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Laser particle size analysis – the influence of density and particle shape on measurement results

Introduction

There are many methods of measuring particle size. Generally, they can be divided into direct methods (applying parameters like the screen size of a particle, Martin's diameter, or Feret's diameter) and indirect methods using physical phenomena and certain calculating techniques (like measuring the viscosity of slurries, light diffraction in suspensions, photoelectric space searching, or particle segregation in a centrifugal field) (Allen 1992; Mączka, Trybalski 1981).

The choice of the measuring method for the purpose of determining the particle size distribution of grained materials depends on the various properties of a representative sample? mainly on the range of particle sizes in the examined sample (Trybalski et al. 2004). It should be noted that each of the methods generates different information on particle size distribution. The applied measuring method highly influences the results because each considers various material characteristics such as geometrical properties, density, type of surface (porosity), etc. (Peszko et al. 2000; Peszko et al. 2007).

In this investigation, the laser method of measuring particle size was applied by employing the phenomenon of laser light diffraction. Thus, the shapes of the examined particles are very important as well as their porosity and the range of particle sizes occurring in the sample. During the measurement of particles varying in size, [the diffractogram's influence on each other and makes the analysis harder (wording cannot be interpreted, please revise)].

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The coincidence of the factors mentioned above influences the error of the measurement and its precision (Haustein, Quant 2011; Kordek 1999). Furthermore, the influence of material density is also important. Materials of higher density may not be sufficiently dispergated and directed to the measuring cell (particle sedimentation) in a way not representative of the entire sample, causing measuring errors.

The advantages and disadvantages of applying the laser method to determine particle size distribution remain the subject of discussion and research around the world (Ramaswamy, Rao 2006; Rodriguez, Uriarte 2009). Despite varying results offered by different methods of determining particle size distribution, various authors claim that the laser method is best either because of the high precision of the results obtained or the short time required for the measurement when compared with the lengthier and harder to perform Pipette method (Loizeau et al. 1994; Konert, Vandenberghe 1997).

The purpose of the study presented here was to verify the influence of density and particle shape on the results of laser particle size analyses.

The adequacy of this analysis was characterized by variation coefficient w for percentage shares of individual particle fractions, and the repeatability of the results was not examined because the determined value of measurement precision of <1.5% was given by the device's manufacturer.

1. Experiments and analyses

The following materials of varying densities were used in this study: coal with a mean density of 1.45 g/cm^3, porphyry 2.78 g/cm^3, and barite 4.45 g/cm^3. However, their granulation was quite similar (0–600 μm). Additionally, the study included copper shale ore and volatile ash originating from hard coal combustion. Their particle shapes were different, ranging from 0–300 μm. The particle shapes were described numerically by an elongation coefficient comparing to circle K_α, which was calculated by means of computer image analysis of the studied materials. An optical microscope was used for this purpose which was equipped with a digital camera and the image analysis program Aphelion.

Figures 1 and 2 present examples of microscopic pictures of copper ore and ash particles. The shape coefficient K_α for shale ore was equal to 1.87 and for ash 1.12 (for circle $K_\alpha = 1$). The differences between coefficients prove the different shapes of particles of both materials. The particles of copper shale ore were characterized by the irregular, elongated shape of their sharp edges (Fig. 1).

The volatile ashes indicated the particles were of a spherical shape, but of various diameters, colors, and crystallization levels (Fig. 2). Some of the particles were microspheres with a diameter of <0.5 mm which contained smaller particles inside. Also, particles of glasslike bubbles containing gas occurred. The thicker fractions of ashes indicated irregular shapes of particles of high porosity.

