CHEMICAL ENGINEERING TRANSACTIONS
VOL. 92, 2022
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it
Guest Editors: Rubens Maciel Filho, Eliseo Ranzi, Leonardo Tognotti

ISBN 978-88-95608-90-7; **ISSN** 2283-9216
DOI: 10.3303/CET2292008

Particle Size Distribution Analysis of Beech Chips Depending on the Measurement Method

Peter Peciar[a*,b], Kristian Jezsó[a], Michaela Kohútová[a], Adam Guštafík[a], Lukáš Krátky[b], Roman Fekete[a,b], Tomáš Jirout[b], Marián Peciar[a]

[a] Slovak University of Technology in Bratislava, Faculty of Mechanical Engineering, Institute of Process Engineering, Námestie Slobody 17, 812 31 Bratislava 1, Slovakia
[b] Czech Technical University in Prague, Faculty of Mechanical Engineering, Department of Process Engineering, Technická 4, 166 07 Praha 6, Czech Republic
peter.peciar@stuba.sk

Biomass products are used as an energy source in many fields of industry and households. One such source can be pellets or briquettes made of wood chips, ideally made of waste wood. The final quality of these products is affected by many parameters, not only the process (production) parameters but also the material properties are important. A common aim of the research is to maximize the calorific value and minimize the emission of greenhouse gases, which is mainly influenced by the particle size distribution of the raw material. A mixture containing very fine grains will not allow the flow of oxygen, which will make it more difficult to burn the briquette, and conversely, if the percentage of the coarse fraction is high, there will be a larger proportion of oxygen, causing a greater emission of contaminating gases. In addition, particle size distribution may also affect the production of briquettes and pellets, and therefore such analysis is necessary before the wood chips are processed.
In the case of wood chips, the irregular–flaky shape is not the most ideal in terms of analysis. During the measurement, there is an effort to characterize each particle with one dimension. There are many ways to achieve this one dimension, while the result of each method of analysis may be different. For this reason, it is necessary to conduct a series of experiments on several devices to evaluate these measurements and compare the results. The first device used is Malvern's Mastersizer 3000, which uses the laser diffraction technique to measure the particle size. The second device used is Malvern's Morphologi G3, which measures the size and shape of the particle by static image analysis. The third usable device is Microtrac's PartAn 3D, which uses the dynamic image analysis method. The sieve analysis with the Retsch AS200 is employed to compare the results by laser and optical methods.

1. Introduction

Powder properties such as size, shape, density of the individual particles, particle size distribution, moisture content and porosity of the bulk material play a crucial role in the processing technology. On the basis of these properties, it is possible to design process equipment and determine the quality of the final product. In most cases, the individual particles forming a particulate material need to be characterized by a single dimension, from which the particle size distribution of the bulk material can be determined. This can be done accurately only in the case of spherical particles, because for such particles there exists one general dimension which will be the same in every direction, and that is diameter (Rhodes, 2008). In any other case a particle can be only described by multiple dimensions. For this reason, there have been introduced so-called equivalent diameters, which help to characterise a particle by a single dimension derived from some measurable property of the material (Peciar et al., 2021). In the past, several methods were developed (Masuda et al., 2006) capable of measuring a given property of the particles and, by calculation, obtaining the dimension by which the particle is characterized. The results of the different measurement methods lead to the same results only in the case of spherical particles. In the case of non-spherical particles, different methods show different results. The aim of the current paper is to show these differences.

Paper Received: 25 January 2022; Revised: 3 March 2022; Accepted: 3 May 2022
Please cite this article as: Peciar P., Jezso K., Kohutova M., Gustafík A., Kratky L., Fekete R., Jirout T., Peciar M., 2022, Particle Size Distribution Analysis of Beech Chips Depending on the Measurement Method, Chemical Engineering Transactions, 92, 43-48 DOI:10.3303/CET2292008

2. Particle size analysis methods

In this section, the measurement methods used will be briefly described. The measurements were performed using four different devices, two of them working on the optical basis (Morphologi G3 and PartAn 3D). The Mastersizer 3000 works on a field scanning method basis and the fourth method was a classical separation with stack of sieves (Retsch AS 200).

