

VALUE OF COMBUSTION HEAT OF BIOMASS MATERIAL DEPENDING ON SIZE OF BIOMASS PARTICLES AND PERIOD OF MEASURING IN CALORIMETER

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Abstract. The paper compares the measured values of combustion heat for partially dehydrated digestate from an agricultural BGP (biogas plant) and for *Miscantus x Giganteus* (for the same dampness of samples). We ascertained that the measured values of combustion heat of the stated biomass materials depend on the size of the biomass particles (level of grinding). The differences between the values of combustion heat for the smallest biomass fraction of 0.1 mm and the biggest of 2.5 mm are approximately 8%. From the achieved results, we experimentally derived the maximum size of the biomass particles for measuring in the calorimeter. Further differences in the measured values of combustion heat are caused by the selected different durations of the whole measuring cycle in the calorimeter (we used adiabatic calorimeter MS-10 with measuring cycles of 5, 8 and 16 minutes) and the differences oscillated within the interval up to 4%. With a longer measuring cycle, we achieved smaller oscillation of the measured values for all biomass materials. All measuring of the combustion heat was statistically processed by a number of methods with small difference in the resulting values.

Keywords: dehydrated digestate, *Miscantus x Giganteus*, particles, nonparametric statistical methods, period of measurement, calorimeter

Introduction

The paper was created in response to a frequent situation during measuring of combustion heat of samples of biomass materials using the AS 10 Calorimeter. With repeated measuring of the same material, various dispersions of the set of the measured values were irregularly achieved, and usually one or two measured values were beyond the pale of the set of the measured data. Naturally, we sought the causes of such state, but even after the executed checks and changes in the measuring procedure and sample preparation, the situation did not change much. After excluding some possible causes of this state, we focused on three further stated causes of bigger dispersion of the measured values of combustion heat and of the values beyond the pale of the data set. They were the following:

- period of measuring the sample using the calorimeter,
- influence of the size of the particles on the value of the combustion heat,
- impact of various ways of calculating combustion heat median and its reliability interval always from five measured values, i.e. the impact of the way of statistical processing of the measured data set.

Two materials with comparable dimensions of particles were selected for the comparative measuring. The first material was partially dehydrated digestate (DG), which has smaller contents of organic matter compared to the other tested material due to the process of digestion (fermentation). The second material was crushed *Miscantus x Giganteus* (MG). In the resulting mixture, MG after crushing included particles of similar size as the particles in DG, the texture of the crushed mixture was similar to the DG mixture, however, MG did not undergo the process in BGP, and therefore its particles included more organic matter than DG [1]. The accuracy of the calorimeter was the same during measuring of samples of both materials, which was checked by combusting benzoic acid. In order to exclude the influence of humidity of the material of the measured samples on the resulting values, always a completely dried-out sample was measured. The producer states the relative error of the machine to be less than one per cent, but the error is practically higher and depends also on the experience of the operator of the machine with preparation and measuring of samples.

Shorter measuring time also brings some inaccuracy, but the producer does not quantify it. With regard to small sets of measured values (always only five values), the selection of a convenient statistical method for their processing is also very important in order for the median and reliability interval to represent the measured set of values of combustion heat [2, 3]. Concurrent effect of all or only some of these influences is most likely the most frequent cause of the deviation in the measured value of combustion heat of a few per cent. The quantification of these influences on the measured value of combustion heat must be sought in Table 1 and Table 2. It must be also noted that the

influence of single change of conditions and environment of measuring that would cause a deviating measured value was not taken into account, because measuring using the calorimeter was conducted under the same conditions. Therefore, we focused on the system errors of measuring (if they occur during measuring) and on quantification of the above-stated three possible causes of deviations of the measured values of combustion heat for both materials.

Materials and methods

The stated comparison measurements using the calorimeter were conducted with partially dehydrated and subsequently completely dehydrated digestate (DG) coming from agricultural biogas plants (BGP) and with crushed and dried-out plant *Miscanthus x Giganteus* (MG). DG was not further mechanically treated, it was only divided in individual size fractions after drying out using the AS 200 sifting machine with screens of mesh size 0.1, 0.25, 0.5, 1.0, and 2.5 mm. During sifting particles smaller than 0.1 mm were caught in a dish with hard bottom. Thus, we got the total of six size fractions of DG particles for which combustion heat was measured on the calorimeter (always for five repetitions) with three different times (5, 8 and 16 minutes). The results of the repeated five measurements were processed using various statistical methods [4]. Everything is clearly stated in Table 1.

