Internal particle size distribution of biofuel pellets

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ABSTRACT

Several methods for disintegration of biofuel pellets were tested and compared for their ability to break up the pellets into the original particles of the raw material. Analyses performed on softwood pellets and straw pellets concluded that wet disintegration in water at ambient temperature is insufficient for a determination of the internal particle size distribution of wood- and straw pellets. When the wet disintegration was performed with water heated to the boiling point and coupled with mechanical disintegration in terms of stirring a more complete disintegration of the pellets was obtained. Based on the results obtained in the initial study a round robin was set up including six European laboratories where the selected method was tested. In the round robin test the method combining heated water and stirring of the slurry was tested on solid biofuel pellets produced of comminute straw, deciduous wood and coniferous wood respectively. With the method a satisfactory disintegration was obtained of all three types of pellets.

Further wet disintegration of coniferous pellets was compared to a dry disintegration using a hammer mill. The dry disintegration of the coniferous pellets resulted in smaller particle sizes compared to the wet disintegration using heated water and stirring of the slurry indicating a further disintegration of the original particles in the hammer mill process.

Overall the wet disintegration combined with mechanical impact was found to be the most suitable method for disintegration of solid biofuel pellets. Combined with sieving analysis the method gives realistic image of the internal particle size distribution of solid biofuel pellets.

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1. Introduction

The growing focus on reduction of greenhouse gases emissions, especially carbon dioxide has led an increasing part of electricity producers to convert coal pulverized systems into co-combustion using solid biofuel in the form of comminuted wood- or straw pellets [1]. Co-combustion of biomass and coal are identified as a short term, low risk and sustainable way for reducing emissions of CO_2, SO_x and NO_x [3]. Advantages of co-combustion are several; environmental in terms of substitution of fossil fuels with solid biofuel, technical as boiler efficiency is improved and economical as the fuel costs are reduced [1]. By that, substitution of a part of the pulverized coal by comminute pellets is an excellent possibility for long term sustainable CO_2 reduction [8].

The growing consumption of pellets, both for direct combustion and for suspension combustion have increased the import of pellets from outside Europe. Considering Europe with the formal 15

countries, the use of imported biomass for electricity production could reduce by about 20% the costs related to the achievement of renewable energy use goals [5–7]. In consequence, the transport of solid biofuel pellets appears profitable both on an economical and environmental point of view, even if milling is necessary before combustion in pulverized systems. The milling shall bring the pellets into particles having the sufficient aerodynamic properties. For pulverized systems the particle size distribution of the fuel is a characteristic of main importance as it influences the fuel feeding, the ignition, the amount of unburned particles and the furnace temperature [4,9,10].

The recommended particle size distribution of the fuel for co-combustion varies, Baxter recommend that all particles shall be below 6.34 mm with most of the particles having a size of 3 mm [3]. Even that the specifications of some consumers are more strict [2] the influence of the maximum biomass particle size in co-combustion is not well defined [11,12]. The shape and size distribution of the particles are depending on the milling parameters (hammer or knives, rotation speed, type and diameter of the grids) and on the raw material characteristics. Moreover, the hammer mill design depends on the quality of the pellets as the internal particle size distribution and the desired particle size after milling [18].

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A method based on a wet disintegration of the pellets is commonly used for describing the internal particle size distribution of the pellets. Assuming that the internal particle size distribution of the pellets reflects the particle size distribution of the comminute pellets the method is used to estimate the behaviour of the comminute product in a suspension boiler.

The present study investigates a number of methods to describe the internal particle size distribution of solid biofuel pellets. The methods are based on either wet-disintegrations of the pellets in water and subsequent drying and sieving of the particles or on a dry disintegration by crushing the pellets in a laboratory scale hammer mill and subsequent sieving of the particles. The obtained particle size distributions from the wet-disintegration methods are compared to the particle size distribution obtained with the dry disintegration. For the present used wet disintegration method an initial study was carried out to investigate whether comparable results were obtainable between laboratories and moreover to see whether the method could be improved. The initial study gave rise to changes in the method and a round robin test with six participating laboratories was subsequently conducted for this revised method. The aim of the round robin test was to verify the revised method and to determine the variation in terms of repeatability and reproducibility in the obtained results.