2.1 Malvern Morphologi G3

This device works on an optical principle, with a microscope used to conduct image analysis. At the beginning of the measurement, a small amount of sample is prepared and placed on the glass plate, such that the individual particles do not overlap. This step can also be carried out by using a dispersion unit. The selected area on the measurement plate is then scanned and image analysis is performed (Malvern Instruments Ltd., 2008). When a particle falls on the glass plate, it tends to occupy the position of greatest stability, i.e., when its centre of gravity is at lowest possible position. In this case, the projection onto the plane of the plate is the largest possible. The characteristic dimension of the particle is calculated as projected area diameter, the diameter of a circle with the same area as the 2D image of the particle. The greatest advantage of this method is that it gives a closer view of the mapped particles, which can then be characterised in more detail using various shape factors. On the other hand, the analysis performance (analysed particles per unit time) is quite low, and the particles are analysed only in one plane.

2.2 Microtrac PartAn 3D

The mentioned weaknesses of the Morphologi G3 are eliminated in dynamic image analysis, e.g., with PartAn 3D. This device also works on an optical method, using a high-speed camera to capture 2D views of the particles during their free fall. During the particle´s fall, a free rotational motion is also performed, so for each particle a larger number of images from different views is captured. The evaluation is performed in an identical way to the previous method, i.e., the projected area diameter is calculated, but in this case multiple times for a single particle, in different views. The particle is then characterized by the average of these diameters (Microtrac, 2015).

2.3 Malvern Mastersizer 3000

This device uses the physical principle of laser diffraction to evaluate particle size. Once the laser light hits the particle, it is then scattered. Based on the angle and the intensity of the scattered light, the particle size can be calculated by use of the appropriate theory. In this case, the particle size is calculated, expressed as volume equivalent diameter (Malvern Instruments Ltd., 2013).

2.4 Retsch AS200

One of the oldest methods still used widely for particle size analysis is sieving. Sieves of given mesh sizes are stacked on each other, and the sample is poured on the top one. Due to vibration, the sample is thrown upwards and due to gravitation it falls back down. During the process, those particles that are smaller than the aperture size pass to the next fraction, while particles that are larger stay on the given sieve. The orientation of the particle at the moment of contact with the sieve surface plays a crucial role. The sieving process should be long enough to ensure that the particles have a sufficient number of contacts with the sieve surfaces.

3. Experimental material

The raw material used in these experiments was milled beech chips, made in a knife mill. The adjustable parameters in the milling process were the number of revolutions per minute plus the type and size of the sieve matrix openings. The last mentioned is used to control the size of the product, as the particles can only leave the milling area if they fall through the sieve matrix. The experiments were carried out at two rotor speeds (1500 min^{-1} and 3000 min^{-1}). Two types of matrixes with different sizes were used, with square holes (*HR*) and with trapezoidal holes (*LI*). Each raw material sample was milled twice, first with a matrix with larger openings followed by a matrix with smaller openings. The milling and grinding of cellulose-based materials was also discussed by Peciar et al. (2019). The cumulative distribution of the raw material can be seen in Figure1, with particles from 1 mm to 1 cm present. The analysis of the raw material was performed on a PartAn 3D equipment.

