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A particle size distribution measurements of selected building materials using laser diffraction method

Pomiary wielkości rozkładu cząstek wybranych materiałów budowlanych z zastosowaniem metody dyfrakcji laserowej

Abstract
This article presents the results of experimental research to determine the size of powder graining in building materials, specifically, gypsums (Dolina Nidy, Pinczow) and adhesives (Atlas Sp. z o.o., Lodz, and Weber Saint-Gobain, Warsaw). A modern laser diffraction method for measuring the distribution of particle sizes with respect to their diameters was applied. During the experiment, three series of measurements were conducted for each material. Data relating to the graining of building materials is crucial because it is one of many aspects that directly affects the binding time and defines its potential applications in the construction industry.

Keywords: laser diffraction, building materials, diameter of dust grains, particle-size distribution, Fraunhofer theory

Streszczenie
W artykule zawarto wyniki badań eksperymentalnych, określających wielkość ziaren w pylistych materiałach budowlanych: gipsach (Dolina Nidy, Pinczów) i klejach (Atlas Sp. z o.o., Łódź oraz Weber Saint-Gobain, Warszawa). W pomiarach zastosowano nowoczesną metodę dyfrakcji laserowej do pomiaru średnic cząstek. Podczas eksperymentu przeprowadzono trzy serie pomiarów dla każdego materiału. Ziarnistość materiałów budowlanych stanowi jeden z wielu aspektów, które bezpośrednio wpływają na czas wiązania, a zatem określają ich użyteczność w budownictwie.

Słowa kluczowe: dyfrakcja laserowa, materiały budowlane, średnica ziaren pyłów, rozkład wielkości cząstek, teoria Fraunhofera

1. Introduction

Laser diffraction using laser particle-size meters is a widely adopted methodology in research and developmental work regarding the quality control of finely-grained products, soil samples and technological processes in different industrial branches such as the pharmaceutical, construction, chemical, food, and metallurgical industries.

Particle-size measurements describe a given product and determine its durability and firmness, which are very significant during the control of production processes [1, 20, 27].

There is no doubt that the graining of building materials is of considerable importance, because it is one of many aspects which directly affects their binding time and thus defines the potential applications of a given material in the construction industry, as well as in everyday life. The greater the fragmentation of a binding material, the shorter the binding time of building gypsum, cement or gypsum adhesives. Furthermore, information regarding particle-size distribution (PSD) parameters of many materials is essential during engineering practice and in experimental research.

Some studies have examined the relationships between particle size distribution in raw materials and various physiochemical and mechanical properties of cement-based mortars. Thus, the influence of cement PSD on hydration kinetics and hardened paste strength [13, 15, 24, 29] and its effect in admixtures (slag and fly ash) on their properties [3, 8, 14, 23, 34, 35] have been examined. The influence of particle size and shape in specific aggregates on the properties of hardened mortar has also been studied [2, 22].

Many research projects have developed and evaluated alternative methods of particle size measurement for soils with gypsum that includes size distribution of gypsum particles. Common laboratory methods for determining the particle size distribution of soils, including those containing large amounts of gypsum, involve the complete removal of gypsum and more soluble salts since they interfere with sample dispersion and the establishment of a stable clay suspension [5, 16, 26]. In this approach, particle size measurements only reflect the size distribution of essentially insoluble, dominantly silicate minerals, and do not reflect the size distribution of the whole soil including gypsum [26]. The authors [6, 17, 31] proposed a method to determine soil particle size with gypsum intact that involves the suspension of the sample in a $BaCl_2$ solution.

The particle-size distribution of selected building materials obtained with the use of the laser diffraction method are presented and analysed in this article. For all building materials, water was the dispersing phase.