2. Materials and methods

2.1. Test material

All test material origin from commercially produced biofuel pellets acquired from the Danish energy company 'DONG energy'. The pellets had a diameter of 8 mm and a maximum length of approximately 40 mm, the straw pellets being slightly shorter than the wood pellets. For the initial study of the wet disintegration method two types of solid biofuel pellet was chosen. One type was produced of coniferous wood and one type produced of comminuted straw. The dry disintegration method was tested on coniferous wood pellets from the same lot used for the wet disintegration study. For the round robin test three types of solid biofuel pellet was chosen. Beside coniferous wood and straw pellets similar to the types used in the initial study a second type of wood pellets produced of deciduous wood was included in the round robin test. For all tests, samples of 50 kg was acquired and subsequently divided into test portions of 300 g with a riffle divider (18 splits with split size of 28.2 mm).

2.2. Initial wet disintegration study

Three laboratories participated in the initial study of the existing method which consists of a wet disintegration of the pellets and subsequently drying and determination of the particle size distribution of the obtained particles. The disintegration was performed by placing a test portion of 300 ± 10 g pellets and 1200 ± 10 ml demineralised water at ambient temperature in a watertight container with a volume of approximately 2000 ml for 24 h for disintegration. After the disintegration the test portion was transferred to a drying container. To secure that all material was transferred the disintegration container was rinsed with demineralised water into the drying container. The material was dried at 35 ± 5 °C in 24 h and subsequently left for 24 h to equilibrate with the ambient temperature and humidity in the laboratory.

Beside the disintegration with demineralised water at ambient temperature additional tests was performed, using demineralised water heated to boiling point and using mechanical impact as stirring with a spoon respectively to facilitate the disintegration. All types of disintegrations were performed in duplicates.

After the equilibration the test portion was divided into two portions each of 150 ± 10 g by means of a riffle divider. One portion was used for moisture determination [13] and one portion was further divided into two test portions of 75 ± 5 g for particle size determination.

Moisture determination was performed by drying the moisture test portion in a drying cabinet at 105 °C for 24 h [13]. The moisture content of the equilibrated material shall be between 5% and 15% in order to avoid that the particles become brittle or agglomerates.

Duplicate particle size analysis were performed with a Retsch AS 200 control 'g' sieving machine using eight Ø200 mm sieves with an aperture size of (mm) 3.15 (round holes [14]), 2.8, 2.0, 1.4, 1.0, 0.5, 0.25 and 0.125 (mesh wire [15]). For wood pellets the sieving analysis was performed on the full portion of 75 ± 5 g whereas the straw pellets was divided into two approximately equal portions and sieved in two rounds to avoid clogging of the wire gauze. The sieving time was set to 30 min using amplitude of 3 mm which secure that the sieving is complete i.e. the mass change in the sieves between two consecutive sievings is below 0,2% [16].

2.3. Dry disintegration study

The dry disintegration was performed in duplicates using a laboratory hammer mill fitted with six equidistantly pivoting hammers. The rotation axis are mounted on a 10 mm diameter steel disc with the pivoting axe centre located 12,5 mm from the edge of the disc, on the diameter line. The hammers are T shaped, with a square section of 5 mm side; the pivoting part is 37.5 mm long from the centre of the axis to the perpendicular part. The milling chamber has a internal diameter of 190 mm and a 55 mm width. A 160 mm internal diameter and 55 mm width grid perforated by 3 mm round holes is centred in the milling chamber. The rotation speed of the engine axe is 2800 rpm, and the engine has a power of 1.1 kW.

The particle size distribution of the dry disintegrated pellets was determined as described in Section 2.2 but using seven Ø200 mm sieves with an aperture size in mm of 3.15 (round holes [13]), 2.8, 2.0, 1.4, 1.0, 0.5 and 0.25 (mesh wire [14]). The choice of sieves reflects the recommended selection from CEN/TS 15149-2 [17].

2.4. Wet disintegration round robin test

Six laboratories participated in the round robin test. The method used was based on the experience from the initial study. The disintegration was performed by placing a test portion of 300 ± 10 g pellets and 2000 ± 10 ml demineralised water heated to a temperature of 95 ± 3 °C in a container with a volume of approximately 5000 ml and carefully mixing the pellets and water to a slurry by stirring with a spoon. The slurry was left for 24 h where after it was transferred to a drying container. To secure that all material was transferred the disintegration container was rinsed with demineralised water into the drying container. The material was dried for 24 h at 60 ± 5 °C in 24 h and subsequently left in the laboratory for 48 h to equilibrate with the ambient temperature and humidity in the laboratory. All disintegrations were performed in triplicates.