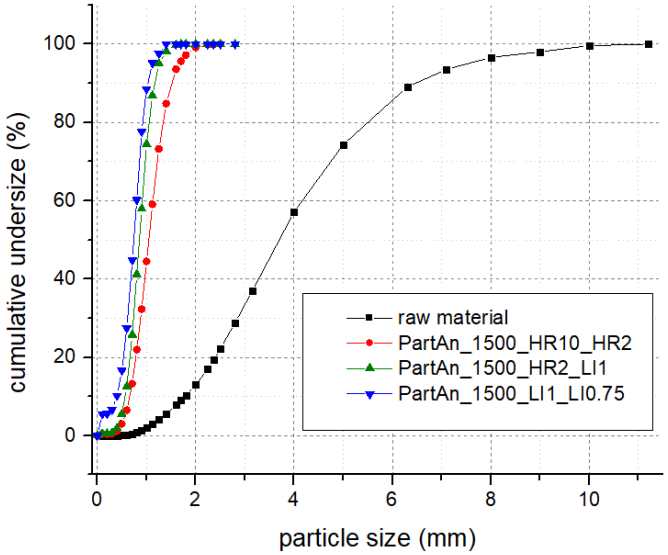

Figure 1: Particle size distribution of the raw material before and after milling at different parameters, as analysed by PartAn 3D

4. Results

The following table shows the details of each sample.

Table 1: Details of individual samples in the milling process

i	*Kinfe mill rotor speed* (min^{-1})	*Type of 1st matrix* $(-)$	*Size of 1st matrix openings* (mm)	*Type of 2nd matrix* $(-)$	*Size of 2nd matrix openings* (mm)
1	*1500*	*HR*	*10*	*HR*	*2*
2	*1500*	*HR*	*4*	*HR*	*2*
3	*1500*	*HR*	*2*	*LI*	*1*
4	*1500*	*HR*	*2*	*LI*	*0.75*
5	*1500*	*LI*	*1.5*	*LI*	*0.75*
6	*1500*	*LI*	*1*	*LI*	*0.75*
7	*3000*	*HR*	*10*	*HR*	*2*
8	*3000*	*HR*	*4*	*HR*	*2*
9	*3000*	*HR*	*2*	*LI*	*1*
10	*3000*	*HR*	*2*	*LI*	*0.75*
11	*3000*	*LI*	*1.5*	*LI*	*0.75*
12	*3000*	*LI*	*1*	*LI*	*0.75*

Based on these data, sample 1 is marked as 1500_HR10_HR2. Figure1 shows the effect of milling on the particle size distribution of the raw material. The product contains significantly smaller particles than the raw material, and the influence of the used matrixes are also observable. The sieve with the smaller openings results in finer particles.

This paper is based on the results of Fekete et al. (2021), in order to analyze milled beech chips using several methods and to compare the obtained results. Figure 2 shows the results of the analyses in the case of sample 1500_HR10_HR2.

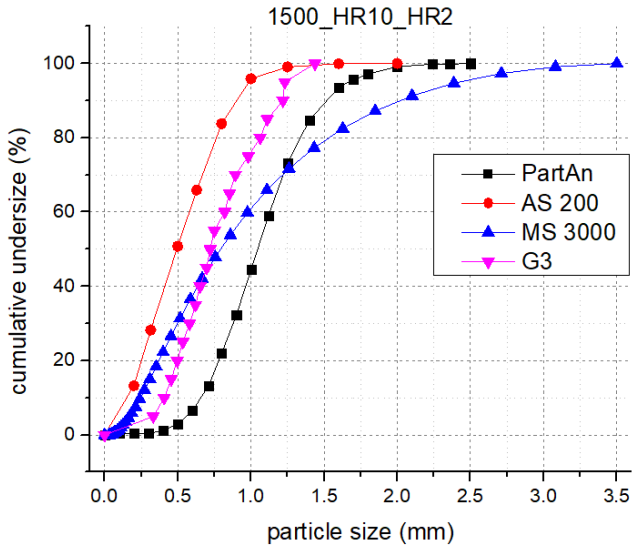

Figure 2: Analysis of the particle size distribution results under different measurement methods (sample: 1500_HR10_HR2)