2. Basic theory

The authors [27] presented the basic assumptions of the Fraunhofer theory shown below. When a collimated laser beam with wavelength λ is incident on a single spherical particle, the light diffracted in the range of a small forward angle can be approximated by the Fraunhofer diffraction theory if the particle size parameter x

satisfies $x \gg 1$ ($x = \pi d/\lambda$, d is the diameter of the particles). According to the Babinet principle and the Fraunhofer diffraction theory, the diffracted light is given by the Airy function:

$$I(\theta,x)=I_0\frac{\pi^2 d^4}{16F^2\lambda^2}\left[\frac{2J_1(x\sin\theta)}{x\sin\theta}\right]^2 \quad (1)$$

where:

- I_0 – the intensity of the incident light,
- θ – the diffraction angle,
- J_1 – the Bessel function of the first kind of order 1,
- λ – the wavelength of the incident light,
- F – the focal length of the Fourier lens.

In practice, $\sin\theta \approx \theta$ because θ is very small; therefore,

$$I(\theta,x)=I_0\frac{\pi^2 d^4}{16F^2\lambda^2}\left[\frac{2J_1(x\theta)}{x\theta}\right]^2 \quad (2)$$

For a suspension of spherical particles, if the concentration is low enough for the optical thickness (τ) of the sample to meet the condition of single scattering, i.e. $\tau << 1$, Equation (2) can be integrated over the range of particle sizes to obtain the distribution of the diffraction intensity as shown in Equation (3) [28]:

$$I(\theta)=I_0\int_0^\infty \frac{\pi^2 d^4}{16F^2\lambda^2}\left[\frac{2J_1(x\theta)}{x\theta}\right]^2 n(x)dx=I_0\int_0^\infty\frac{J_1^2(x\theta)x^2n(x)}{F^2k^2\theta^2}dx \quad (3)$$

Here, $n(x)$ is the number of particle distributions and $k = 2\pi/\lambda$ is the wave number; Equation (3) is a Fredholm integral equation of the first kind [7, 21, 27].

3. Experimental set-up

The diffractometric method is based on the phenomenon of laser diffraction. The theoretical basis for the diffractometric method is the description of the phenomenon of optic diffraction with the use of Fraunhofer's transform. The possibility of the practical use of this description emerged as a result of building the laser and calculating methods and measuring devices for analysing optical images.

An optical system (Fig. 1) for the analysis of optical images consists of: a monochrome light source (laser beam); an optical system forming its beam; an optical image that was made of suspended powder grains in liquid which enables the passage of light and causes its diffraction on the edge of the grains; an optical system transforming the beam of transmitted light; an image detector that was created by the beam of light; a converter; a computer recording all data. Diffraction methods are used for the granulometric assessment of grains in many branches of industry such as the construction, food and pharmaceutical industries [3, 9, 11, 12, 18, 25, 30, 32–34].

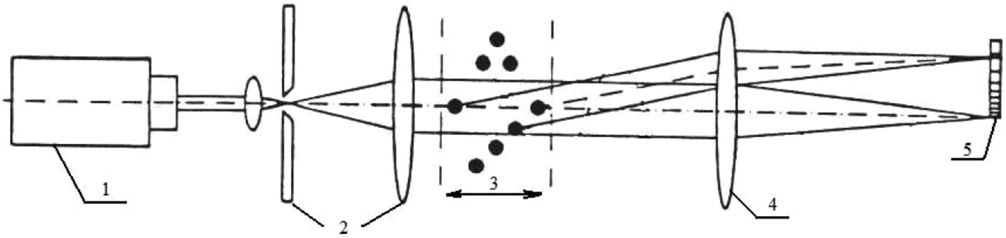

Fig. 1. Laser granulometer [19]: 1 – laser, 2 – optical system forming laser beam, 3 – measuring cell with grain material, 4 – lens, 5 – image detector

The image transformed in the granulometer and its transformation are placed in focal lenses. The diffraction of the light wave takes place at the border of the permeable phase (liquid) and the impermeable phase (powder grains) – this generates an image in a form of diffraction grating. All diffracted and transmitted light beams are focused by the lens creating a diffraction image, the so-called diffractogram in a form of light and dark stripes or spots; such an image enables calculation of the distances of the grating stripes. The determination of the grain diameter is based on the measurement of the stripe distance from the optical axis and its brightness intensity. The obtained image shows that the greater the stripe distance is from the optical axis, the smaller the size of the examined grains. Moreover, the higher the brightness intensity of the stripe, the more grains of particular dimensions can be found in the whole sample.