The material of all size fractions of DG was, prior to removal of samples for calorimeter measuring, homogenized on a rotary crusher so the particles were all of the same size. The same procedure was used for the other measured material (MG). It was crushed on a hammer crusher of FQ type with screens of mesh size of 8 mm for mixture of particles of various sizes. This mixture was then divided in individual size fractions using the AS 200 sifting machine. Measuring of MG samples on the calorimeter was conducted in the same way as with DG. The results for MG are clearly stated in Table 2.

The weight differences of individual size fractions of both materials achieved using the AS 200 sifting machine are stated in Table 3. Further text includes formulas for calculation of the median of the set and the reliability interval for the median, the Horn's method and Lambda transformation, since these statistical methods are less frequent. The last two stated methods belong to the group of statistical non-parametric methods. We chose for tests phytase feed enzyme specially developed to increase the digestibility of phytin-bound phosphorus, calcium energy and amino acids in poultry and pig diets. It is supplied as off-white to light tan fine granular product. The agent is produced in accordance with the FAO/WHO JECFA and FCC recommendations. It has to be used at a rate of 0,025 – 0,1 kg/tonne (0,0025-0,01 %) of finished feed, included direct or via a premix.

The used calorimeter is a device with an isothermal coat and fully automated process of measuring including the calculation correction of heat exchange. The calorimeter water container is enclosed by a double coaxial coat. The temperature is measured inside the calorimeter container at two places using a thermistor with the frequency of 0.5 s with 0.00025 K resolution within 293 to 303 K temperature scale. From the measured data that are stored in the memory, the calorimetric heat jump and correction for the exchange of heat between the calorimeter and its coat (surroundings) are calculated at the end of measuring. The resulting value will be displayed on the display as a corrected heat jump – dTk. Correction to the heat exchange is conducted according to the Newton law [5]. Apart the Newton constant of heat conduction, the calculation also includes another constant correcting the production of heat created in the calorimeter by mixing. Both constants are calculated and automatically saved in the memory in the work mode CALIBRATION which takes 32 minutes. This work mode is used only rarely (to stipulate the heat capacity of the calorimeter). Standard time of sample measuring in calorimeter is 16 minutes, and the temperatures are therefore deducted in the stable thermal state of the calorimeter.

However, this period of measuring can be shortened to 5 or 8 minutes while keeping good accuracy of the achieved results. With these shortened periods of measuring, balanced temperatures are calculated using mathematical extrapolation from the time progress of the heat curve. The computer gathers the empirical factors necessary for the extrapolation from the preceding standard, 16-minute measuring and stores them in its memory. Therefore, before using a shortened period of combustion heat measuring (for 5 and 8 minutes), it is necessary to conduct at least ten standard measurements (of 16 minutes). These values are then used for the extrapolation of the temperature values

during shorter measuring periods. Combustion heat of the sample inserted in the calorimeter is calculated from the following relation (1):

$$Q_s = \frac{dT_k \cdot T_k - Q_n}{M} \quad (1)$$

where Q_s – combustion heat of the completely dried-out sample (the sample has 0 % humidity), $J \cdot g^{-1}$;
 dTk – corrected heat jump (information displayed on the calorimeter display), K;
 Tk – heat capacity of the calorimeter (calorimeter constant), $J \cdot K^{-1}$;
 Q_n – correction to the created nitric acid and heat created by burning of paper in which the sample is usually wrapped (this value can be also stated as a constant), J;
 M – weight of the completely dried out sample, g.