At first sight, the individual distributions look different, but they do have a systematic character. The sieve analysis shows the finest distribution. Theory says that by sieving, it is possible to determine the minimum size of the particles. With the appropriate orientation of the particle, it passes through the sieves with descending openings until there is no minimum particle dimension larger than the aperture size of the sieve. The particles must have a sufficient number of contacts with the sieve. Therefore, 2 important requirements must be fulfilled: the length of the sieve analysis must be long enough, and the amount of the raw material must be sufficient (Jezsó and Peciar, 2022). Otherwise, not all particles will have contact with the sieve.
In this case, the results of the sieve analysis should also be in agreement with the sizes of the mill matrix. This is shown in Figure2, since the size of the second matrix was 2 mm in the illustrated example, and according to the sieve analysis, the maximum particle size is about 1.25 mm. The other samples show a similar behavior.
The results of dynamic image analysis (PartAn 3D) show the coarsest distribution. The results of the static image analysis (G3) are between the curves of AS 200 and PartAn 3D. The cumulative distribution curve of laser diffraction (MS 3000) intersects the PartAn 3D curve at around 70 %. These trends are typical for each sample. Based on the results from (Fekete et al., 2021), higher rotor speed means finer product. Figure3 shows that this dependency is also demonstrated by other analysis methods.

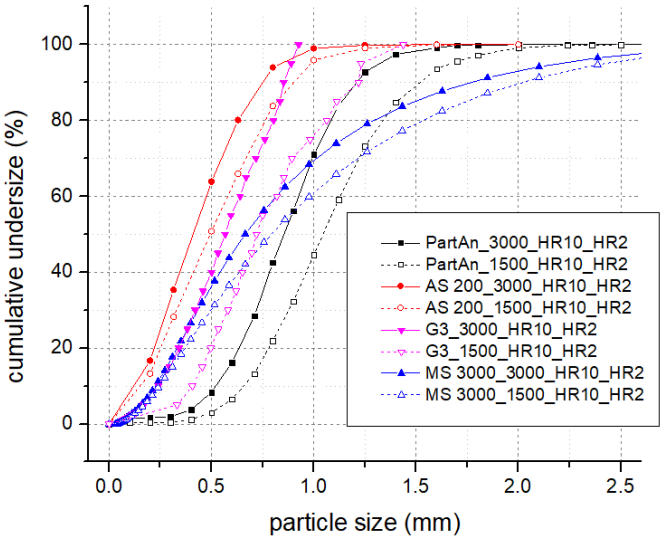

Figure 3: Comparison of the cumulative undersize of sample HR10_HR2 at rotor speeds of 1500 min^{-1}and 3000 min^{-1} by different analysis methods

The solid curves in Figure3 show the cumulative undersize distribution of the milled product at higher rotor speeds, the dashed lines at lower speeds. Every method confirms the prediction that higher rotor speed means finer product.
In the mentioned paper (Fekete et al., 2021), it was also reported that milling of such wood chips gives a material with linear characteristic in the interval $D_{10} - D_{90}$. D_{10} is the particle size from which 10 % of the particles are smaller. For D_{50} this is 50 %, and for D_{90} it is 90 %. For the three selected samples, these values are shown for all four analysis methods in Figure4.