In this experimental set up, an innovative method was used during the inspection of many technological processes for determining the usefulness of powdered building materials on the basis of their granulation. This method counts all measurements of irregular particles and takes them into account in the results – this demonstrates that this is a highly precise method.

The innovative method of inverse Fourier system was used in a measuring system. A new method may be used in determining equivalent diameters, important in the processes of heat exchange. For particle size analysis, the Analysette 22 MicroTec Plus particle size analyser produced by FRITSCH GmbH Milling and Sizing was used (Fig. 2) – this applies an inverse Fourier system in accordance with ISO 13320 [19]. The device consists of two lasers – one green and one infrared. The particle size analyser is of a modular structure which includes a measuring unit and corresponding sample preparation units for wet and dry measurements. The particle size analyser was applied in this research to study the samples of mixture in a suspended liquid in water. The larger unit is a measuring unit and the smaller unit is the dispersing one.

The sample was added to the dispersing unit, which was then pumped into the measuring cell, which consists of two semiconductor lasers of different wavelength ranges. The green laser was used to detect smaller particles and the infrared laser was used to detect those of larger dimensions. The device was controlled by MaScontrol software, which enables operation of the particle size analyser. It contains a list of standard procedures, including instructions for filling the measuring cell with water, cleaning and conducting a complete measurement. The measurements are conducted automatically and the user is regularly provided with information about each step.

Fig. 2. Laser particle-size meter Analysette 22 MicroTec Plus applied in the research

Grain diameters within the range of 0.08 μm and 2100 μm can be measured with the device shown in Fig. 2. The measurement of particle-size distribution with the use of the diffractometric method and the application of a particle size analyser and a computer is very precise. The device does not require any calibration based on the basic physical properties. When we use Fraunhofer's theory, there is no need for specific knowledge of physical properties of the materials such as density. The measuring time for the sample grains was between 2 and 3 minutes [10]. During the measurement, the beam absorption should be within the range of 10–15% – this indicates the optimal amount of added sample required for proper dispersion. A special photodiode is part of the measuring device and plays a significant role in the beam absorption process as it measures the intensity of a non-dispersed laser beam. This intensity is 100% when there is no sample in the cell. While adding the sample, some of the light is dispersed – this causes a reduction in the intensity that falls onto the photodiode.

Research on the diameters of grains of the following four building materials is presented: building gypsum (Dolina Nidy, Pinczow, Poland); filling gypsum (Dolina Nidy, Pinczow, Poland); gypsum adhesives (Atlas Sp. z o.o., Lodz, Poland); gypsum adhesives (Weber Saint-Gobain, Warsaw, Poland). The basic physical properties of the building materials used in the research are presented in Table 1. E. Bock [4], showed gypsum as being slightly soluble in water (~2.0 g/dm^3 at 25°C) and, unlike most other salts, it exhibits reverse solubility, becoming less soluble at higher temperatures. Therefore, in the presented work, gypsum solubility was considered to be insignificant.

Table 1. Physical properties applied in the research of building materials

Building material	**Relative density d_R [kg·m^{-3}]**	**pH [–]**	**Water solubility K [g·dm^{-3} H_20]**	**Appearance**
building gypsum	2300	7–8	8.9	grey-yellow powder
filling gypsum	900	7–8	insoluble	white powder
atlas gypsum adhesive	1600	8–11	insoluble	grey powder
weber gypsum adhesive	1200	11–12	insoluble	grey powder

4. Experimental results

All studied samples of building materials were characterised with having good wettability. Standard measurement parameters were applied for each sample: 60% of use of pump power and 100% of use of ultrasonic and light power. The time needed to remove air bubbles from the liquid was 10 seconds, and the time of adding each sample to the dispersing unit did not exceed 30 seconds. There were three measurements conducted for each building material (Table 2), making it possible to observe all changes that appeared in the size of the diameters of the particles dispersed in water. For all samples, absorption in the range of between 10 and 15% was achieved. Therefore, the amount of the analysed sample needed for dispersion was optimal.

