SIZE ANALYSIS OF SOLID PARTICLES USING LASER DIFFRACTION AND SIEVE ANALYSIS

Michaela Hrnčířová*, Jiří Pospíšil*, Michal Špiláček*

The article compares two methods of determination of the particle size distribution of fine ash. The respective methods compared are the sieve analysis and laser diffraction. The former is one of the oldest methods in use, applicable to size separation of large grain size particles. The latter is a modern advanced method that employs laser diffraction to determine the size distribution. For the measurement itself, the sieve analyser Analysette 3 PRO and the universal laser diffraction device Analysette 22 MicroTec plus, both from the Fritsch company, were applied. The comparison was made by analysing fine ash particles from biomass burners. The particles were in the size range of 0.08 μm to 2000 μm.

Keywords: *solid particles, size distribution, laser diffraction, sieve analysis*

1. Introduction

The particle size determination is a parameter of great importance in materials science, medical science, biology, energetics and other science branches. By the size of a particle, its linear dimension, i.e. length, is understood. The size can be unequivocally determined only for a spherical particle by measuring the particle length that corresponds to its diameter. For non-spherical particles in order for them to be comparable with the spherical ones, there is a need to transform them into spherical ones of a corresponding diameter. The equivalent diameters are the most common transformations. These define the diameters of equivalent spheres that characterise the original non-spherical particles correctly from a physical point of view, depending on further utilization of the determined diameters (the maximum particle dimension, equivalent aerodynamic diameter etc.) The sieve analysis compares the diameter of a circle circumscribed about a non-spherical particle with the equivalent sieve diameter, D_{sieve}, which corresponds to a maximum diameter of a sphere that passes through a particular size of a sieve mesh, the sieve mesh being either square or round. The equivalent diameter, D_{L}, determined by laser diffraction is a diameter of a sphere that would result in the same electronic response to an optical signal in a detector – diffraction pattern – as if it were the case with the non-spherical particle and the same detector. Assuming the Fraunhofer approximation, the diameter should correspond to an equivalent plane diameter of a particle. The equivalent plane diameter, D_{P}, is a diameter of a sphere or circle of the same projected area as the non-spherical particle; the diameter as such depends on the orientation of the particle especially when anisometric.

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2. Methods of particle size determination

Apart from laser diffraction and sieve analysis, there are other methods for the size distribution assessment of solid particles such as sedimentation method, microscopic image analysis etc.

The sedimentation method is one of the traditional methods of particle size analysis applicable to particles 0.02–200 μm. The method determines the particle size indirectly from its sedimentation rate when exposed to gravity or centrifugal force in either liquid or gaseous environment.

The microscopic image analysis of picture analysis is to transform the amount of visual information contained in the picture into simple interpretable quantitative information in form of simple charts or even only few values. With optical microscopy only sizes bigger than few μm can be reliably measured due to the diffraction occurring at the edges of small particles.

The sieve analysis is based on utilization of a set of sieves of a known mesh size. It is mainly used for separating the coarse particles from the fine ones. One of the advantages of this method is the known size range of particles captured on a particular sieve. The main disadvantages are its time demanding character and its damaging effect on fragile particles that affects granularity. During the sieving process, these particles tend to reduce their size dramatically due to abrasion and collisions which leads to inaccurate results that do not reflect the real size distribution of particles in the measured sample.

Laser diffraction is the present most widespread method used for the particle size measurement. The physical principle has been known since the beginning of the 20^{th} century, but it was the invention of suitable laser devices and computers that made it advanced enough. The method supersedes the others in its flexibility and speed nowadays.

3. Description of the experimental apparatus

The sieve analysis uses a set of sieves placed on top of each other, each with a different mesh size that decreases in the direction of the gravitational gradient – sieve stack. For the analysis itself, standardized round sieves with a rectangular mesh made of metal fibres are used. These sieves are usually used for analysing powder samples with particle size ranging from 40 μm to 4 mm. In our case, the dry method of sieving employed ANALYSETTE 3 PRO (Fig. 1) measuring device. It is a standard piece of equipment in which the sieves vibrate noticeably to the naked eye. The device makes use of a cradle to which a stack of sieves is mounted. The cradle is connected to an actuator that controls the amplitude of vibrations and sieving time.