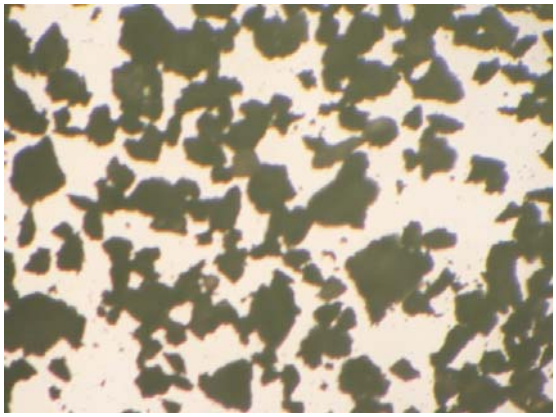

Fig. 1. Microscopic image of the copper shale ore particles

Rys. 1. Zdjęcie mikroskopowe przedstawiające ziarna rudy łupkowej Cu

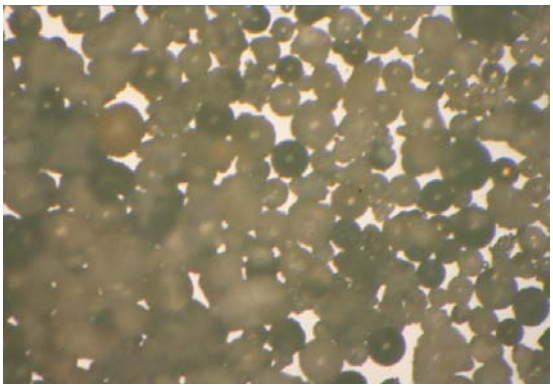

Fig. 2. Microscopic image of fly ash particles from coal combustion

Rys. 2. Zdjęcie mikroskopowe ziaren popiołu lotnego ze spalania węgla

The samples under study were material suspensions which were classified several times in a laboratory hydrocyclone giving two products each time: fine-grained overflows and thick-grained outflows (the conditions of classification were not the subject of this paper). Three representative samples were collected each time from the feeds and classification products for the purpose of calculating the variation coefficients *w*. Figures 3 and 4 present the mean particle size distribution of the examined products.

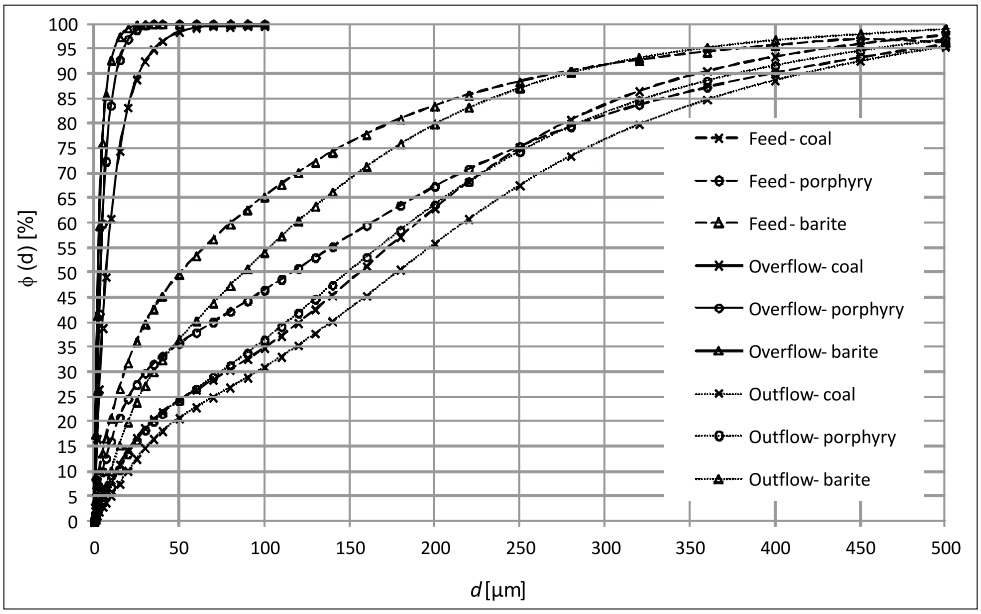

Fig. 3. Mean granulation of analyzed products originating from materials of varying densities

Rys. 3. Średnie uziarnienie badanych produktów materiałów różniących się gęstością