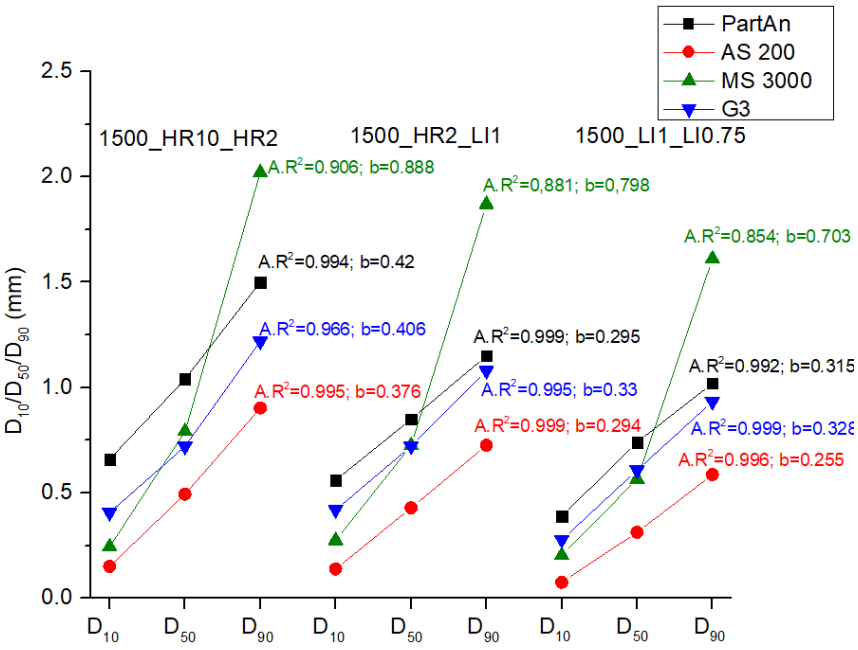

Figure 4: Particle size distribution presented by $D_{10} - D_{90}$ for samples: 1500_HR10_HR2, 1500_HR2_LI1 and 1500_LI1_LI0,75 with statistical indicators

Figure4 is divided into 3 parts. The left part refers to sample 1500_HR10_HR2, the middle part to sample 1500_HR2_LI1 and the right part to sample 1500_LI1_LI0.75. The different colours represent the different analysis methods. Figure4 discusses the linearity of the intervals $D_{10} - D_{90}$ and the similarity of the results within a single sample using linear regression using the equation $y = a + bx$. As mentioned above in Figure2, the different methods show different particle size distribution for the non-spherical particles.
The similarity of the results from the individual samples can be analysed by using statistical indicators. Within one sample, the similarity is expressed by the parameter b, which is the slope of the regression line. In the case of the 1500_HR10_HR2 sample when analysed using AS 200, the slope of the regression line joining D_{10} and D_{90} is $b = 0.376$ with adjusted $R^2 = 0.995$ $(A.R^2)$, confirming the assumption of linearity. When analysed using G3, $b = 0.406$ and $A.R^2 = 0.966$ and when analysed using PartAn 3D, $b = 0.42$ and $A.R^2 = 0.994$. The slope values in these cases are almost identical. Mathematically evaluated: the mean value of $\bar{b} = 0.401$ with standard deviation $S = 0.0183$, which means a relative standard deviation (RSD) of 4.56 %. This confirms the similarity of the AS 200, G3 and PartAn 3D methods in the case of sample 1500_HR10_HR2. Regarding MS 3000, neither similarity to the other methods $b = 0.888$ nor linearity $A.R^2 = 0.906$ is met. This may be because this method works on a completely different evaluation principle, based on the volumes of the individual particles.
The values of the slopes, regression accuracy, standard and relative deviations for the other samples are shown in Table 2.

Table 2: Statistical indicators of individual samples

	AS 200		G3		PartAn 3D		MS 3000			
i	b_i	$A.R_i^2$	b_i	$A.R_i^2$	b_i	$A.R_i^2$	b_i	$A.R_i^2$	$\bar{b}_i$*	RSD* (%)
1	*0.376*	*0.995*	*0.406*	*0.967*	*0.420*	*0.994*	*0.888*	*0.909*	*0.401*	*4.58*
2	*0.341*	*0.998*	*0.375*	*0.966*	*0.405*	*0.997*	*0.864*	*0.916*	*0.374*	*6.95*
3	*0.294*	*0.999*	*0.330*	*0.995*	*0.295*	*0.999*	*0.798*	*0.885*	*0.306*	*5.56*
4	*0.305*	*0.996*	*0.392*	*0.999*	*0.360*	*0.998*	*0.783*	*0.880*	*0.352*	*10.17*
5	*0.310*	*0.998*	*0.428*	*0.989*	*0.355*	*0.999*	*0.858*	*0.908*	*0.364*	*13.29*
6	*0.256*	*0.997*	*0.328*	*0.999*	*0.315*	*0.992*	*0.703*	*0.859*	*0.299*	*10.53*
7	*0.316*	*0.996*	*0.306*	*0.997*	*0.340*	*0.999*	*0.773*	*0.887*	*0.321*	*4.42*
8	*0.379*	*0.998*	*0.316*	*0.995*	*0.520*	*0.996*	*0.918*	*0.935*	*0.405*	*21.11*
9	*0.252*	*0.999*	*0.276*	*0.994*	*0.310*	*0.999*	*0.617*	*0.886*	*0.279*	*8.57*
10	*0.306*	*0.984*	*0.315*	*0.999*	*0.395*	*0.999*	*0.703*	*0.879*	*0.339*	*11.77*
11	*0.251*	*0.997*	*0.366*	*0.993*	*0.475*	*0.956*	*0.639*	*0.868*	*0.364*	*25.06*
12	*0.205*	*0.998*	*0.274*	*0.999*	*0.280*	*0.999*	*0.480*	*0.891*	*0.253*	*13.55*

