

## **THERMAL PROPERTIES OF LIGNOCELLULOSE PELLETS**

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### **Abstract**

*This article deals with the characterization of biomass pellets using Differential Scanning Calorimetry. We used three types of industrially produced and commercially available pellets as samples: wood pellets containing grass, wood pellet containing bark and wood pellets without bark. Each of the samples were examined using the DSC method. Based on the measurements in atmosphere of air and nitrogen temperature, the changes caused by thermal degradation of various kinds of test fuels were observed. Subsequently, limits of exothermic processes, reaction enthalpy changes and the temperature at which exothermic reactions reached peaks were determined.*

### **Key words**

*lignocellulose pellets, wood, Differential Scanning Calorimetry (DSC), biomass, thermal decomposition, pyrolysis*

### **INTRODUCTION**

One of the solid fuels are biomass-based pellets. This form of biomass fuel allows combustion boilers to operate partially or fully automatically (1). Lignocellulose pellets are granules of circular cross-section with a diameter of about 6 – 8 mm and a length of 10 – 30 mm with a relatively low humidity (below 12%) (2, 3). Pellets are made exclusively out of a waste material such as sawdust and shavings without any chemical additives. Pellets are made by a relatively simple and cheap process (e.g. compacting). High pressure is applied in order to achieve a high density of fuel (4).

In comparison to raw biomass, biomass pellets have obviously improved fuel quality. However, biomass pellets may still not be the ideal fuel because of the high moisture affinity and the relatively weak durability, especially for the pellets made from agricultural residues (5, 6, 7).

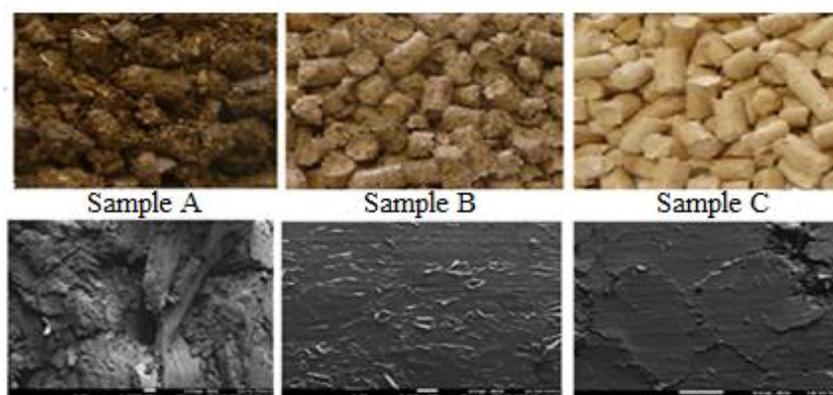
Most studies on the heat of biomass pyrolysis have been carried out using differential scanning calorimetry (DSC) or simultaneous thermogravimetry (TG) and DSC (8, 9, 10, 11).

DSC method provides data about endothermic and exothermic effects about various materials under different conditions during thermal degradation.

Owing to the large variety of lignocellulose material used for the pellets production it is appropriate to know about the thermal phenomena accompanying combustion. The DSC method may be a suitable alternative to studying the heat generation process in an oxygen and nitrogen atmosphere.

## MATERIALS AND METHODOLOGY OF EXPERIMENT

Three samples of industrially produced lignocellulose pellets which properties are shown in Table 1 were used. The determination of carbon and sulfur content was performed by the combustion of the samples in a stream of pure oxygen. Measurements of the samples were carried out using a LECO SC-144DR device. The sulfur and carbon content was calculated from the SO<sub>2</sub> or CO<sub>2</sub> content in the combustion products of the sample, expressed as a mass fraction in percentage or ppm (12). The experiment results are shown in Table 1.