When the sieving process ends, each sieve retains a fraction of the original sample. Each fraction contains particles with the size range that matches the mesh size of the current and previous sieve. These fractions are then weighted, resulting in mass fractions of particular size ranges, which are considered a great advantage of the method over the other ones.

ANALYSETTE 22 MicroTec plus (Fig. 2) is a universal analysing device for particle measurement of suspensions, emulsions and solid matters that utilizes laser diffraction. The device is composed of a central measuring unit and a dispersion unit. There are two semiconductor lasers, each with the output of 7 mW and wavelength of 532 nm and 940 nm respectively, located in the central measuring unit. The measuring range is from 0.08 μm

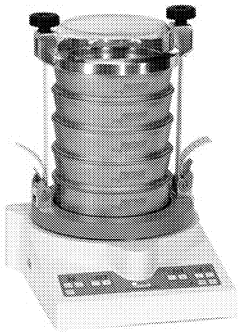

Fig.1: Sieving analyzer

Fig.2: Laser analyser

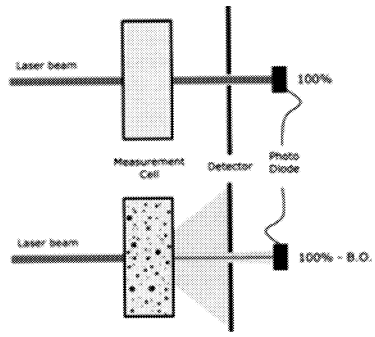

Fig.3: Concept of the laser beam shielding

up to 2000 μm. An ultrasonic water bath comprises the wet dispersion unit and can deliver vibrations at the power output of 50 W of and frequency of 40 kHz to the liquid; organic liquids or inorganic saturated salt solutions may replace water for short term measurements without any risk of damage to the device. The entire device is governed by MaScontrol software.

The laser diffraction method makes use of a laser light which is scattered by a suspension particle, when passing through. Depending on the size and optical properties of the particles, the light scattered at particular spatial angle. A Fourier lens concentrates the scattered light into the focal plane of a detector array which measures the light intensity distribution due to the light scattering. Based on the light distribution obtained, the size distribution of particles is calculated.

The concentration of particles in the sample inserted into the device must be sufficiently small to prevent too much scattering resulting in opacity. On the other hand, too few particles in a sample cause to be too weak the optical signal. The optimum optical thickness lies between 10 and 15 % for the wet dispersion unit used. The unit commences the measurement process when the sample concentration is large enough. Fig. 3 illustrates the shielding effect of a sample on a laser beam. There is no sample inserted into the device in the upper figure thus 100 % light intensity is captured by the photo diode. The lower figure depicts what happens to the laser beam when a sample is inserted into the device. For optimum evaluation of the particle size distribution, both Mie solution to Maxwell's equations (small particles) and Fraunhofer approximation (particles of a size much larger than the light wavelength) are applied.

4. Experiments and result

Two types of ash were examined both produced by biomass furnaces. The first one (P1) came from Dakon Damat Pyro 20G furnace, which was fuelled with spruce and pine logs. The ash was taken from the furnace ash pan. The second one (P2) resulted from combustion of walnut wood chips in Slokov Variant SL 22D furnace. Further experiments were conducted on the ash collected from the bed of a fluidized-bed boiler after the burn out and cooling down. These samples chiefly consisted of an inert material – sand.

4.1. Assessment of ash samples

Sieving analyser

First, both samples were sieved in order to remove coarse particles. Multiple sieving courses were performed with gradually increased amplitude of vibrations and sieving times. The ash sample P1 underwent the sieving procedure three times (subsamples 1, 2 and 3) unlike the ash sample P2, which was processed twice (subsamples 4, 5). Nine sieves with different mesh sizes were used for the analysis. Meshes with the size of 2 mm and 1 mm were used to remove the coarsest particles. After this, the samples went through the rest of the sieve stack with sieve sizes of 500 μm, 200 μm, 160 μm, 125 μm, 90 μm, 63 μm and 45 μm. The masses retained on sieves below 500 μm (included) were then weighted and used for a detailed analysis employing the laser analyser.

Tables 1 and 2 indicate the mass fractions as captured on a particular sieve. The charts in figures 4 and 5 show the ash size distributions for samples P1 and P2 which correspond to the data stated in tables 1 and 2.