According to data collected from the graphs and from Table 1, it was found that for building gypsum (Fig. 3a) as well as for Weber gypsum adhesive (Fig. 3c), the diameter sizes of particles increased in subsequent measurements. This indicates the formation of agglomerates and the lumping of particles into larger aggregates, which occurs during the mixing process. Such a phenomenon was not observed during the measurements for filling gypsum (Fig. 3b) and Atlas gypsum adhesive (Fig. 3d), in which the obtained experimental results were almost identical in all three samples.

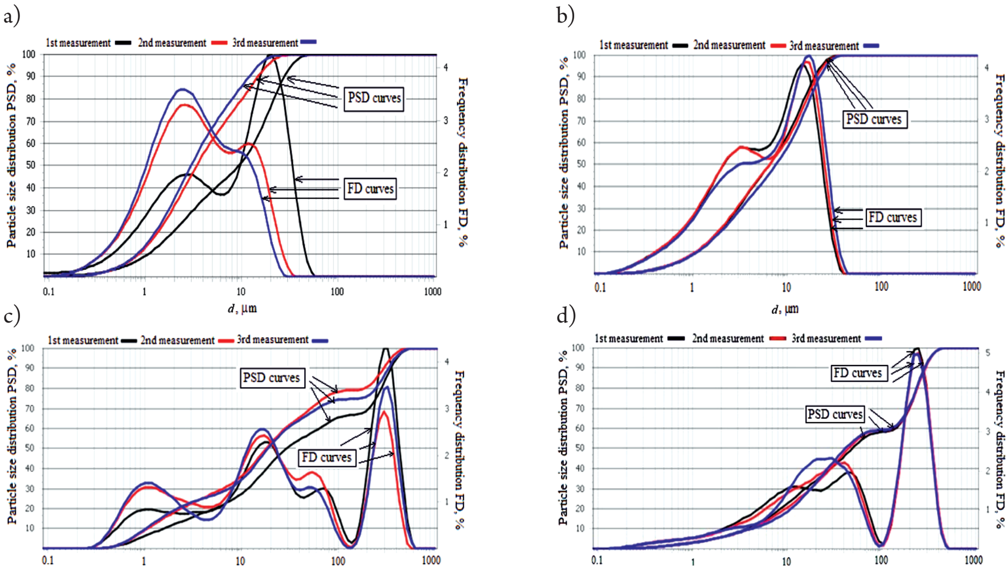

Fig. 3. Graph of dependency PSD = $f(d)$ for the studied building materials: a) building gypsum, b) filling gypsum, c) Weber gypsum adhesive, d) Atlas gypsum adhesive

The studied gypsum adhesives are characterised by greater graining in comparison with building gypsums. It was found that the largest particles for the studied gypsum adhesives amounted to 500 mm, while those for the building gypsums were ten times smaller. Their maximum diameters were just 50 mm (Table 2).

