furthermore, the effects of particle size are studied more as there is no standard for shape characterization, making it difficult to characterize the shapes of real particles because of their complexity. For 1-dimensional and 2-dimensional images, it can easily be obtained using image analysis; however, measuring the 3-dimensional parameters such as volume, surface area, and sphericity is more complicated, and previous studies have yet to investigate the matter comprehensively.

Sphericity is a critical parameter that can describe a particle's transport and sedimentation properties, specifically an irregular one. With this, 3-dimensional parameters require specific instruments that are often time-consuming and not suitable for measuring a large number of large particles [4]. For most indirect measurements of particle size, it is assumed that the particles in a product yield are round and about 30μm in size. This assumption is incorrect and could lead to an incorrect way of analyzing the behavior of a specific raw material and to an unsuccessful decision-making process.

## 3. Particle Characterization Techniques

Different particle characterization techniques can be used in measuring particulate samples depending on their condition. No universal technique works for every situation and sample; hence, a set of criteria is considered when choosing an appropriate technique for a particular particulate sample. There are different size ranges that specific techniques can handle. Knowing the sample's size range is essential to find a suitable technique to yield the desired product.

### 3.1. Sieve Analysis

Sieve analysis is the most commonly used method in determining and measuring particle size. It is a traditional method consisting of several stacked sieves with an increasing mesh size. The stack is placed into a sieve shaker that vibrates for 5 to 10 min, distributing the particles from the uppermost sieve to different fractions according to the particle sizes until the sample mass on each sieve becomes constant. Sieve analysis is preferably used because of its tendency and ability to determine the particle's width.

### 3.2. Laser Diffraction Particle Sizing

The laser diffraction method detects the amount of light intensely pushed through the measuring cell and dispersed on the soil particles. The detector array, positioned at an angle around the measurement cell, records the dispersed light. The signal captured by the detectors enables PSD computation. These calculations are often performed using the Fraunhofer approximation or the Mie theory, which both need an understanding of the sample's optical properties [5].

### 3.3. Dynamic Light Scattering

Dynamic light scattering (DLS) estimates particle size distributions using Brownian motion. A sensor detects a laser beam after passing through a sample between two polarizing filters. Particles disperse light as it travels through the sample. The light dispersed by the individual particles is exposed to destructive and constructive interference at the detector, resulting in a speckled pattern. As the particles in solution go via Brownian motion, the interfering pattern between them changes. The particle size distribution may be inferred by observing this shifting interference pattern [6].

### 3.4. Dynamic Image Analysis

Lighting, focus, depth of focus, and particle orientation significantly impact optical imaging technologies. If the flow is laminar, the particles typically match each other for dynamic imaging in liquid. However, the size and form of the 2-dimensional projected picture rely on particle orientation in all situations.

Dynamic image analysis provides precise measurements of particle shape by recording the 2D forecast area of a large population of particles at a random position. Two different DIA methodologies can be used: 2-dimensional (2D) and 3-dimensional (3D). 3D DIA records particle pictures from 8–12 different views of chosen particles while tracking the movements of individual particles as they fall in the image plane. The average, maximum, or lowest values from every angle of each particle are used to generate the final particle size and shape characteristics. Moreover, 3D DIA may produce particle size distribution using Feret and EQPC diameters and offers particle shape descriptors like aspect ratio, convexity, and sphericity. These three points of a particle's dimensions are closer to the real shape of the particle [7].

## 4. Application and Innovation

Particle characterization can have different real-life applications. Characterizing an object, sample, or particle allows for the understanding of how a particle behaves, which is a critical factor for quality control and performance of a product in many different fields and industries to ensure the safety and quality of the used products. Each industry has different techniques used in particle characterization depending on the requirements or suitability of the particles. Some industries that use particle characterization are pharmaceuticals, food and beverages, coatings, and paint industries. Specifically, other applications are stated below.