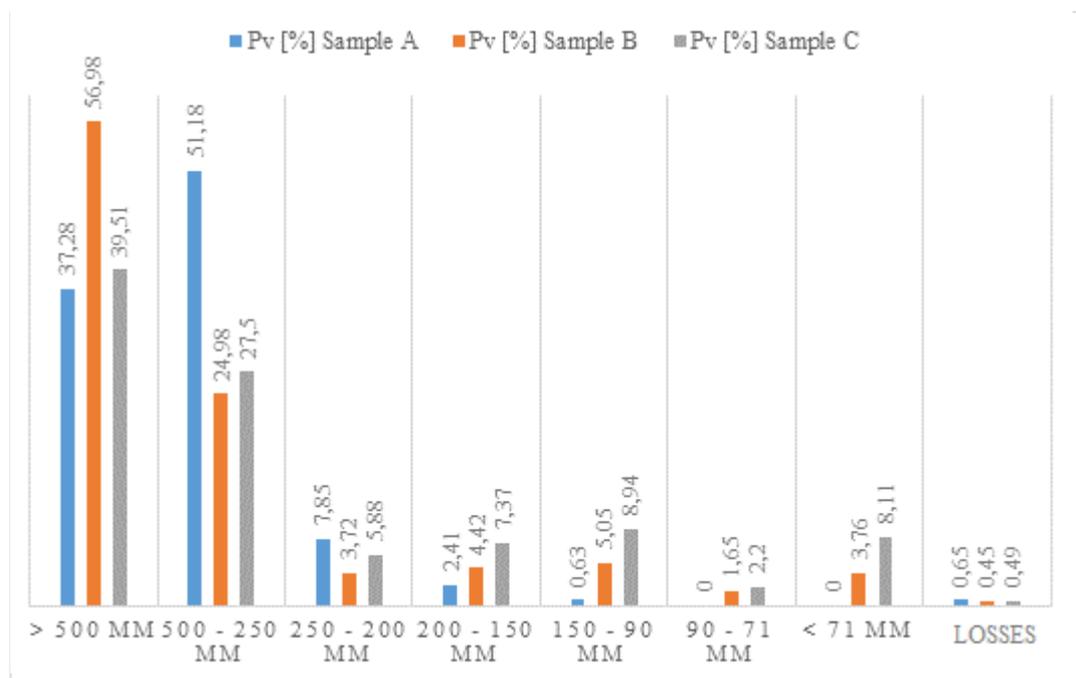
**Fig. 1** Selected lignocellulose pellet samples and a view of their microstructure using SEM (13)

**Table 1** Parameters of lignocellulose pellets containing grass (A), lignocellulose pellets containing bark (B) and lignocellulose pellets without bark (C)

<i>Lignocellulose Pellets</i>	<i>Unit</i>	<i>A</i>	<i>B</i>	<i>C</i>
Diameter	[mm]	-	6.00	6.00
Length	[mm]	-	10 – 40	10 – 40
Humidity	[%]	6.65	7.70	7.09
Mechanical resistance	[%]	-	99.40	99.50
Bulk density	[kg.m <sup>-3</sup> ]	-	650.00	650
Fine proportion of < 3 mm	[%]	-	0.20	0.20
Calorific value	[MJ.kg <sup>-1</sup> ]	18.80	18.70	18.70
Ash	[%]	0.25	0.51	0.170
S	[%]	0.23	0.03	0.03
C	[%]	46.10	47.82	47.98

To determine the dispersion properties of the dust, a sieve analysis was conducted. The analysis was conducted by mechanical vibrations of using a sieving machine Retch AS 200. The

samples were stabilized before the analysis in a desiccator for 24 hours at 24 °C and at a humidity of 33%. Figure 2 shows the results of the sieving analysis.



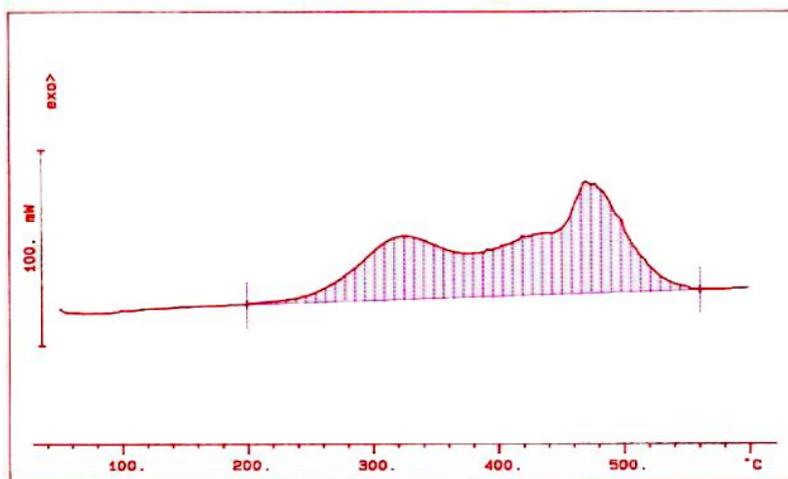
**Fig. 2** Percentage of the dust fractions obtained by sieving analysis