After the equilibration the test portion was divided into two portions each of 150 ± 10 g by means of a riffle divider. One portion was used for moisture determination as described in Section 2.2 and one portion was further divided into two test portions of 75 ± 5 g for particle size determination.

Particle size analyses were performed using the sieving equipment and procedure described in Section 2.3. For the two types

of wood pellets the sieving analysis was performed on the full portion of 75 ± 5 g whereas the straw pellets was divided into two approximately equal portions and sieved in two rounds to avoid clogging of the wire gauze.

2.5. Statistics

For comparisons of the results from the round robin test (2.4) are the 25%-, 50%- and 75% quantiles calculated. The variation in the particle size distribution obtained within and between the laboratories is calculated as the standard deviation:

$$SD = \sqrt{\frac{(x - \bar{x})^2}{(n - 1)}} \tag{1}$$

where x is the value, $\bar{x}$ the mean value, n is the number of values.

All data, which cannot be rejected due to procedure errors, are applied in the calculation of the standard deviations. Thereby no *a priori* identification of outliers is employed since any variation between the distributions is assumed to be within the range that is achievable with the method.

Differences between the calculated quantiles from each laboratory are identified using a one-way ANOVA with following model:

$$Quantile = \alpha_0 + \beta_1 \, value + \varepsilon \tag{2}$$

where α_0 and β_1 are the parameters for fixed effects, ε the $\sim N(0, \sigma_2)$ and *value* the mean of the three replicate measurements of the particle size for the specific quantile.

Standard deviation of repeatability (S_r) and standards deviation of reproducibility (S_R) of the quantiles are calculated with the Mean Square from the one-way ANOVA where:

$$S_r = \sqrt{MS_{withingroups}} \tag{3}$$

The repeatability is calculated under test conditions where independent analysis is obtained using the same method, equipment and operator in the same laboratory on identical samples. The repeatability limit or repeatability (r) is the maximum value under which is situated, with a 95% probability, the absolute value of the difference between two results obtained in conditions of repeatability.

$$S_R = \sqrt{MS_{betweengroups}} \tag{4}$$

The reproducibility is calculated under test conditions where independent analyses are obtained using the similar method and equipment in different laboratories on identical samples. The reproducibility limit or reproducibility (R) is the maximum value under which is situated, with a 95% probability, the absolute value of the difference between two results obtained in conditions of reproducibility.

3. Results and discussion

3.1. Initial wet disintegration study

Solid biofuel pellets produced of coniferous wood and straw were tested at three different laboratories using the existing method and modifications hereof. The obtained cumulative particle size distributions indicate that there is a considerable influence from the applied disintegration method on the distribution (Figs. 1 and 2). For the wood pellets it is evident that using demineralised water at room temperature result in an insufficient disintegration of the pellets. Water at room temperature is prescribed in the existing method. Applying heated water and mechanical impact through stirring of the slurry improve in various degrees the disintegration of wood pellets. However, there is some overlap in the particle size distribution curves between the three methods i.e. water at room temperature, heated water and water at room temperature combined with stirring of the slurry (large circle in Fig. 1). Combining the two effects, heated water and stirring of the slurry, resulted in a more complete disintegration which is seen as the thick solid distribution curves which are separated in the upper part of Fig. 1 (HW + STG, the small circle).