### *4.1. Metallurgical Industry*

The mineral processing and metallurgical industries have acknowledged using image-processing tools to characterize particle size and distribution. The 3D printing and particulate matter (PM) technologies have gained rapid advancement and quality standards for superalloy powders, aiming to have a more rigorous and accurate evaluation of particle shape rather than controlling the other parameters. Image processing techniques make the algorithms for characterizing particle morphologies comprehensible, leading to more dynamic process modification and control of the manufactured powders used in 3D printing and other advanced technologies [8].

The powder flowability and packing density are two prominent advantages and benefits of the spherical particles. Scientists and researchers frequently utilize X-ray tomography and scanning electron microscopy (SEM) to describe particle morphologies. A study by Zhang et al. in 2022 investigated how various image processing algorithms characterize the morphology of superalloy powder particles. A comparative study focusing on the analysis of a single particle's eight shape descriptors, validation of simulation result, calculation of the probability density distributions of the eight shape descriptors, extraction operators, identification of the correlation between mean shape descriptors and angle of repose (AOR), and simulation of standard graphics were also covered and discussed. Therefore, utilizing image processing techniques is an effective way to describe the particle morphology of powders of nickel-based superalloys. Additionally, a correlation between the powder flowability and the different shapes' descriptors showed that it is possible to anticipate the performance of the powder during the 3D printing process [8].

### *4.2. Pharmaceutical Industry*

To be safe and successful, nanoparticle-enabled drugs (NEP) must be steady in complex biological environments and have a particular particle size distribution (PSD). The size distribution of nanoparticle formulation in pharmaceuticals can be measured using a variety of techniques, including electron microscopy (EM), laser scattering, field flow fractionation (FFF) combined with multiple sizing detectors, centrifugal, tunable resistive pulse sensing (TRPS), and particle tracking atomic force microscopy (AFM). A recent dossier analysis submitted to the FDA between 1973 and 2015 stated that nanomedicine stakeholders commonly use dynamic light scattering (DLS) in sizing methods regardless of the low-resolution outputs it produces. Some of the utilized sizing techniques 8% of the applicants used in profiling their products are gel permeation chromatography, field-flow fractionation (FFF) in an asymmetrical flow, and centrifugation [9].

The Brownian motion (autocorrelation function) of a suspension of nanoparticles is used to calculate the correlation of time-dependent fluctuations in the diffused light in dynamic light scattering (DLS). Using the Stokes–Einstein equation, the particles' hydrodynamic diameter (or radius) is computed, and the nanoparticle diffusion coefficient (or coefficients) is determined by fitting the autocorrelation function. The analysis requires familiarity with the solution's viscosity and the assumption that the measured item is spherical. In nanomedicine, DLS is frequently used in "batch" mode, a low-resolution approach with severe limits for analyzing polydisperse materials. Due to the fact that scattering strength increases significantly with the sixth power of particle size, a very small number of big particles may easily conceal smaller ones. In conclusion, utilizing DLs in a batch is a helpful and user-friendly tool for assessing the integrity and stability of a nanoformulation when exposed to highly concentrated salts, inappropriate pH, or plasma [9].

*4.3. Food Industry*

Numerous granules, emulsions, suspensions, and particles are available as food items. The particle size distribution may influence the final product's flavor, appearance, stability, processability, and utility. Nanotechnology, the technology used to manipulate nanomaterials for various purposes, is crucial to the agriculture and food industries because it helps to increase crop yields, enhances food quality and safety, and provides novel and innovative health benefits. Nanomaterials are microscopic particles with diameters between 1 and 100 nm, are insoluble or bio-persistent, can be produced in various ways, and have applications in numerous sectors, including the medical, electrical, agricultural, and food industries [10].

*4.4. Cosmetics Industry*

One of the fastest-growing industrial sectors, the cosmetics industry continually develops by adding innovative but environmentally friendly products. Skin care, color cosmetics, hair treatments, makeup, and body care products are just a few examples of the various formulas that make up cosmetics. The cosmetic industry uses nanoparticles (NMs) to achieve more stability and long-lasting effects on their products. Nanomaterials' large surface areas allow the substances to be absorbed into the skin more effectively. The effective penetration of nanomaterials into the skin for increased ingredient delivery, new color components, transparency, and long-lasting effects might be some of the critical goals of their use in cosmetics. When employing NMs, the cosmetic industries' main objectives are to achieve long-term stability and distribute the proper number of substances to the targeted body sections [11].