Results and discussion

The measured values of combustion heat for both materials (DG and MG), three times of measuring using the calorimeter (5, 8 and 16 minutes) and size fraction of particles of both materials from 0.0 to 2.5 mm are stated in Table 1 and Table 2, which clearly show small difference in the measured average values of combustion heat for both materials (lines designated in both tables as $\Sigma 5$, $\Sigma 8$ and $\Sigma 16$). One of the causes of this state can also be a very similar texture of both materials including similar division of the sample in individual size fractions, as stated in Table 3. For both materials, length of particles is the dominant dimension. DG particles are not so straight, they are more tangled together (creating a network) and when they are dry, they are harder to move individually. On the contrary, the MG particles are straight and they move easier in the sample. Individual size fraction of both materials does not have to have always the same substance compositions, as stated by [6]. The digestate particles are, after the process in BGP, more mineralized (their organic matter is “consumed” during the BGP process compared to MG particles that did not undergo this process). That is why dry DG is also used as a “light” mineral fertilizer. For the purpose of comparing the average median values of the combustion heat, we compared the lines in both charts designated $\Sigma 5$, $\Sigma 8$ and $\Sigma 16$). Also, in both charts, medians of combustion heat of both materials calculated by a non-parametric statistical method called “lambda transformation” (LT) for standard period of calorimeter measuring of 16 minutes were taken as basis for further comparison. Combustion heat shows difference in the measured values depending on the size of the particles. The progress of this difference is similar for both DG and MG. The smallest size fraction of particles (0.0 to 0.1 mm) also includes larger amount of dust and small mineral particles than the other size fractions. Visually, this can be confirmed under a microscope or electronic magnifying glass. This can be the reason of the smaller value of combustion heat for both materials with this particle size. Both charts also show that the maximum value of the measured combustion heat corresponds to the size of the particles between 0.1 and 0.5 mm. For unequivocal confirmation of this phenomenon, a higher number of measurements would have to be conducted. A partial reason can also be the fact that the crushed MG stem does not have the same composition along its whole length. This reason is partly eliminated in the case of DG. The size of the sample combusted in the calorimeter was always the same, from 0.5 to 0.55 g. The used condenser paper in which the samples were wrapped was of the same size and kind, as well as the used ignition wire. The difference in the combustion heat value (with regard to the value calculated according to LT), amounts, depending on the size of particles of both materials, to the maximum of a few per cent.

The way of processing the set of the measured values has greater influence on the median of the combustion heat (regardless of the size of the particles of both materials) [3; 4]. With regard to the small size of the measured data sets, also two non-parametric methods (HM and LT) were used. In particular, the latter method is much more demanding for the calculation of a median, but its use provides more comparable and homogenous values of combustion heat including the small width of the reliability interval. Therefore, the median values of combustion heat calculated using this method for standard time of 16 minutes of measuring in the calorimeter were taken as a basis for calculation of the differences in the combustion heat values depending on the re-measuring and the particle size. The dispersion of the measured values matters a lot.

Table 1

Mean value of combustion heat of dehydrated digestate ($\text{kJ}\cdot\text{g}^{-1}$) and its confidence interval for the processing data by various statistical methods