It is apparent from the charts that repeated sieving at gradually increased amplitude, interval and overall sieving time cumulatively increases the weight for sieves with smaller mesh. The presumption is that the particles undergone abrasion which is causing breakup into bigger amount of smaller particles. Input values for each measurement are listed in table 3.

	x [μm]	500	200	160	125	90	63	45
P1, sample 1	m [g]	7.9844	15.2033	17.7154	18.3719	18.6402	18.7095	18.7440
P1, sample 2	m [g]	2.1791	6.1464	8.1324	10.3205	13.0092	16.0908	18.0090
P1, sample 3	m [g]	1.6208	7.3865	9.0455	12.7262	15.2944	17.1447	18.1217

Tab.1: Cumulative mass of sample P1 (sieve analyser)

	x [μm]	500	200	160	125	90	63	45
P2, sample 4	m [g]	4.3596	8.2407	9.7828	12.6219	14.0381	14.7743	15.0855
P2, sample 5	m [g]	3.5827	7.4638	10.2013	14.6475	16.0726	16.9372	17.3228

Tab.2: Cumulative mass of sample P2 (sieve analyser)

	unit	P1 sam. 1	P1 sam. 2	P1 sam. 3	P2 sam. 4	P2 sam. 5
Initial weight	[g]	20.002	20.009	20.001	30.005	30.003
Period time	[min]	1	3	5	5	5
Total time	[min]	1	3	5	1	5
Amplitude	[–]	0.2	0.6	0.8	0.5	0.5

Tab.3: Input values for the sieve analysis

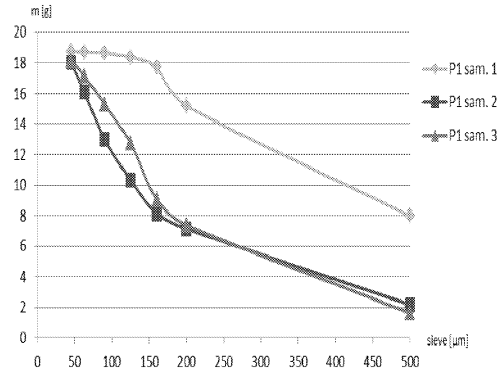

Fig.4: Size distribution of ash sample P1

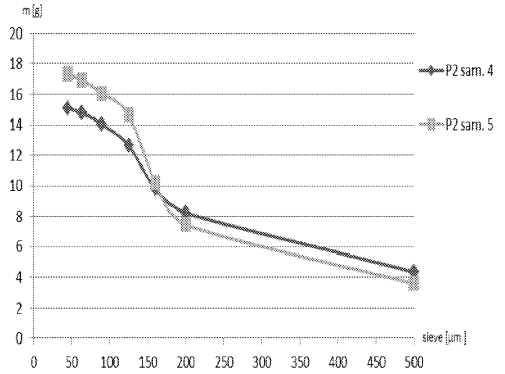

Fig.5: Size distribution of ash sample P2

Laser analyser

The laser analyser used displays the measurement results in the form of data sheets containing relative frequencies of the particular size range of particles. All the data acquired are illustrated diagrammatically in figures 6 and 7.

