Optimization and application of advanced laser nanoparticle sizer on wide range of materials

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Abstract. Particle size distribution is very important parameter in many industries, such as mining, food or pharmaceutical industry. Ditto, research of new advanced materials or nanomaterials as well as particle-based computational modelling, involves advanced knowledge of particle sizes. Determination of particle size distribution of different type of particles (various in shape, composition, density, physical and chemical properties, etc.) by advanced laser nanoparticle sizer was challenging. The ANALYSETTE 22 device with movable measuring cell position between the Fourier lens and the detector provides innovative approach in particle size determination method. Also, a comparative analysis of dry and wet measurements of the same samples (where it was possible) was carried out. Sample quantity and its physical properties were greatly designated which method (dry or wet) and which calculation theory (Fraunhofer or Mie) would be applied. After many analysis, obtained results showed that accuracy and repeatability of produced results exclusively depends of sample preparation and its physical and chemical properties.

Keywords: optimization, particle size distribution, laser diffraction, ANALYSETTE 22 NanoTec plus.

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1 Introduction

In recent decades, particle-based techniques and their application in engineering and scientific research have become increasingly important [1, 2]. These techniques include synthesis and characterization of powders regardless they are inputs or outputs of material production. Accordingly, determination of relations between particle shape/size and final material properties is also the subject of many studies. In the other hand, particle-based computational models can be effectively used for solving a variety of problems in engineering. Hence, particle size measurements are of great interest in many research and industrial areas. Since laser diffraction method conducts reliable and repeatable results and is generally easy to operate with, it is very often primary method for investigation of particle size distribution [3].

In the present study, an attempt has been made to determinate important parameters which have influence on reliable results of analysed particle system by using advanced laser nanoparticle sizer ANALYSETTE 22 NanoTec plus (Figure 1) [4].

Fig. 1. Advanced laser nanoparticle sizer ANALYSETTE 22 NanoTec plus [4].

Methods

The laser diffraction method is based on very simple principle: when the particle is exposed to laser beam, the light diffraction occurs which resulting in a characteristic, ring-shaped intensity distribution behind the sample which is measured by a specially shaped detector. The Fritsch GmbH was the first company which used an inverse Fourier optic for the particle size determination and the first model was the ANALYSETTE 22 (Figure 2a). The spacing of these occurred rings is used for calculation of particle size: the small particles cause widely spaced characteristic rings (Figure 2b) while large particles produce more closely located rings (Figure 2c).

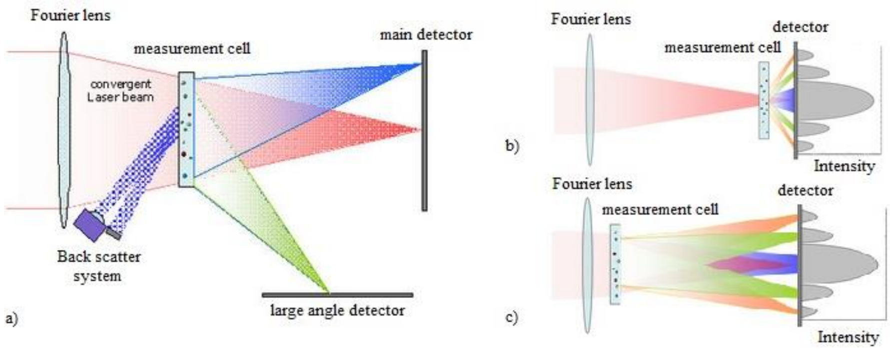

Figure 2. a) The basic outline of ANALYSETTE 22 model with an inverse Fourier optic, b) the measuring cell position for small particle and c) the measuring cell position for large particles [5].

Results

The advanced laser diffraction measurement procedures were optimized and applied on different type of materials (Figure 3): beverage emulsions in concentrated forms, sodium bicarbonate and copper matrix composite powder.

Beverage emulsion   Sodium bicarbonate   Copper matrix composite powder

Figure 3. Images of analysed materials.

Fraunhofer diffraction pattern was chosen for determination of particle sizes, by applying equation [4]:

$$I(P) = |U(P)|^2 = I_0\left[\frac{2 \cdot J_1(k \cdot a \cdot u)}{k \cdot a \cdot u}\right]^2$$

Whereby: u=arc tan (r/f) and k=2π/λ,

If the particles are uniform i.e. have the same size, then particle size can be determent by equation [4]:

$$d = \frac{1.84 \cdot f \cdot \lambda}{R_0}$$

, R –radius of the rings, f – focal width of the lens.

However, during experimental measurements usually there is non-uniform particle sizes and in that case an equation system of both equations must be applied. After detection and calculation of particle size (equivalent diameter), the results are presented as graphic diagrams (Figure 4). The overall data is arranged according to the geometric dimension of the particle (for example the equivalent diameter x) and plotted to the x-axis of a coordinate system [4]. The components that are associated with the size of the individual elements and indicate the shares of individual particles within the overall distribution are depicted along the y-axis. The cumulative curve of the distribution particle size Q_r (x) shows a standardised total quantity of all particles with equivalent diameters less than or equal to x. Parameter q_r (x) shows the particle size range i.e. value is between x_1 and x_2.

$$q_r(x) = \frac{x^r \cdot q_0(x)}{\sum_{i=1}^{n} x_i^r \cdot q_{0i}(x_i)} = \frac{dQ_r(x)}{dx}$$

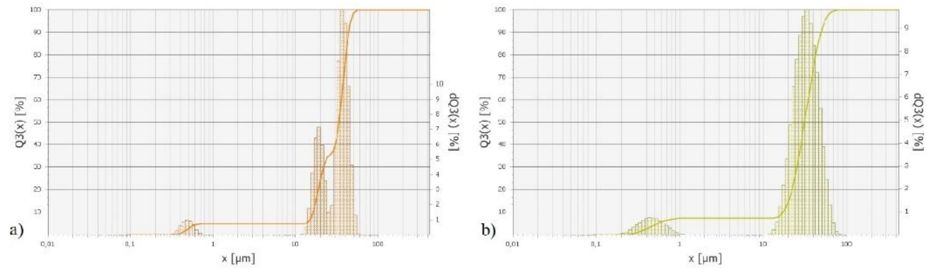

Figure 4. Measurements of copper matrix composite a) poor and b) good result.

The sodium bicarbonate were examined by dry measurements and beverage emulsions with wet measurements. Copper matrix composites were investigated by both type of measurements and were used for comparative analysis of dry and wet method. Experimental analysis showed that reliable and repeatable results can be obtained only if few parameters are taken in consideration, such as: chemical composition of samples, physical and chemical properties of sample, methodology of measurements, sample quantity, etc.

Conclusions

This study provides a basic review for those who are starting to apply any particle-sizing technique based on laser diffraction. The ANALYSETTE 22 device produced by Fritsch GmbH is the first model with an inverse Fourier optic which is innovative approach in analysing and determination the particle size distribution. This innovation provides the movable measuring cell position between the Fourier lens and the detector. For large particles measuring cell is closer to the Fourier lens while for small particles is closer to the detector. Optimization of laser diffraction measuring methods was of great importance for method verification and accuracy of obtained data. Results obtained by analysing different types of materials point out that e.g. sample quantity, physical and chemical properties of sample and/or methodology of measurements have great influence on result accuracy.

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