The disintegration of the straw pellets was much more complicated and some of the experiments were abandoned. In some of the experiments the material absorbed all of the 1200 ml water and in several of the experiments was found agglomerated particles in the material for the sieve analysis. Compared to the tests using water at ambient temperature (Fig. 2, W), the tests applying heated water (HW) improved the disintegration. Opposite the tests using water at ambient temperature and stirring (W + Stg) only improved the disintegration in one of the five performed tests. Finally the one test applying both heated water and stirring (HW + Stg) showed the best disintegration compared to all other combinations. (Fig. 2). By that it was decided to proceed with a round robin test using the method where the two effects, heated water and stirring, are combined.
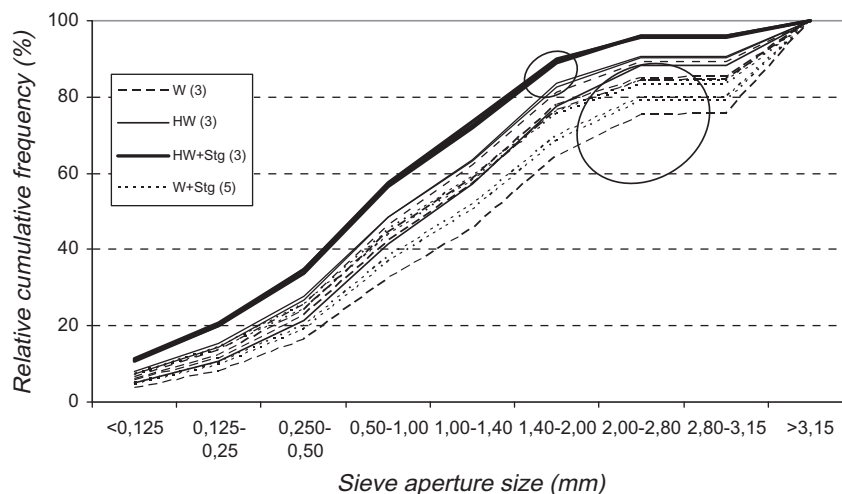

Fig. 1. Particle size distribution of wood pellets disintegrated with water at ambient temperature (W), heated water (HW), heated water combined with stirring (HW + Stg) and water at ambient temperature combined with stirring (W + Stg). Value in brackets in inserts indicate the number of replicate of the specific combination of treatment.

Fig. 2. Particle size distribution of straw pellets disintegrated with water at ambient temperature (W), heated water (HW), heated water combined with stirring (HW + Stg) and water at ambient temperature combined with stirring (W + Stg). Value in brackets in inserts indicate the numer of replicate of the specific combination of treatment.
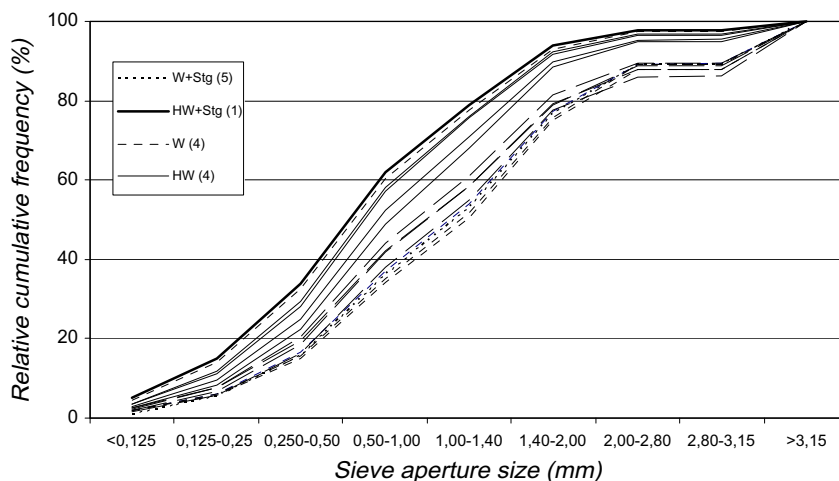
3.2. Dry disintegration study

Solid biofuel pellets from coniferous wood was disintegrated in a hammer mill as described in Section 2.3 at one laboratory. The test was performed on three samples in triplicates and the obtained particle size distributions show smaller particle sizes for the samples disintegrated with a hammer mill compared to the particle size distributions for wet disintegrated pellets (Fig. 3). The difference is a consequence of both the aperture size of the sieve applied to the hammer mill and the hammer mill's further disintegration of the particles into even smaller particles. Applying other aperture sizes would result in particle size distribution that would differ from the present. As a mean for estimating the particle size distribution of the pellet powder after the hammer mill at a specific production facility a method using dry disintegration in laboratory scale hammer mills could be considered. The laboratory hammer mill should then be fitted with sieves having an aperture size corresponding to the full scale hammer mill. However, the obtained results will then only be valid for the specific laboratory and full scale hammer mill and therefore not applicable as a standard test method for inter-laboratory comparisons.