Polyester sheets are used to make cosmetic glitters cut into small hexagons, squares, or rectangles after being sprayed with colors and authorized by the Food and Drug Administration. On the other hand, mica is made with intricate aluminosilicate crystals divided into flat sheets-like layers, making up shimmer particles. Mica is a mineral that occurs naturally and may also be made synthetically. Metallic oxides of various sorts and thicknesses have been applied to mica particles to create cosmetic shine. The many hues of a particular color are possible because of the varying thicknesses. While shimmer particles can be found within a particular size range, unlike glitter, their form is wholly random and arbitrary. The EDS enables the analyst to compare and identify two samples by analyzing the utilized polymer's elemental composition, mica substrate, and coatings that generate apparent particle color. Additionally, the EDS enables quick analysis, with sample runs frequently being finished in a few minutes. High-resolution visual examination of the cosmetic particles was possible due to the microscope, which was also coupled to an Oxford Instruments, Abingdon, UK-based Oxford 7353 EDX detector for elemental analysis. The study aimed to investigate whether the SEM-EDS analysis can classify unknown cosmetic particles to provide another technique for forensic analysis on crime scenes or assaults involving cosmetic products. In the result of the study, using SEM-EDS, classification systems for two different kinds of cosmetic particles were effectively developed. Unknown samples may be accurately recognized using LDA, KNN, and SVM, which have 94.4–100% classification rates [12].

Structure, size, and content all substantially impact the physicochemical properties, order, release profile, epidermal interactions, and cosmetic utility of encapsulated cosmetic components. Component safety evaluation is critical for skincare products that come into touch with human beings. Microscopic techniques such as optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), and confocal laser scanning microscopy (CLSM) can be utilized to analyze the carrier system's microstructure. Materials must commonly be chilled to immobilize before being investigated using cryo-electron microscopy on lipid-based carriers. In addition, differential scanning calorimetry (DSC) and X-ray diffraction (XRD) can be used to identify the crystal structure of nanoparticles. Small-angle X-ray scattering (SAXS) can analyze multilayered vesicle formations. The size distribution and homogeneity of the droplets have a considerable influence on the liquid system's stability. Different approaches can be utilized to measure the size distribution of pills, particles, and emulsion drops, such as photon correlation spectroscopy, laser diffraction, dynamic light scattering, and nano-tracking analysis [13].

## 5. Future Outlook

Particle characterization is essential in manufacturing industries, medicine, forensics, and other fields involving nanoparticles. Various methods have been widely established and used based on the studies reviewed. For example, using a variety of inexpensive light sources, including laser diodes and high-intensity light-emitting diodes (LEDs), are becoming widely accessible, and particle characterization scattering, and photography technologies are being employed more and more in research and for the quality assessment of the manufacturing of various goods across multiple industries.

As manufacturing industries produce a wide range of products in support of the utilization of humankind, the need to look at and monitor particle characterization and its applications are necessary to produce better quality materials to obtain better end-products in the market. Few of the industries mentioned have already shown the need for better particle characterization techniques to produce better materials, as particle size and other parameters define the properties of the products produced in the said industry. However, it was clear that despite those techniques, processes are expensive and still need more improvements to offer the optimized procedure to produce such products. Nonetheless, the advances in particle characterization in industries are important potentials that innovators must focus on to acquire accessible and efficient processes and techniques in creating industrial products.

Particle characterization distribution data can be obtained using a variety of techniques. The selected characteristic is the most important factor when quantifying particle size and size distribution. When describing a property, it is essential to consider the potential for error and bias introduced by the measuring instruments and methods applied. It is essential to consider the aspect ratios of the particles, as they may have a substantial effect on the result. Optimally, size data reporting methods should consider the distribution's shape and extent and specific typical characteristics such as the median and moment. It is further recommended to conduct more research using other methods, such as acoustic and electrical methods, as it was observed that most industries focus on using imaging or optical methods in particle characterization.

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