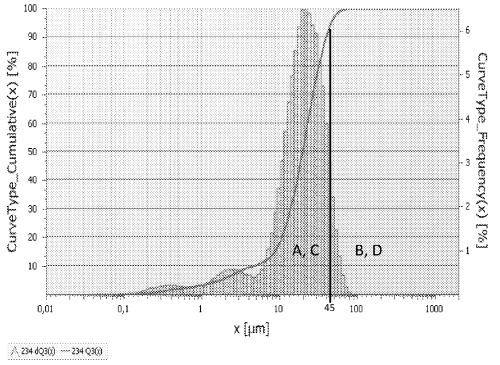

Fig.6: Ash P1, sample 2, 45 μm

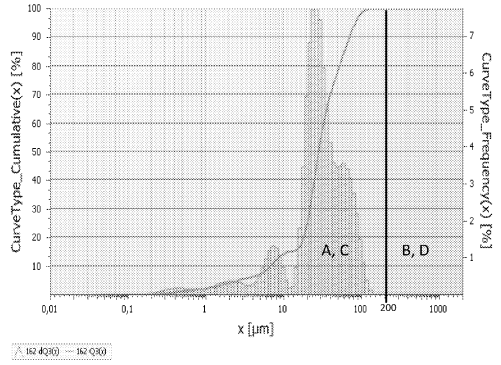

Fig.7: Ash P1, sample 1, 200 μm

At figures 6 and 7 are the results of laser diffraction analysis. Specifically the figure 6 contains information about specimen that was retrieved from a sieve with 45 μm mesh. According to the setup of the sieve analyser this specimen should contain particles the same or bigger than 45 μm up to 63 μm that is mesh size placed above the 45 μm mesh sieve. From the graph it is clear that the specimen contains a big amount of particles smaller than 45 μm. Logical assumption is that the small particles agglomerate and then remains on sieves with bigger mesh. This leads to distortion and inaccurate results. As was mentioned above with more intensive sieving leads into a better particle separation. However, a mechanical abrasion occurs too and the particles break up leading into distortion as well. With the sieve analysis, each specimen must be approached individually. Unlikely, when wet dispersion is used the agglomerates in the specimen are separated by ultrasound and all and each ash particles are detected. The resulting graph shows that the specimen contains a small amount of particles bigger than 63 μm. These particles get in the specimen due to their shape that allowed them to fall through the mesh.

Similar result is shown in Fig. 7 which represents fraction retained on the sieve of the mesh size of 200 μm. The only difference is there were no particles greater than 200 μm left

on the sieve found by the laser analyser. All the particles as identified by the laser analyser were smaller than the sieve size. Similar results were obtained for most of the ash samples analysed.

In table 4 are listed percentage results from the measurement from laser diffraction using values when after each executed analysis existed particles bigger than the respective mesh (see figure 6). Value A represents number portion of particles detected as smaller than respective mesh from which the sample was taken. Opposite is the value B shows the number portion of bigger particles. Both values were acquired from graph belonging to each sample. All acquired data were transformed in weight portions (C, D [%]) with premise that all particles have spherical shape with density of $1200\,\mathrm{kg/m^3}$. At figures 8 and 9 are graphs that show dependence of number (B [%]) and weight portion (D [%]) to respective mesh size.

Sample	Meas. No.	Mesh	Number portion		Weight portion		Sample	Meas. No.	Mesh	Number portion		Weight portion	
		[µm]	A [%]	B [%]	C [%]	D [%]			[µm]	A [%]	B [%]	C [%]	D [%]
2	1/1	45	82	18	54	46	5	1/1	500	78	22	6	94
2	1/2	45	91	9	56	44	5	1/2	500	79	21	37	63
2	1/3	45	88	12	56	44	5	2/2	500	84	16	28	72
2	1/4	45	87	13	53	47	5	1/1	200	85	15	19	81
2	2/4	45	88	12	52	48	5	2/1	200	90	10	16	84
3	1/1	45	87	13	54	46	5	1/2	200	85	15	17	83
3	2/1	45	88	12	54	46	5	2/2	200	89	11	15	85
3	3/1	45	88	12	53	47	5	1/1	45	68	32	42	58
4	1/2	500	87	13	28	72	5	2/1	45	75	25	40	60
4	1/3	500	85	15	36	64	5	1/2	45	80	20	36	64
4	1/1	200	87	13	15	85	5	1/3	45	80	20	60	40

Tab.4: Non-zero relative frequencies and mass fractions of samples assessed

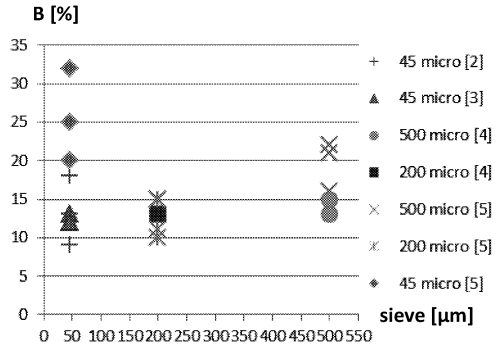

Fig.8: Relative frequency of particles greater than the mesh size of a corresponding sieve