3.3. Wet disintegration round robin test

Solid biofuel pellets produced of coniferous and deciduous wood and comminuted straw were tested in a round robin test at six different laboratories. Except for one laboratory the obtained particle size distributions of the disintegrated deciduous wood pellets showed very little variation (Fig. 4). The reason to the large deviation for the one laboratory is assumed to be use of too small amplitude in the sieving operation compared to prescribed amplitude of 3 mm. The amplitude is identified to have a significant influence on the result of a sieving operation [16] and the deviating result was excluded in the calculation of repeatability and reproducibility. As with the coniferous wood pellets the particle size distribution of the deciduous wood pellets was very similar between five of the six laboratories (Fig. 4). The exception is the one laboratory which also obtained deviating results for the coniferous wood pellets and the deviating result was here also excluded in the calculation of the repeatability and reproducibility (Table 2).

The sieving of the straw pellet material caused problem in three of the laboratories where mass losses of the disintegrated material during the sieving operations caused rejection of the results. However, for the three laboratories where the disintegration of straw
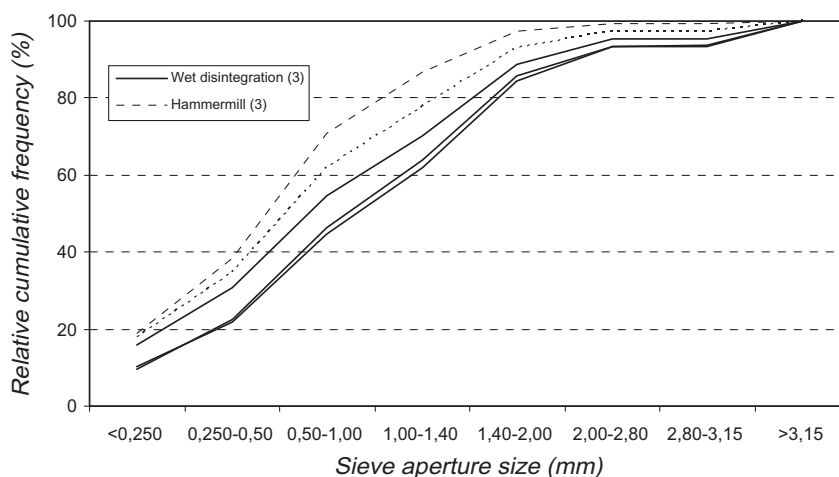