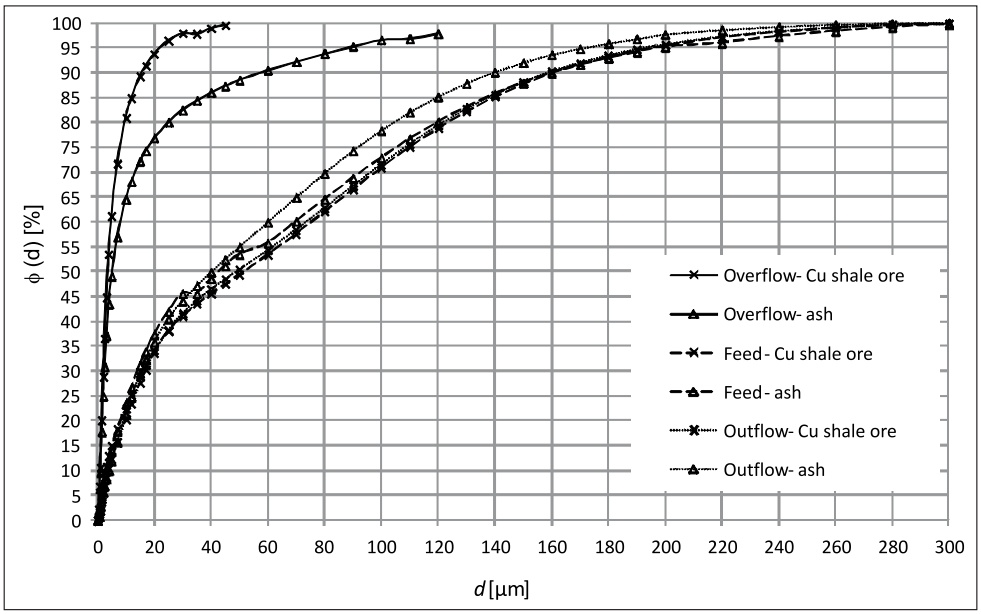

Fig. 4. Mean granulation of analyzed products originating from materials of varying particle shapes

Rys. 4. Średnie uziarnienie badanych produktów materiałów różniących się kształtem ziaren

2. Measurement method

A laser analyzer of particle size Analysette 22 was used for the particle size analyses, which was equipped with a helium-neon laser, optical system, flowing measure cell for suspensions, and dispersing unit.

The rule of measurement is based on laser stream diffraction of the measured particles. The laser stream x-rays the measured cell and diffracts on the particles proportionally to their sizes. When the particle population occurs, the volumetric distribution of their size is described by the intensiveness of the distributed light diffracted on them. The diffraction occurs on the edges dividing two media of various light diffraction coefficients. These edges then become the new sources of the waves. The interference phenomenon accompanying this event creates the diffractograms – alternate bright and dark spherical rings. According to the diffraction rule, small particles refract laser light at higher angles, and larger particles result in smaller refraction angles from the original light stream. The luminosity is proportional to the contents (amount) of the individual particles. The diffraction image produced is identified by a system of photosensitive detectors. The obtained signals are used to calculate the particle size distribution. To accomplish this, the Fraunhofer transformation is applied which describes the phenomenon of the diffraction of light passing through the diffraction grating (studied sample). It allows for the calculation of the relations between the diffraction grating constant, the geometrical parameters of the measuring system, the light wavelength, and the diffraction effect registered by the detector.

$$Particle\ diameter = \frac{1.84 \cdot f \cdot wave\ length}{R_o}$$

where:

R_o – radius of diffraction rings,
f – focal length of lens.

This equation may be applied only when the analyzed particles are of a similar size. In the case of two particles of different sizes, diffractograms influence each other and make the analysis harder to perform. Two equations are then needed to determine their diameters and the intensiveness of the diffracted light as a function of the diffraction angle measuring their mutual volumetric ratios. The number of equations and number of particles for each equation is equal to the number of measured particle fractions. A set of matrices is obtained which allows for the determination of the size of particles and their mutual volumetric ratios. These calculations are being realized automatically by the calculating system of the measuring device (De Boer et al. 1987; Instrukcja… 1994; Syvitski, ed. 1991).

3. Analysis of results

In order to determine the influence of mineral density and particle shape on the accuracy of laser particle size analyses, the following basic statistical parameters were calculated: standard deviations s and variation coefficients w_i for particle fractions' shares in individual products, according to formula 1.

$$w_i = \frac{s}{\overline{a}} \cdot 100\% \tag{1}$$

where:

$\overline{a}$ – mean contents of the i^{th} particle fractions in material [%],

s – standard deviation of the i^{th} particle fractions contents in the material.

Table 1 shows the examples of data and statistical calculations for coal overflows as an example of the calculation methodology applied by elaboration of the results.