Table 2. Results of laser diffraction analyses of building materials

Studied building materials	**Particle diameter** d **[mm]**	**Average particle size distribution** $f_{ave}(d)$ **[%]**	**Particle size distribution** $f_1(d)$ **[%]**	**Particle size distribution** $f_2(d)$ **[%]**	**Particle size distribution** $f_3(d)$ **[%]**
1	2	3	4	5	6
building gypsum	0.1	1.8	1.2	1.9	2.2
	0.5	5.7	4.8	5.9	6.3
	1	11.7	9.9	12.4	12.8
	2	29.8	21.2	33.2	35.1
	4	50.1	34.8	55.5	59.9
	6	62.0	44.2	67.9	73.8
	8	67.9	48.1	75.4	80.1
	10	72.1	50.1	79.9	86.2
	20	90.6	76.3	96.5	98.8
	25	94.9	85.9	99.0	99.0
	30	98.1	94.3	99.9	100
	45	99.8	99.4	100	100
	50	100	100	100	100
filling gypsum	0.1	0.8	0.7	0.8	0.9
	0.5	2.9	2.9	2.9	2.9
	1	9.5	9.6	9.4	9.5
	2	23.5	20.6	20.4	19.9
	4	37.9	39.1	38.9	35.7
	6	47.8	48.8	48.9	45.8
	8	55.8	57.2	57.7	52.6
	10	60.9	63.2	61.5	58.1
	20	88.6	90.9	88.9	85.9
	25	95.5	96.8	95.8	93.8
	32	99.2	99.6	99.4	98.7
	45	100	100	100	100
Atlas gypsum adhesive	1	2.8	2.8	2.8	2.8
	5	12.6	12.3	12.5	13.1
	10	20.6	22.1	20.2	19.4
	20	33.1	34.0	32.5	32.8
	25	37.6	37.5	37.0	38.2
	32	42.8	41.8	42.4	44.2
	45	50.2	48.5	50.3	51.8
	63	56.2	54.8	56.5	57.2
	90	58.6	58.0	58.9	59.0
	125	59.5	58.9	59.6	59.9
	180	67.0	66.3	66.9	67.9
	250	82.9	82.4	82.5	83.7
	355	97.1	97.0	97.0	97.4
	500	100	100	100	100

tab. 1 cont.

1	2	3	4	5	6
Weber gypsum adhesive	1	8.8	7.1	9.8	9.6
	5	25.4	28.4	27.7	20.1
	10	37.1	35.9	36.6	38.8
	20	48.9	42.3	52.6	51.8
	25	53.9	47.2	57.6	56.9
	32	58.3	51.6	62.1	61.3
	45	63.1	55.9	67.6	65.9
	63	67.9	60.0	73.3	70.5
	90	72.1	64.6	77.9	73.9
	125	73.4	66.6	79.0	74.7
	180	74.4	67.9	79.8	75.3
	250	80.4	75.5	85.2	80.6
	355	92.8	90.5	95.4	92.5
	500	100	100	100	100

The results of the statistical parameters of all four building materials are presented in the figures (Figs. 4–7): diameters of characteristic grains d_{10}, d_{50}, d_{90}, mode and span, where the last parameter is described by equation (4):