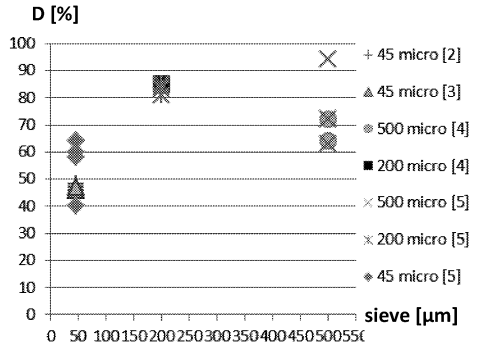

Fig.9: Mass fractions of particles greater than the mesh size of a corresponding sieve

The ash samples analysed show there are large amounts of fine particles in the individual samples. By converting the frequencies into mass fractions, the dominant effect of relatively small number of large particles on the results obtained by sieving becomes apparent. The ash samples analysed exhibit significant cohesion of ash particles which leads to over estimation of the ash mass fractions as obtained from the sieves of large mesh sizes. Further test proved inefficient in removing the over estimation drawback despite repetitive screening and/or more intense shaking of the sieve stack.

With respect to the ash particles cohesion, it seems that a high-quality dispersion of the sample in the liquid bath, which prevents particles from agglomerating although not

from disintegration of the fragile ones, is the key factor for laser diffraction when compared with the sieve analysis. The accuracy of laser diffraction as well as the device was validated against comparative measurement performed with the use of calibrated particles supplied by Fritsch.

Fig.10: Ash P1, sample 2, 200 μm

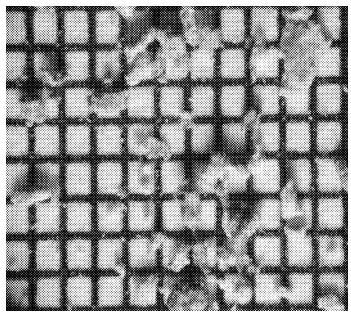

Fig.11: Ash P2, sample 5, 200 μm

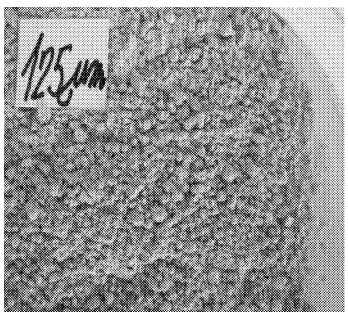

Fig.12: Ash P2, sample 5, 125 μm

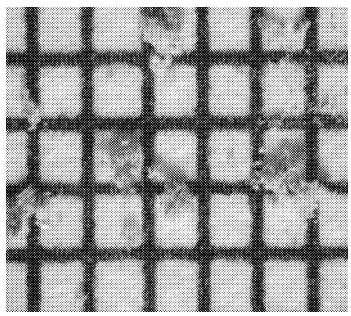

Fig.13: Ash P2, sample 5, 125 μm

Fig. 10 shows remnants of a sample on the sieve of the mesh size of 200 μm after sieving. A naked eye inspection identifies two distinct particle sizes there. The larger ones are supposed to have their size greater or equal to the sieve size of 200 μm. The much smaller ones just did not drop through the sieve. There is an ash sample of the size of 200 μm in Fig. 11 as seen through a microscope. The microscopic view revealed the presence of small particles. It displays them the same way as Fig. 13 does, i.e. sprinkle over a grid of lines 0.05 mm thick and 1 mm apart. Particle agglomerates are also noticeable in Fig. 12 which captures finer fraction of the ash sample. Because of the particles cohesion, the dry dispersion is not perfect which influences results of the sieve analysis. Fig. 13 shows particle agglomerates at a magnification of 100×.

4.2. Inert material experiment

Based on the above mentioned results, it is clear the greatest trouble of ash particles analysis by means of sieving lies in poor dispersion, which causes the fine particles to stick the large ones and/or to agglomerate. This chapter brings up a comparison with an analysis of fine particles which are assumed not to cluster or disintegrate due to mechanical abrasion. The sample particles originate from a fluidized- bed boiler collected after the fuel had burnt out and cooled down. The major part of the sample resembles an inert material – sand.

Table 5 states the frequencies and mass fractions as obtained from the laser analysis of a 'sand' sample, which experienced the same sieving procedure with the use of the same sieve stack as described in the above chapters. All experiments including this sample were carried out under the same conditions as was the ash sample.