TABLE 1

Contents of particle fractions in overflows of individual experiments conducted for coal with statistical data

TABELA 1

Zawartości klas ziarnowych w przelewach poszczególnych eksperymentów dla węgla wraz ze statystykami opisowymi

Particle fractions [mm]	Contents of particle fractio a_i					Statistics		
						a	s	w_i
0–0.5	1.04	1.35	1.18	1.15	1.02	1.15	0.13	11.51
0.5–1	3.97	4.12	3.95	3.94	3.87	3.97	0.09	2.32
1–2	8.9	8.82	8.65	8.82	8.78	8.79	0.09	1.04
2–3.15	8.57	8.43	8.34	8.45	8.43	8.44	0.08	0.98
3.15–5	11.01	10.85	10.75	10.82	10.84	10.85	0.10	0.88
5–7	9.87	9.65	9.63	9.63	9.68	9.69	0.10	1.05
7–10	12.4	11.99	12.13	12.1	12.17	12.16	0.15	1.24
10–15	15.54	14.93	15.27	15.33	15.35	15.28	0.22	1.45
15–20	10.44	10.1	10.28	10.48	10.37	10.33	0.15	1.46
20–25	6.71	6.58	6.62	6.86	6.67	6.69	0.11	1.62
25–30	4.27	4.26	4.22	4.42	4.25	4.28	0.08	1.83
30–35	2.7	2.75	2.7	2.83	2.71	2.74	0.06	2.02
35–40	1.72	1.78	1.74	1.8	1.74	1.76	0.03	1.87
40–50	1.87	2.04	1.99	2.0	1.96	1.97	0.06	3.24
50–60	0.83	1.02	1.53	0.93	0.98	1.08	0.28	26.17

Figures 5–9 present the mean values of variation coefficients for feeds, outflows, and overflows for individual materials.

An analysis of figures 5–9 indicates that the precision of analyses measured by variation coefficient is different for particular particle size fractions for classification products. The

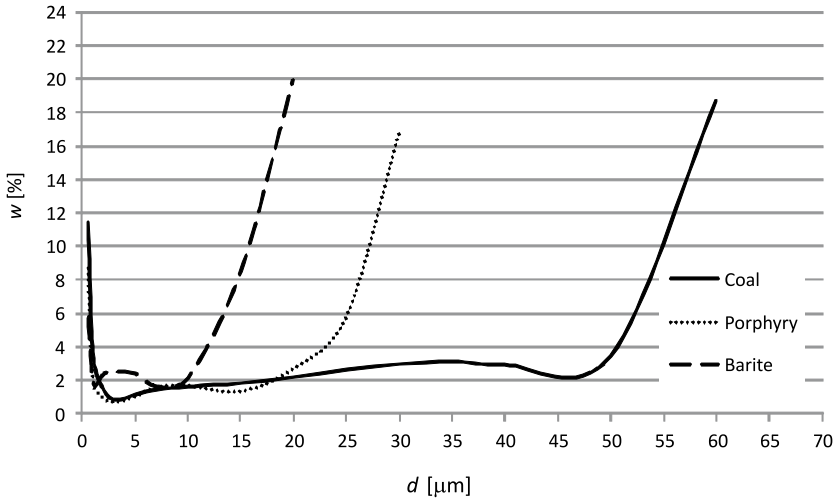

Fig. 5. Variation of mean values of variation coefficients of individual particle fractions shares in analyzed material overflows

Rys. 5. Zmienność wartości współczynników zmienności udziałów poszczególnych klas ziarnowych w analizowanych przelewach

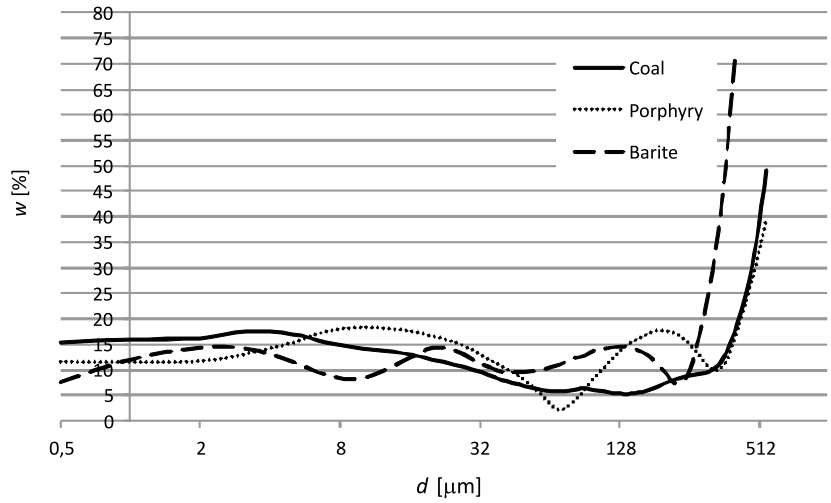

Fig. 6. Variation of mean values of variation coefficients of individual particle fractions shares in analyzed material feeds