$$\text{Span} = \frac{d_{90} - d_{10}}{d_{50}} \tag{4}$$

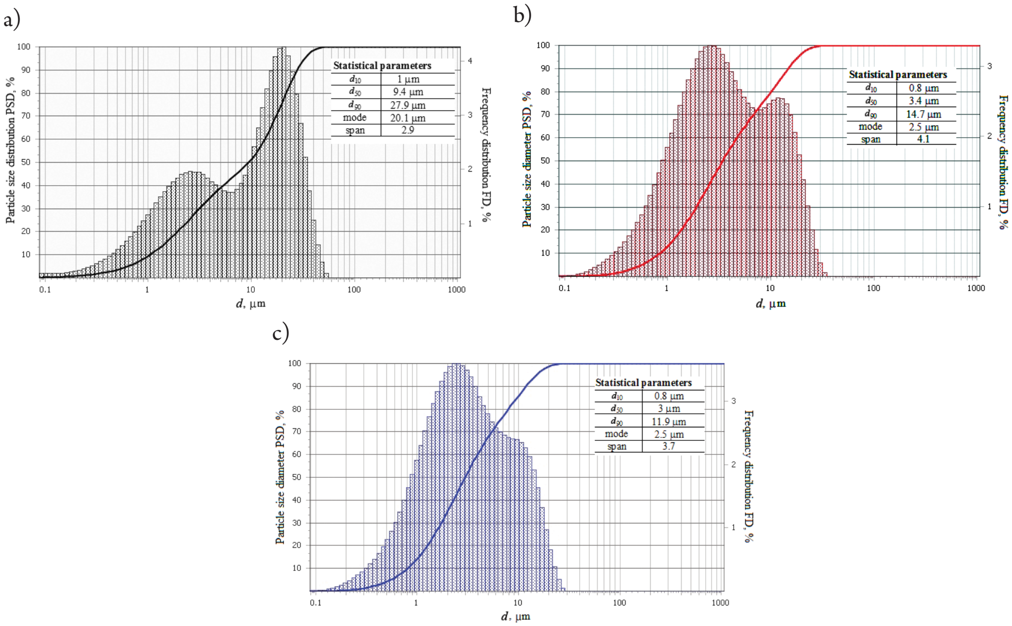

Fig. 4. Sizes of characteristic particles determined with the use of the laser diffraction method in the samples of building gypsum: a) 1st measurement, b) 2nd measurement, c) 3rd measurement

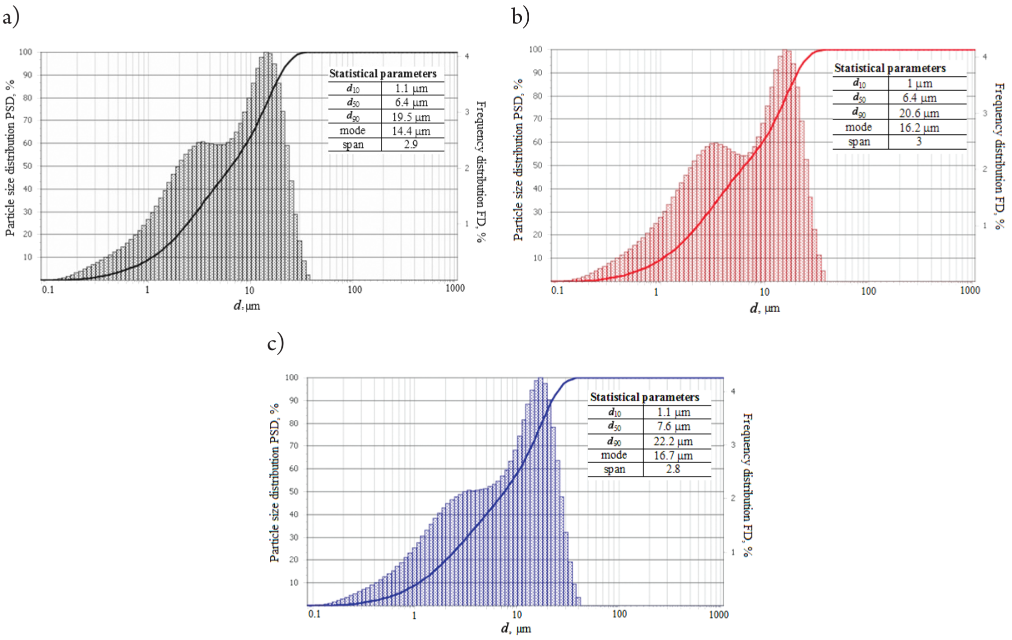

Fig. 5. Sizes of characteristic particles determined with the use of the laser diffraction method in the samples of filling gypsum: a) 1st measurement, b) 2nd measurement, c) 3rd measurement