Fig. 3. Particle size distribution of wood pellets disintegrated in heated water combined with stirring and wood pellets disintegrated in a hammermilll. Value in brackets in inserts indicate the number of replicate of the specific combination of treatment.
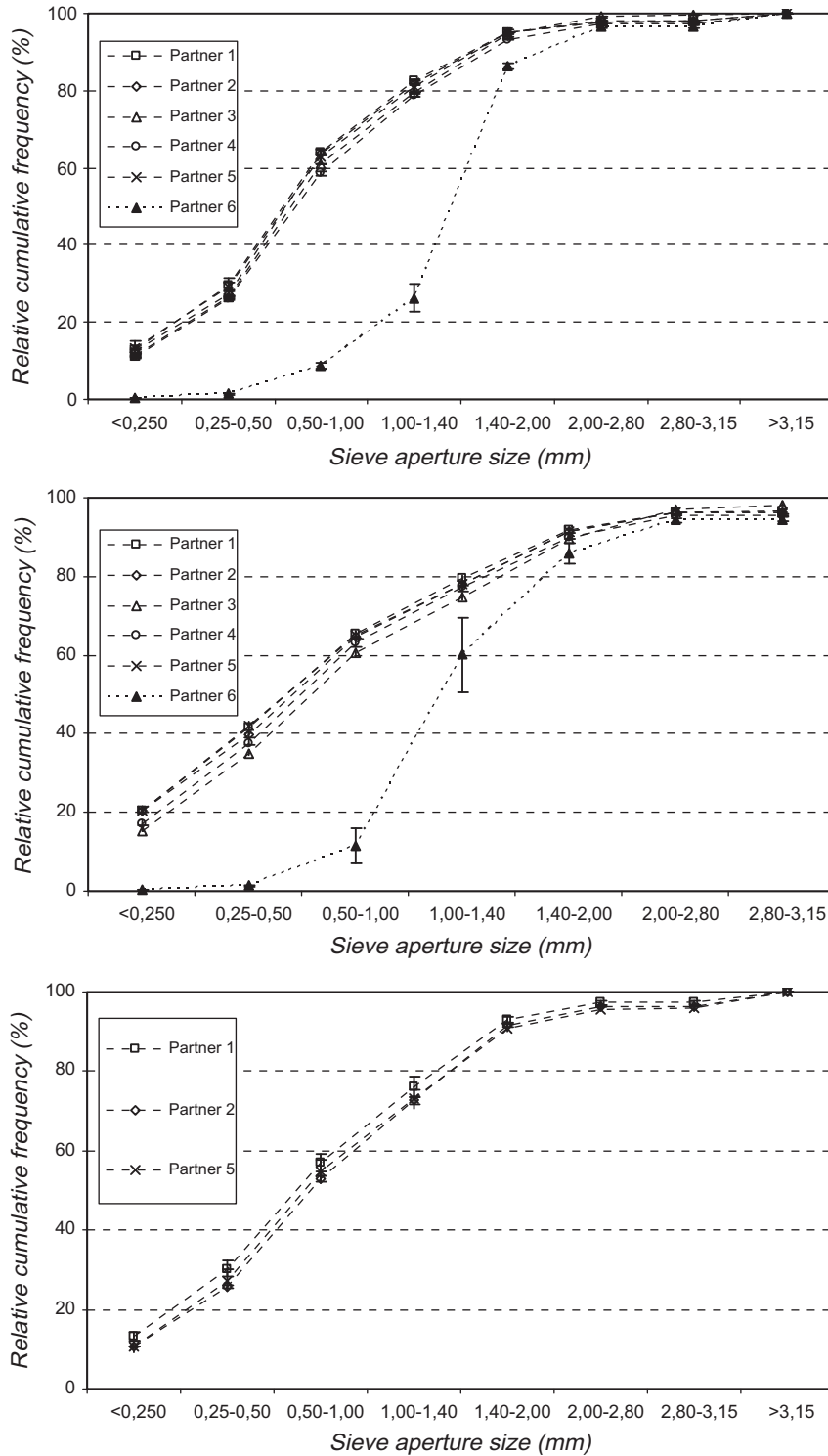
Fig. 4. Particle size distribution tested in a round robin with six laboratories. Coniferous pellets (upper figure), deciduous pellets (middle figure) and straw pellets (lower figure) are disintegrated using heated water combined with stirring of the slurry. The sieving of the straw pellet material caused problem in three of the laboratories, hence only the results from three of the six laboratories are presented.

pellets was completed without problems the resulting particle size distributions was very similar (Fig. 4). In the following evaluation of the method concerning straw pellets only the results from three of the six laboratories are used.

The mean value, the standard deviation (SD) and the coefficient of variance (CV) at the 25%-, 50%- and 75% quantile for the cumulative particle size distributions of straw-, coniferous- and

deciduous pellets is presented in Table 1. The coefficient of variation expresses the standard deviation as a percentage of the sample mean making the size of variation relative of the size of observation. In all but three tests (Lab 2, straw and Labs 3 and 4 deciduous) the CV decrease with increasing quantiles indicating that the relative accuracy of the particle size determinations decline with decreasing particle size. This most likely reflects the

Table 1
Mean value, standard deviation and coefficient of variance for the 25%-, 50%- and 75% quantiles from the particle size distribution of the tested solid biofuel pellets.