The assessment of the particle size distribution of the sample with the use of laser diffraction identified some amount of particles greater than the corresponding mesh size in all cases. Furthermore, the frequency analysis of particles greater than the mesh size indicated there was 37.8 % of a large particle on average in the sample. If the sample is evaluated based on the mass of individual fractions, we find out the particles greater than the mesh size carry 97.8 % of the sample mass on average. It can be said that the particles greater than the mesh size that were captured represent the major mass of the sample; the mass associated with the smaller particles is below 5 % of the sample.

Sample	Meas. No.	Mesh	Number portion		Weight portion		Sample	Meas. No.	Mesh	Number portion		Weight portion	
		[μm]	A [%]	B [%]	C [%]	D [%]			[μm]	A [%]	B [%]	C [%]	D [%]
6	1/1	500	41	59	1	99	6	1/1	125	78	22	3	97
6	2/1	500	48	52	1	99	6	1/2	125	73	27	3	97
6	1/2	500	31	69	0	100	6	2/2	125	72	28	2	98
6	1/3	500	41	49	1	99	6	1/1	90	72	28	3	97
6	2/3	500	45	55	1	99	6	2/1	90	76	24	2	98
6	1/1	200	60	40	0	100	6	1/1	63	74	36	5	95
6	2/1	200	71	29	2	98	6	2/1	63	73	37	5	95
6	1/2	200	68	32	3	97	6	1/1	45	64	36	2	98
6	1/1	160	68	32	3	97	6	2/1	45	58	42	2	98
6	2/1	160	78	22	2	98							

Tab.5: Frequencies and mass fractions of sand sample

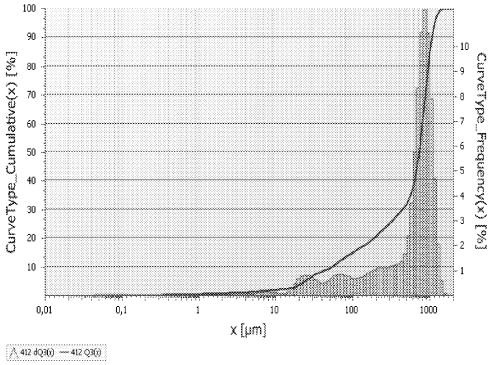

Fig.14: Sand, 500 μm

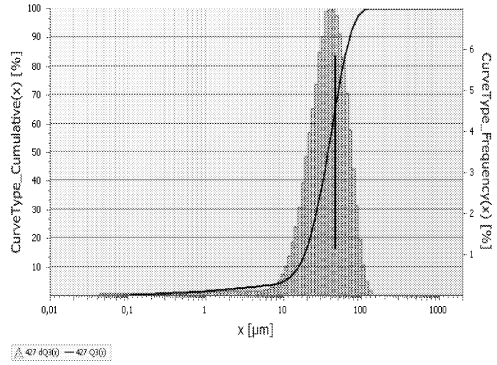

Fig.15: Sand, 45 μm

The laser analysis results of sand are illustrated by Fig. 14 and 15. The former represents the fraction retained on the sieve size of 500 μm. Notice that almost 70 % of the particles in the sample were greater than the sieve size. Although the finer particles are more frequent in Fig. 15, the major mass (95 %) is still carried by the particles greater than 45 μm.

A complete overview of the results is given in table 6 (all zero and non-zero frequencies and mass fractions) for comparison. The data were assessed using the same procedure as it was the case with ash sample P2, subsample 5 (see chapter 4.1). When looking at the data in table 6, it is clear the laser diffraction identified fraction of particles greater than the respective mesh size is both in frequencies and mass fractions significantly smaller for the ash sample P2, subsample 5, than for a sand sample. Moreover, particles greater than the mesh size were not identified in many cases at all.