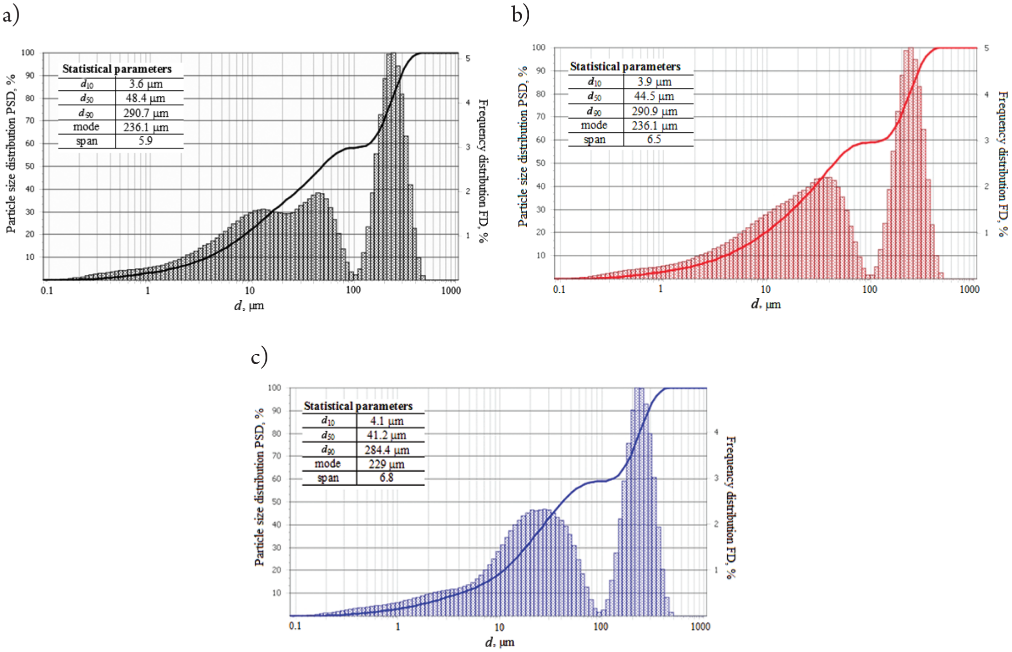

Fig. 6. Sizes of characteristic particles determined with the use of the laser diffraction method in the samples of Atlas gypsum adhesive: a) 1st measurement, b) 2nd measurement, c) 3rd measurement

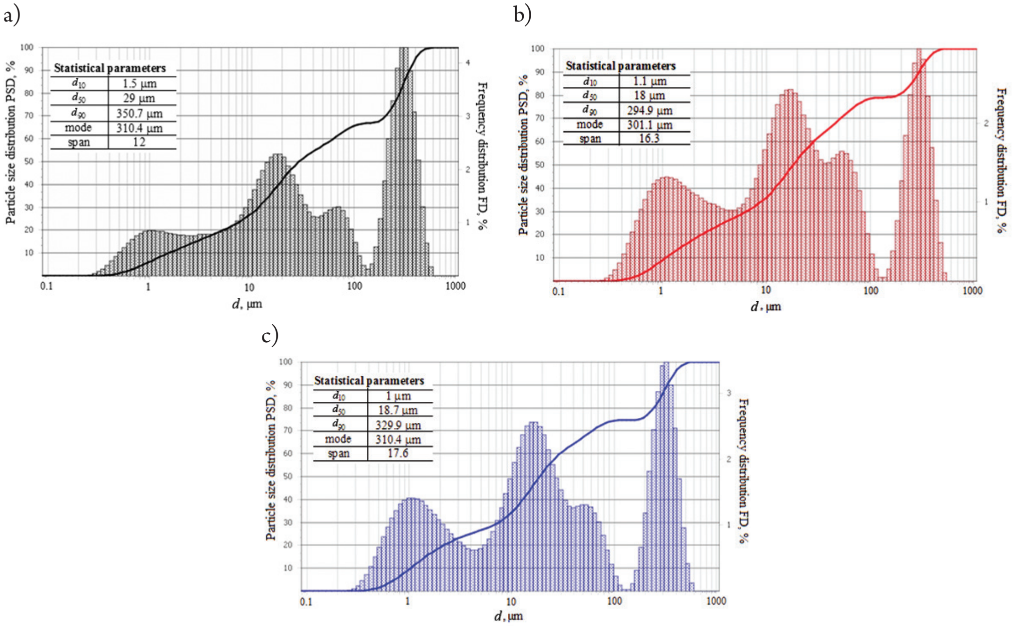

Fig. 7. Sizes of characteristic particles determined with the use of the laser diffraction method in the samples of Weber gypsum adhesive: a) 1st measurement, b) 2nd measurement, c) 3rd measurement

The graphs (Figs. 4–7) show that harmful, highly carcinogenic dust fractions, 2.5 mm in diameter can be found in the studied building materials. Harmful particles in building gypsums are between 2 and 3% of all grains, and between 0.5 and 1% in gypsum adhesives.