Rys. 6. Zmienność wartości współczynników zmienności udziałów poszczególnych klas ziarnowych w analizowanych nadawach

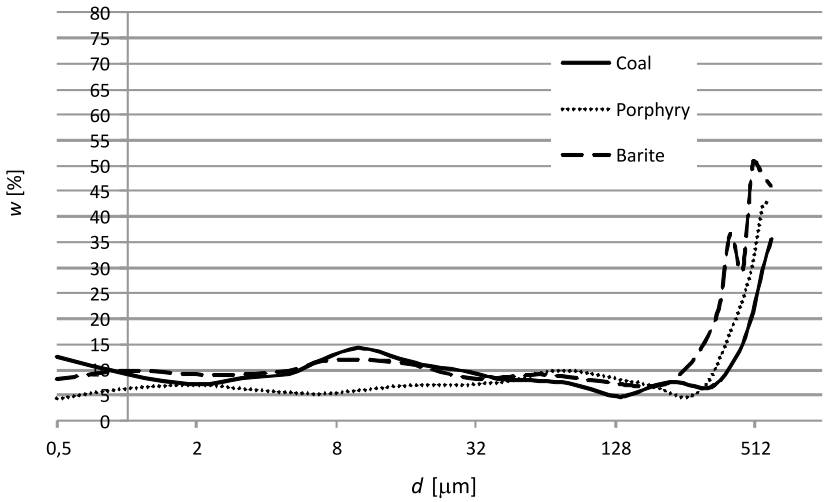

Fig. 7. Variation of mean values of variation coefficients of individual particle fractions shares in analyzed material underflows

Rys. 7. Zmienność wartości współczynników zmienności udziałów poszczególnych klas ziarnowych w analizowanych wylewach

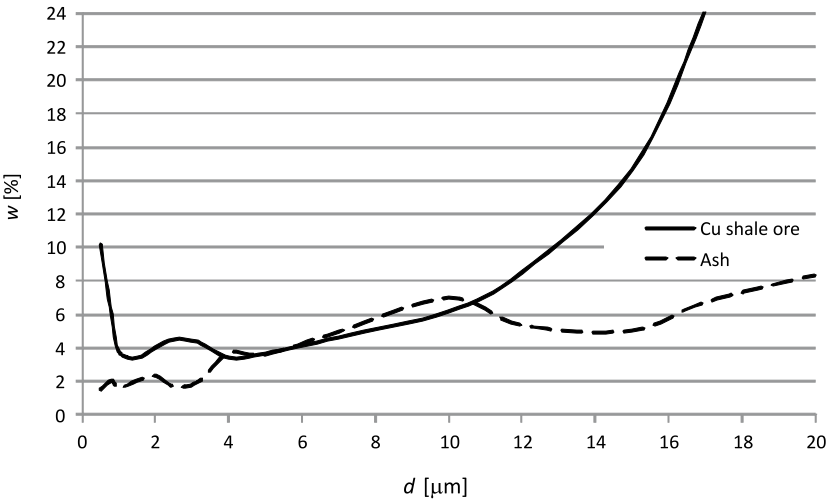

Fig. 8. Variation of mean values of variation coefficients of individual particle fractions shares in analyzed material overflows

Rys. 8. Zmienność wartości współczynników zmienności udziałów poszczególnych klas ziarnowych w przelewach

highest values of these coefficients were obtained for feeds and outflows and the lowest for overflows. The range of granulation of overflows in comparison with outflows was much narrower, proving that the particle size analyses for overflows were the most precise.