Sample	Meas. No.	Mesh	Number portion		Weight portion		Sample	Meas. No.	Mesh	Number portion		Weight portion	
		[μm]	A [%]	B [%]	C [%]	D [%]			[μm]	A [%]	B [%]	C [%]	D [%]
5	1/1	500	78	22	6	94	5	1/1	125	100	0	100	0
5	1/2	500	79	21	37	63	5	2/1	125	100	0	100	0
5	2/2	500	84	16	28	72	5	1/1	90	100	0	100	0
5	3/2	500	90	10	100	0	5	2/1	90	100	0	100	0
5	1/1	200	85	15	19	81	5	1/1	63	100	0	100	0
5	2/1	200	90	10	16	84	5	2/1	63	96	4	100	0
5	1/2	200	85	15	17	83	5	1/2	63	94	6	100	0
5	2/2	200	89	11	15	85	5	2/2	63	99	1	100	0
5	1/1	160	100	0	100	0	5	1/3	63	97	3	100	0
5	2/1	160	100	0	100	0	5	1/1	45	68	32	42	58
5	3/1	160	100	0	100	0	5	2/1	45	75	25	40	60
5	1/3	160	100	0	100	0	5	1/2	45	80	20	36	64
5	2/3	160	100	0	100	0	5	1/3	45	80	20	60	40

Tab.6: Frequencies and mass fractions of P2 samples, subsample 5

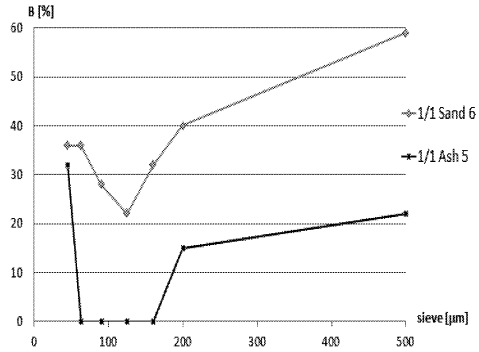

Fig.16: Mesh size effect on agglomeration of ash P2, subsample 5, and 'sand', particle size distribution against frequency

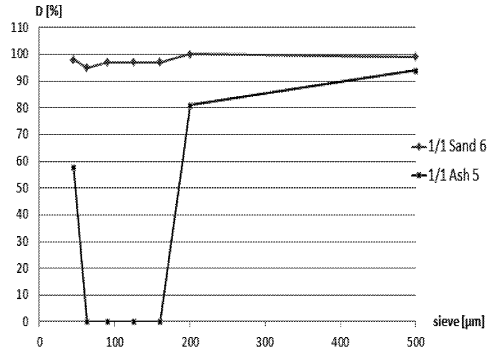

Fig.17: Mesh size effect on agglomeration of ash P2, subsample 5, and 'sand', particle size distribution against mass fraction

Fig. 16 and 17 express graphically the obtained frequencies and mass fractions of solid particles which size exceeds the mesh size of a respective sieve as identified by laser diffraction for both the ash and sand samples. By comparing the data, it is evident the mass fractions of particles acquired for a sand sample are greater than the respective sieve size is virtually independent of the sieve size. The frequencies of such particles tend to decrease along with the decrease of the mesh size. The ash sample shows an entirely irregular course both in frequency and mass fraction data, containing even zero frequencies and fractions; therefore it is impossible to comment on the data correctly.

5. Conclusion

The sieve analysis is a commonly used method of size distribution assessment of solid residues of combustion processes both for its low financial and temporal cost. Its correct application is closely related to the origin of solid residues. The fine fraction of combustion products comprise incombustible fuel residues (ash) and small fractions of inert substances especially used in fluidized-bed boilers to create a stable fluidized-bed (sand), and/or applied to absorb selected pollutants (limestone). Hence it creates a complex set of particles, containing all types of particles of specific proportions. When using the sieve analysis it is good to know the origin and compositions of the tested sample.

The experiments performed rendered the sieve analysis to be of high accuracy (97 %) when assessing gravimetrically the size distribution of the inert mineral material (sand) of

granularity ranging from 45 μm to 500 μm. On the contrary, the method proved unsuitable for fine fractions of ash coming from organic fuel combustion (biomass). Such fine ash particles tend to agglomerate, stick together and break up significantly while being sieved. For that reason, the results of the sieve analysis are significantly inaccurate as particular values depend on specific ash properties. For the ash samples the inaccuracy of measurement using sieve analysis nears 100 %.

Usage of modern method for determination of size distribution of solid particles based on laser diffraction seems like a more appropriate for analysis of ash particles than the sieve analysis. Nevertheless, also with this method mechanical degradation of sample occurs when the particles are being dispersed by the ultrasound in water bath. Fine ash particles partially break up and this process is significantly supported by the water environment in which the particles must by dispersed. The most appropriate approach is to use a combination of both methods.

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