*marked values are calculated without MS 3000

Table 2 shows that the regression accuracy values in the AS 200, G3 and PartAn 3D cases are very close to 1. This confirms the linearity in the interval D_{10} – D_{90}. In the case of MS 3000, these values are significantly lower, indicating that the mentioned interval is not clearly linear. This shows that in most cases, a relationship/similarity between the results of the different analysis methods can be found.

5. Conclusions

In the case of particle size distribution analysis of spherical particles using equivalent diameters, it is clear that the measurement method chosen does not significantly affect the results, and that the measuring can be considered to be equal. However, if non-spherical particles, for example particles produced by wood milling, are used for the analysis, the case is different. From the measured data it is clear that for non-spherical particles the measurement method is one of the key parameters. The method of observing the individual particles in the system, as well as the orientation of these particles when they are measured, causes significant differences. This difference will be influenced by shape factors; sieve analysis is influenced by the correct orientation of the particle as well as by the particle flow in the measuring equipment and by the rotation of the particles in free fall. Based on these facts, it is necessary not only to be familiar with the measuring ranges of the individual equipment, as well as their correct operation, but above all with the physical principles on the basis of which they measure. According to these details, an appropriate method can then be chosen.

Acknowledgments

This research was supported by the Ministry of Education, Youth and Sports of the Czech Republic under OP RDE grant number CZ.02.1.01/0.0/0.0/16-019/0000753 “Research centre for low-carbon energy technologies”. The authors wish to acknowledge the Ministry of Education, Science, Research and Sport of the Slovak Republic for the financial support of this research by grant KEGA 036STU-4/2020.

References

Fekete R., Peciar P., Kohútová M., Jirout T., Krátky L., Peciar M., 2021, Flow Properties of Bulk Material as a Product of the Beech Chips Grinding, Chemical Engineering Transaction 86.

Jezsó K., Peciar P., 2022, Influence of the selected sieving parameters on the sieving efficiency of material MCC Avicel PH102, Strojnícky časopis – Journal of Mechanical Engineering 72 (1).

Malvern Instruments Ltd., 2008, Morphologi G3 User Manual, Malvern Instruments Ltd. <federation-fermat.fr/wp-content/uploads/2019/09/Morphologi_G3_User-Manual_MAN0410-1.1_0.pdf >.

Malvern Instruments Ltd., 2013, Mastersizer 3000 User Manual, Malvern Instruments Ltd. <montana.edu/eal-lres/documents/Mastersizer-3000-user-manual-English-MAN0474-2-1.pdf>.

Masuda H., Higashitani K., Yoshida H., 2006, Powder Technology Handbook, CRC Press, Boca Raton, Florida.

Microtrac, 2015, PartAn3D Dry Particle Image Analyzer Operation and Maintenance Manual, Microtrac <microtrac.com/products/particle-size-shape-analysis/dynamic-image-analysis/partan-3d>.

Peciar P., Fekete R., Peciar M., Jirout T., 2019, Milling of Materials on the Base of Cellulose, 27th European Biomass Conference & Exhibition, Lisbon – Portugal.

Peciar P., Juriga M., Guštafík A., Kohútová M., Jezsó K., 2021, Procesné strojníctvo – príklady (Process engineering – exercises), Spektrum STU, Bratislava, (in Slovak).

Rhodes M., 2008, Introduction to Particle Technology, John Wiley & Sons Ltd, Chichester, UK.