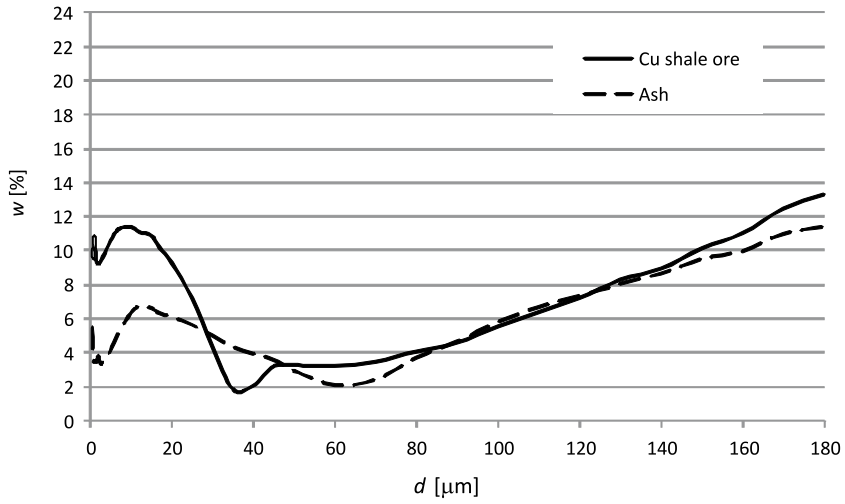

Fig. 9. Variation of mean values of variation coefficients of individual particle fractions shares in analyzed material underflows

Rys. 9. Zmienność wartości współczynników zmienności udziałów poszczególnych klas ziarnowych w wylewach

This is directly connected with the measurement method (light diffraction) used in the laser analysis. The diffractograms produced during analysis originated from the measured particles' mutual overlap, making the analysis harder (De Boer et al. 1987; Instrukcja… 1994; Peszko, Niedoba 2006; Syvitski, ed. 1991). The most deranging the measurement (meaning unclear, please revise) are diffractograms originating from the biggest particles. The extremely large particles influence strongly the precision of the finest particles' measurement, and the fine particles cause the opposite effect without resulting in significant errors in measuring the larger particles.

The differences in particle size analyses' adequacy of overflows, outflows, and feeds were most visible for materials of varying particle density. The cause of such a situation could be the wider range of their granulation (0–600 μm) in comparison to materials of a different shape coefficient (0–300 μm) connected also with greater errors during the sampling process.

The variation coefficients for particle fractions ranging from extremely fine particles to extremely thick are quite high, obviously connected with the randomness of measurement for such a small population of these particles, indicating high values of errors. Insignificant mean contents of these fractions cause higher values of calculated factors w_i. Furthermore, laser analysis for the finest particles categorized by limited sensitivity of the measurement due to the boundaries of the device's measuring range (de Boer et al. 1987; Kordek 1994).

Analyzing the influence of material density on the preciseness of particle size laser analyses, no significant differences were observed in the values of variation coefficients.

For barite (the density of which is the greatest), however, the coefficients for particle fractions indicated slightly higher changeability.

Considering the influence of material particles' shapes on the preciseness of particle size laser analyses, it can be stated that ash provides more stable results especially for the extremely fine particle range. This can be due to the more spherical shape of ash particles. The variation coefficients of particle fractions' contents in both products of ash classification were lower in comparison to coefficients obtained for copper ore. However, these differences are not large.

Conclusions

The results of this study demonstrated that:

— Laser particle size analysis gives precise information about particle size distribution.
— No significant influence was observed of material density on the preciseness of particle size analyses performed by the laser method.
— The influence of the particle shape for the examined materials on the adequacy of the particle size analyses performed using the laser method was demonstrated. This occurs because the laser diffraction method is based on calculating all of the dimensions of irregular particles. All of these are taken into consideration when preparing the results of the analysis.
— The preciseness of laser particle size analyses varies for individual material streams and is described by a variation coefficient of particle fractions' shares. The most precise results are obtained for overflows, while the least precise results were obtained for feeds and outflows.

As the conducted research indicated, the observed differences occur mainly from the measuring method applied in order to determine particle size distribution. This is based on the laser diffraction phenomenon as well as from different particle size distributions in classified material streams.