Lab	Quantiles	Straw pellets			Deciduous wood pellets			Coniferous wood pellets		
		25%	50%	75%	25%	50%	75%	25%	50%	75%
1	Mean (mm<)	0.42	0.87	1.38	0.30	0.68	1.27	0.43	0.80	1.24
	SD (mm<)	0.025	0.041	0.054	0.011	0.020	0.023	0.011	0.013	0.015
	CV	5.81	4.71	3.91	3.75	2.92	1.81	2.63	1.57	1.25
2	Mean (mm<)	0.49	0.95	1.47	0.31	0.71	1.31	0.46	0.81	1.25
	SD (mm<)	0.006	0.011	0.026	0.007	0.010	0.017	0.014	0.005	0.003
	CV	1.32	1.19	1.75	2.21	1.48	1.30	3.05	0.63	0.23
3	Mean (mm<)	–	–	–	0.37	0.80	1.41	0.48	0.84	1.30
	SD (mm<)	–	–	–	0.002	0.012	0.032	0.005	0.003	0.002
	CV	–	–	–	0.57	1.46	2.30	0.98	0.30	0.13
4	Mean (mm<)	–	–	–	0.34	0.74	1.33	0.48	0.87	1.33
	SD (mm<)	–	–	–	0.006	0.014	0.028	0.006	0.007	0.008
	CV	–	–	–	1.64	1.92	2.11	1.29	0.84	0.60
5	Mean (mm<)	0.47	0.91	1.46	0.30	0.68	1.31	0.43	0.81	1.27
	SD (mm<)	0.036	0.052	0.079	0.011	0.023	0.037	0.035	0.031	0.035
	CV	7.71	5.71	5.38	3.61	3.35	2.83	8.13	3.84	2.78
6	Mean (mm<)	–	–	–	1.11	1.32	1.72	1.38	1.63	1.88
	SD (mm<)	–	–	–	0.045	0.065	0.157	0.075	0.026	0.014
	CV	–	–	–	4.07	4.94	9.12	5.45	1.57	0.74

Table 2
Performance characteristic for the method calculated as repeatability and reproducibility.

Material	Quantiles (%)	N	X mm	s_r (mm)	s_R (mm)	S_r (%)	S_R (%)
Coniferous pellets	25	5	0.46	0.018	0.039	3.94	8.53
	50	5	0.83	0.016	0.049	1.94	5.93
	75	5	1.28	0.018	0.063	1.40	4.92
Deciduous pellets	25	5	0.33	0.0082	0.055	2.51	16.80
	50	5	0.72	0.016	0.089	2.22	12.36
	75	5	1.33	0.028	0.087	2.11	6.54
Comminuted straw	25	3	0.46	0.025	0.056	5.46	12.23
	50	3	0.91	0.039	0.067	4.30	7.37
	75	3	1.44	0.057	0.092	3.96	6.31

N: number of values, X: mean value, s_r: estimate of repeatability, S_r: estimate of relative repeatability, s_R: estimate of reproducibility, S_R: estimate of relative reproducibility.

increasing probability of clogging of the openings in the sieves with small apertures.

The repeatability was found to be smaller than the reproducibility indicating that the disintegration and sieving procedure varies between the laboratories and thereby influence the result of the method.

4. Conclusion

The result of the initial wet disintegration study illustrated that wet disintegration in water at ambient temperature is an unsuitable method for determining the internal particle size distribution of wood- and straw pellets. The method does not adequately separate the particles in the pellets from each other and thereby result in an overestimation of larger particles. When the wet disintegration was performed with water heated to the boiling point and coupled with mechanical disintegration in terms of stirring a more complete disintegration of the pellets was obtained. In the round robin test the method combining the use of heated water and stirring of the slurry was tested on solid biofuel pellets produced of comminuted straw and deciduous- and coniferous wood. With this revised method a satisfactory disintegration was obtained of all three types of pellets.

The dry disintegration of coniferous pellets resulted in smaller particle sizes compared to the wet disintegration using heated water and stirring of the slurry. The particle size distribution of the dry disintegrated pellets to a large extend reflects aperture size of the applied sieves in the hammer mill and the further comminution by the hammer mill of the disintegrated particles.

It is not possible from the present study to identify whether wet or dry disintegration as applied here, describes the resulting particles size distribution of a large scale hammer milling operation in the best manner. However, as shown in the round robin test the revised wet disintegration method gives highly repeatable and reproducible results and the method may provide both a relative measure of the particle size distribution of the produced fuel powder in a large scale milling operation and the particle size distribution of the raw material used for the pellet production respectively. Further, the wet disintegration method can be conducted by any laboratory having access to sieving equipment, whereas the dry disintegration demands a hammer mill and a procedure which secure that the dry disintegration results in comparable results between laboratories. By comparing results obtained with the wet disintegration method to the particle size distribution for the actual produced fuel powder in large scale hammer mills a functional relationship might be constructed. The relationship would be explicit for the specific hammer mill and the operation hereof.

Acknowledgements

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