File Characteristics		Arithmetic Mean				Geometric Mean				Harmonic Mean			
Time measur., min	Mesh Size, mm	Mean	Confidence Interval			Mean	Confidence Interval			Mean	Confidence Interval		
			Min	Max	Width Interval		Min	Max	Width Interval		Min	Max	Width Interval
5	0.00	17.63	17.49	17.23	0.8	17.48	16.99	17.97	1	17.84	17.21	18.40	0.8
5	0.10	19.32	17.56	19.22	1.8	19.31	18.50	20.16	1.7	19.29	18.78	19.83	1.1
5	0.25	19.27	18.96	19.59	0.6	19.28	18.95	19.58	0.6	19.27	18.11	19.62	1.5
5	0.50	18.65	18.30	19.00	0.7	18.65	18.30	19.00	0.7	18.76	18.23	19.70	0.8
5	1.00	18.37	17.90	18.64	0.6	18.37	18.90	18.65	0.6	18.45	18.15	18.59	0.4
5	2.50	18.39	17.56	19.22	1.7	18.38	17.58	19.21	1.6	18.15	17.78	18.97	1.2
Σ5		18.60	17.94	19.26	1.7	18.59	17.93	19.27	1.3	18.61	17.83	19.38	1.6
8	0.00	17.52	17.17	17.85	0.7	17.51	17.18	17.85	0.7	17.51	17.14	17.88	0.7
8	0.10	19.44	19.12	19.75	0.6	19.35	19.70	19.64	0.6	19.48	19.23	19.73	0.5
8	0.25	19.41	19.80	19.74	0.7	19.41	19.80	19.74	0.7	19.41	19.13	19.69	0.6
8	0.50	19.73	19.42	20.30	0.6	19.73	19.42	20.40	0.6	19.73	19.43	20.30	0.6
8	1.00	19.23	18.50	19.89	1.3	19.22	18.57	19.90	1.3	19.22	18.51	19.93	1.4
8	2.50	18.86	18.53	19.20	0.7	18.86	18.53	19.19	0.7	18.86	18.55	19.17	0.6
Σ8		19.30	18.20	19.83	1.6	18.96	18.12	19.84	1.7	19.00	18.40	19.60	1.2
16	0.00	17.54	16.86	18.21	1.7	17.53	16.87	18.22	1.4	17.52	16.85	18.19	1.3
16	0.10	19.86	19.27	20.46	1.2	19.86	19.26	20.47	1.2	19.85	19.36	20.35	1
16	0.25	19.83	19.58	20.90	0.5	19.83	19.58	20.90	0.5	19.83	19.29	20.37	1.1
16	0.50	19.10	18.74	19.47	0.7	19.10	18.74	19.47	0.7	19.10	18.73	19.47	0.7
16	1.00	18.64	17.99	19.29	1.3	18.64	17.99	19.30	1.3	18.63	17.96	19.30	1.3
16	2.50	18.29	17.75	18.83	1.1	18.29	17.76	18.83	1.1	18.28	17.76	18.80	1
Σ16		18.88	17.93	19.83	1.9	18.82	17.93	19.77	1.8	18.83	18.50	19.61	1.6
File Characteristics		Median				Horn's Method				Lambda Transformation			
Time measur., min	Mesh Size, mm	Mean	Confidence Interval			Mean	Confidence Interval			Mean	Confidence Interval		
			Min	Max	Width Interval		Min	Max	Width Interval		Min	Max	Width Interval
5	0.00	17.84	17.43	18.26	0.8	17.55	16.26	18.83	2.6	17.63	17.24	18.20	0.8
5	0.10	19.30	18.78	19.83	1.1	19.46	17.72	21.20	3.5	19.31	18.50	20.16	1.7
5	0.25	19.27	18.91	19.62	0.7	19.27	18.34	20.20	1.9	19.27	18.95	19.60	0.7
5	0.50	18.76	18.35	19.18	0.8	18.65	17.71	19.59	1.9	18.65	18.30	19.00	0.7
5	1.00	18.44	18.15	18.78	0.6	18.45	18.26	18.63	0.4	18.37	18.80	18.65	0.6
5	2.50	18.15	17.56	18.74	1.2	18.13	17.99	18.27	0.3	18.38	17.59	19.21	1.6
Σ5		18.60	17.73	19.46	1.7	18.70	17.52	19.88	2.3	18.59	17.94	19.27	1.3
8	0.00	17.57	17.20	17.94	0.7	17.48	16.58	18.38	1.8	17.51	17.18	17.85	0.7
8	0.10	19.59	19.35	19.84	0.5	19.53	19.11	19.58	0.5	19.48	19.22	19.75	0.5
8	0.25	19.45	19.17	19.73	0.6	19.46	19.34	19.58	0.2	19.41	19.70	19.74	0.7
8	0.50	19.99	19.69	20.29	0.6	19.82	19.32	20.32	1	19.73	19.42	20.40	0.6
8	1.00	18.99	18.28	19.70	1.4	19.37	20.92	21.12	0.2	19.22	18.57	19.89	1.3
8	2.50	18.86	18.54	19.16	0.6	18.93	18.48	19.37	0.9	18.86	18.53	19.19	0.7
Σ8		19.22	18.63	19.80	1.2	19.23	18.61	19.85	1.2	19.20	18.18	19.90	1.7
16	0.00	17.51	16.84	18.18	1.3	17.56	17.56	19.72	2.2	17.53	16.87	18.22	1.4
16	0.10	19.98	19.49	20.48	1	19.98	19.67	20.29	0.6	19.86	19.26	20.46	1.2
16	0.25	19.84	19.30	20.37	1.1	19.85	19.60	20.80	0.5	19.83	19.58	20.90	0.5
16	0.50	19.30	18.66	19.40	0.7	19.19	18.34	19.72	1.4	19.10	18.74	19.47	0.7
16	1.00	18.89	18.22	19.56	1.3	18.62	17.32	19.92	2.6	18.64	18.00	19.30	1.3
16	2.50	18.25	17.73	18.78	1.1	18.18	17.43	18.93	1.5	18.29	17.76	18.83	1.1
Σ16		18.96	17.81	20.10	2.3	19.10	17.28	20.73	3.4	18.86	17.91	19.84	1.9