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LASEROWA ANALIZA UZIARNIENIA – WPŁYW GĘSTOŚCI I KSZTAŁTU ZIAREN NA WYNIKI POMIARÓW

Słowa kluczowe

Laserowe analizy granulometryczne, dokładność analiz granulometrycznych

Streszczenie

Artykuł dotyczy dokładności określania składów ziarnowych materiałów drobnoziarnistych metodą laserową. Wybór metody pomiarowej do określenia składu ziarnowego materiałów uziarnionych zależy od różnych właściwości reprezentującej go próbki, głównie jednak od zakresu wielkości ziaren znajdujących się w badanej próbce. Należy jednak zdawać sobie sprawę, że każda z metod pomiarowych generuje z założenia różną informację o rozkładzie wielkości cząstek. Na wyniki oznaczeń główny wpływ ma bowiem stosowana metoda pomiaru, wykorzystująca różne właściwości materiałów: np. właściwości geometryczne, gęstość, charakter powierzchni (porowatość) itp.

Badano więc wpływ gęstości oraz kształtu cząstek na wyniki pomiarów metodą dyfrakcji laserowej, która to metoda staje się standardową w pomiarach uziarnienia proszków mineralnych. Analizy składów ziarnowych surowców wykonano przy użyciu laserowego granulometru Analysette 22 firmy Fritsch. Badania obejmowały pomiary uziarnienia surowców różniących się gęstością (węgiel kamienny, porfir, baryt) oraz kształtem ziaren (łupkowa ruda miedzi, popiół lotny ze spalania węgla). Gęstość surowców określono metodą piknometrii helowej, natomiast kształt ziaren wyrażono współczynnikiem kształtu obliczonym na podstawie wielkości geometrycznych

cząstek. Geometrię ziaren badano przy użyciu mikroskopu optycznego z cyfrowym zapisem zdjęć, które poddano komputerowej analizie obrazu. Dokładność laserowych analiz granulometrycznych wyrażono współczynnikiem zmienności udziału wąskich klas ziarnowych. Wyniki analiz potwierdziły, że laserowa analiza granulometryczna dostarcza dokładnych informacji o rozkładzie wielkości cząstek najdrobniejszych. Nie zaobserwowano istotnego wpływu gęstości materiału na dokładność analiz granulometrycznych. Wpływ kształtu ziaren badanych materiałów zaznaczył się stabilniejszymi wartościami współczynnika zmienności dla cząstek o kształcie bardziej sferycznym, co ma związek z zastosowaną laserową metodą pomiaru. Dokładność laserowych analiz granulometrycznych różni się w zależności od zakresu uziarnienia mierzonych cząstek, najdokładniej analizowane są materiały w wąskich klasach ziarnowych.

LASER PARTICLE SIZE ANALYSIS – THE INFLUENCE OF DENSITY AND PARTICLE SHAPE ON MEASUREMENT RESULTS

Key words

Laser granulometric analysis, accuracy of granulometric analysis

Abstract

The paper concerns the accuracy of determining particle size distributions of the fine-grained materials by means of laser diffraction method. Selection of measuring method for determination of materials granulation depends on various properties of the sample, but mainly on the range of particle size in the sample. It must be taken into consideration that each of the measurement methods inherently generate different information about particle size distribution. The applied measurement method generates the main impact on the results of research because it uses various material properties, like: geometric properties, density or type of the surface (porosity).

Influence of density and particle shape on the results of measurements by laser diffraction was studied in the paper. This method becomes a standard for measuring particle size of mineral powders. Analysis of raw materials particle size distribution was performed using a laser particle-meter Analysette 22. Investigations included measurements of particle size of raw materials characterized by various densities (coal, porphyry, barite) and the shape of the particles (copper shale ore, fly ash from coal combustion). The density of raw materials was determined by helium pycnometer, while the particle shape was expressed by coefficient which was calculated on the basis of particles geometric parameters. Geometry of the grains was measured using an optical microscope with a digital record of images by means of image analysis method. The accuracy of laser granulometric analyzes was expressed by variation coefficient of narrow particle fractions contents. Results of analyzes confirmed that the laser granulometric analysis provides accurate information about the finestparticle size distribution. No significant effect of the material density on the accuracy of granulometric analysis was observed. Effect of particle shape of the tested materials caused more stable values of the variation coefficient for particles of more spherical shape what is related to the applied method of laser measurement. The accuracy of laser granulometric analyzes varies dependably on the measured particle size range of particles. The most accurate analyzed materials are these ones being the part of narrow particle fractions.