Figures 8a–8d show the results of all studied building materials – these present the average particle size distribution as a function of the grain diameter.

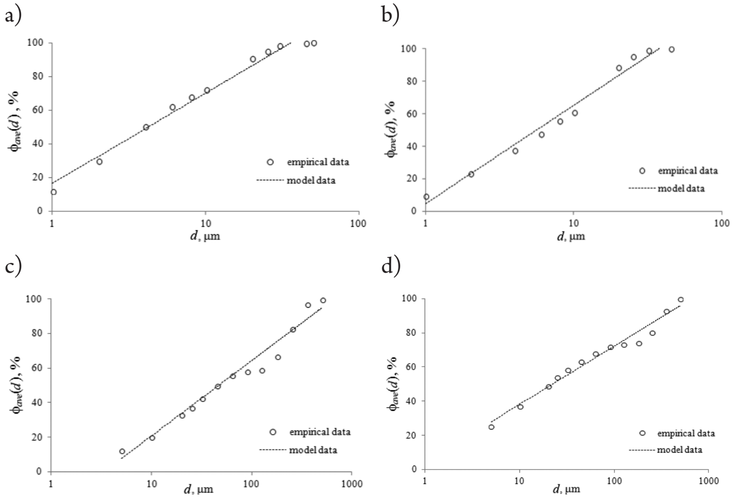

Fig. 8. Approximation results of average particle size distribution of the studied building materials with the use of the laser diffraction method: a) building gypsum, b) filling gypsum, c) Atlas gypsum adhesive, d) Weber gypsum adhesive

For all powder building materials studied in the experiment, general dependency was proposed and is described by equation (5):

$$\varphi_{ave}(d) = A \cdot \ln(d) + B \qquad (5)$$

where:

A, B – constant equation values.

High values of the R^2 parameter were obtained in all cases (Table 3) – this shows a good coherence of experimental results with the proposed mathematical dependency on parameters. Moreover, A and B constants are listed below in Table 3.

Table 3. Constants *A* and *B* of equation (5)

Studied building materials	Constants		R^2
	A	*B*	
building gypsum	23.30	16.40	0.9799
filling gypsum	26.30	4.74	0.9814
Atlas gypsum adhesive	18.90	–22.90	0.9655
Weber gypsum adhesive	14.80	4.31	0.9748

5. Conclusions

A short measurement time is very characteristic of the laser diffraction method applied in the research. Moreover, high reliability and repeatability of this method is an additional advantage.

It was confirmed that the innovative method of inverse Fourier system and laser grain size analysis provides accurate information about the particle size distribution within the range of fine grains (< 500 μm).

It was found that gypsum adhesives have larger grain diameters in comparison with building gypsums. The largest particles were found in gypsum adhesives (500 mm), while those in building gypsums were ten times smaller. Their maximum diameter was up to 50 mm. The filling gypsum was the most stable. The experimental values obtained for this building material were almost identical in three subsequent research samples. It showed only slight changes in the diameters of particles.

All samples were characterised by good wettability and did not require the application of any additional surfactant aimed at reducing surface tension on the edge between the liquid and the solid. In this research, water turned out to be an appropriate dispersing liquid.

It was stated that studied building materials contain powder fractions with grains of 2.5 mm in diameter – these are easily accumulated in the human body and are harmful to human health. Higher amounts of harmful dusts were found in building gypsums, up to 3% of all particles. The conducted experiment has shown that the building materials used in the research are both of good quality and also have good physical properties. For all studied building materials, approximate results of average particle size distribution with the use of laser diffraction method have been shown (Figs. 8) and a general mathematical dependency has been proposed (Equation 5).

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