Table 2

Mean value of combustion heat of *Miscantus x Giganteus* (kJ·g⁻¹) and its confidence interval for the processing data by various statistical methods

File Characteristics		Arithmetic Mean				Geometric Mean				Harmonic Mean			
Time measur., min	Mesh Size, mm	Mean	Confidence Interval			Mean	Confidence Interval			Mean	Confidence Interval		
			Min	Max	Width Interval		Min	Max	Width Interval		Min	Max	Width Interval
5	0.00	17.77	17.51	18.30	0.52	17.77	17.51	18.03	0.52	17.77	17.61	17.93	0.32
5	0.10	18.42	18.12	18.72	0.59	18.42	18.08	18.72	0.64	18.42	18.04	18.80	0.76
5	0.25	18.29	18.04	18.55	0.51	18.29	18.06	18.57	0.52	18.31	17.73	18.90	1.17
5	0.50	18.20	17.88	18.62	0.83	18.20	17.71	18.61	0.90	18.19	17.54	18.85	1.30
5	1.00	18.55	18.00	19.90	1.09	18.55	18.02	19.09	0.07	18.54	17.92	19.17	1.24
5	2.50	15.57	14.43	16.71	2.28	15.55	14.46	16.73	2.28	15.53	14.88	16.18	1.30
Σ5		17.80	16.61	18.98	2.37	17.76	16.56	19.05	2.49	17.66	17.72	18.14	0.36
8	0.00	18.03	17.91	18.15	0.40	18.03	17.91	18.15	0.24	18.02	17.92	18.12	0.20
8	0.10	18.40	18.24	18.55	0.31	18.40	18.24	18.56	0.31	18.40	18.24	18.56	0.32
8	0.25	18.33	18.16	18.51	0.34	18.33	18.16	18.50	0.34	18.34	18.27	18.39	0.11
8	0.50	18.31	18.29	18.34	0.04	18.31	18.29	18.34	0.05	18.31	18.29	18.33	0.40
8	1.00	18.39	18.15	18.64	0.48	18.30	18.26	18.36	0.10	18.30	18.20	18.59	0.39
8	2.50	15.79	15.43	16.16	0.73	15.79	15.43	16.17	0.74	15.79	15.48	16.09	0.60
Σ8		17.87	16.79	18.95	2.16	18.36	18.09	18.51	0.42	17.81	17.58	18.03	0.45
16	0.00	17.88	17.72	18.05	0.33	17.87	17.39	18.37	0.98	17.88	17.69	18.01	0.32
16	0.10	19.08	18.91	19.26	0.34	19.36	18.91	18.26	0.34	19.08	18.87	19.29	0.41
16	0.25	19.14	18.67	19.61	0.94	19.14	18.68	19.61	0.93	19.14	18.64	19.63	0.99
16	0.50	18.73	18.29	19.17	0.88	18.73	18.30	19.17	0.87	18.73	18.02	19.43	1.40
16	1.00	19.33	18.95	19.81	0.96	19.12	18.93	19.33	0.40	19.13	18.88	19.37	0.49
16	2.50	17.38	16.88	17.89	1.01	17.38	16.88	17.90	0.02	19.13	18.88	19.37	0.49
Σ16		18.59	18.59	19.41	1.64	18.65	17.76	19.57	1.81	18.37	17.96	18.84	0.88
File Characteristics		Median				Horn's Method				Lambda Transformation			
Time measur., min	Mesh Size, mm	Mean	Confidence Interval			Mean	Confidence Interval			Mean	Confidence Interval		
			Min	Max	Width Interval		Min	Max	Width Interval		Min	Max	Width Interval
5	0.00	17.72	17.57	17.88	0.31	17.71	17.68	17.75	0.06	17.76	17.51	18.04	0.53
5	0.10	18.47	18.04	18.86	0.76	18.49	18.42	18.55	0.13	18.42	17.75	19.10	1.34
5	0.25	18.35	17.90	18.79	0.88	18.35	17.82	18.89	1.06	18.31	18.05	18.57	0.51
5	0.50	18.13	17.98	18.23	0.21	18.37	18.07	18.66	0.59	18.19	17.79	18.61	0.82
5	1.00	18.38	16.99	19.77	2.78	18.36	18.15	18.58	0.43	18.54	18.01	19.09	1.07
5	2.50	15.37	14.71	16.01	1.30	15.66	13.87	17.46	3.59	15.55	14.45	16.72	2.27
Σ5		18.25	17.77	18.72	0.95	18.36	18.28	18.40	0.12	17.77	16.62	18.98	2.36
8	0.00	18.04	17.94	18.15	0.21	18.05	17.86	18.23	0.37	18.14	17.69	18.62	0.93
8	0.10	18.33	18.23	18.43	0.20	18.42	17.95	18.71	0.24	18.40	18.25	18.56	0.31
8	0.25	18.28	18.25	18.32	0.07	18.31	17.54	18.07	0.53	18.38	18.22	18.54	0.32
8	0.50	18.31	18.30	18.33	0.03	18.30	18.28	18.36	0.08	18.31	18.29	18.33	0.04
8	1.00	18.33	18.20	18.45	0.25	18.33	18.20	18.45	0.25	18.39	17.86	18.64	0.78
8	2.50	15.88	15.69	16.07	0.38	15.89	15.64	16.15	0.51	15.70	15.43	16.16	0.73
Σ8		18.30	18.07	18.52	0.45	18.29	18.26	18.32	0.06	17.86	16.77	19.01	2.24
16	0.00	17.94	17.75	18.13	0.38	17.92	17.75	18.09	0.35	17.88	17.72	18.05	0.33
16	0.10	19.10	18.89	19.31	0.41	19.04	18.75	19.33	0.58	19.08	18.92	19.25	0.34
16	0.25	19.14	18.65	19.64	0.99	19.01	18.02	19.62	1.60	19.14	18.59	19.59	1.01
16	0.50	18.66	17.95	19.36	1.40	18.73	18.17	19.30	1.13	18.73	18.30	19.15	0.86
16	1.00	19.20	18.76	19.65	0.89	19.10	18.65	19.54	0.89	19.13	19.10	19.16	0.06
16	2.50	17.42	16.72	18.13	0.49	17.33	15.92	18.74	2.82	17.38	16.88	17.89	1.52
Σ16		18.88	18.01	18.87	0.88	19.12	42204.	19.16	0.09	18.55	17.79	19.31	2.08

Table 3

Weight proportion of particles in the sample from MG and DG depending on their size

Mesh Size, mm	0.00	0.10	0.25	0.50	1.00	2.50
Miscantaeus x Giganteus (MG), %	0.42	2.61	6.05	12.27	18.64	60.01
Dehydrated Digestate (DG), %	0.58	1.81	3.12	10.37	16.64	67.66

With small dispersion, comparable results are achieved with most of the used methods of calculation, as shown in both charts. The error of the calorimeter is declared to be the maximum of two per cent. It depends not only on careful weighing of the sample to at least three decimal positions and measuring of differences in temperature to thousands of Kelvin, but also on the laboratory routine of the calorimeter operator.

Conclusions

Based on the executed laboratory measuring, the following conclusions can be drawn:

1. Median value of combustion heat depends on the period of measuring of the sample in the calorimeter only slightly. With error of up to 2 per cent, the shortened period of measuring of 8 minutes can be well used with the stated calorimeter type. Using the 5-minute period may as much as double the error of the result.
2. The size of the particles influences the resulting value of the combustion heat less than the measuring period. The measured sample is a mixture of particles and thus the distinctive difference in the combustion heat value for the smallest (biggest) fraction is automatically corrected (because the weight share of the smallest and biggest size fraction is small).
3. Correct selection of the method of processing of the measured data in the set with regard to their dispersion and size of the set has much more importance and influence on determination of the combustion heat median.

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