### *Differential scanning calorimetry*

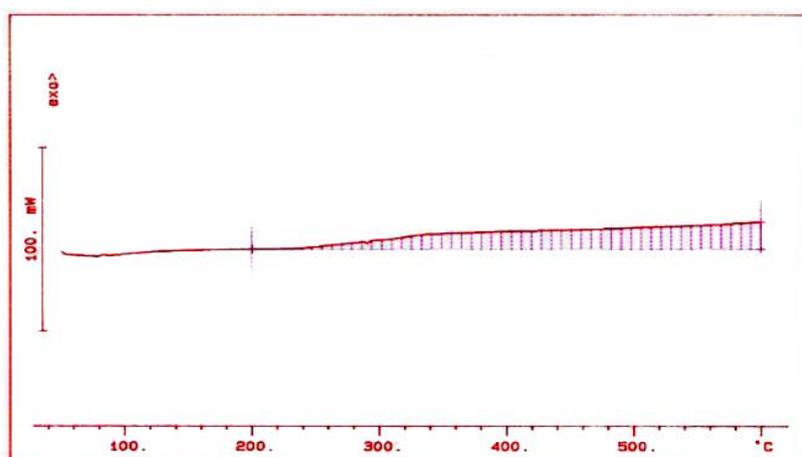
Standard ASTM E 537: 2002 - Standard method for enthalpy measurement validation of differential scanning calorimeters specifies a procedure for conducting the test. Samples testing was performed using a Mettler Thermal Analyzer 3000 TA with a processor TC 10A and a DSC 20 measuring cells in a dynamic atmosphere of air and nitrogen with a flow rate of 50 ml.min<sup>-1</sup> at a heat rate of 10 °C min<sup>-1</sup> to a maximum temperature of 600 °C.

## **RESULTS AND DISCUSSION**

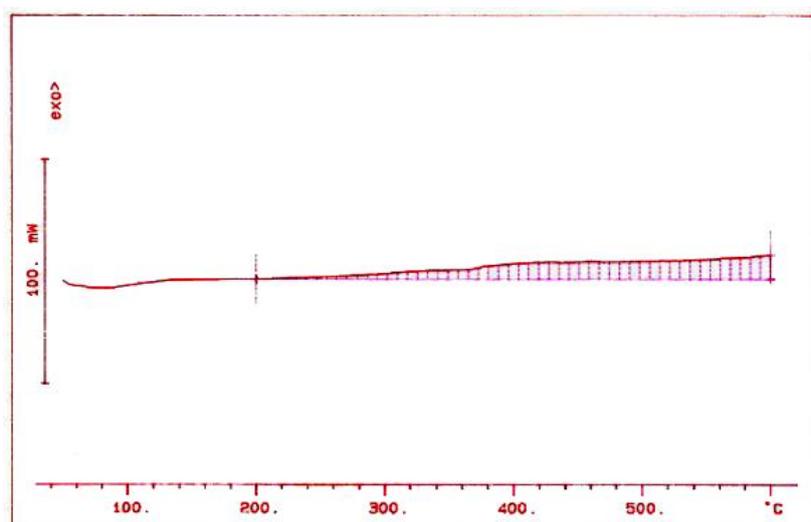
Figures 3 – 9 and Table 2 shows an increase in the reaction enthalpy of the samples investigated by DSC analysis. The temperature at the maximum peak and the enthalpy reaction change were recorded for each sample in the respective temperature range.



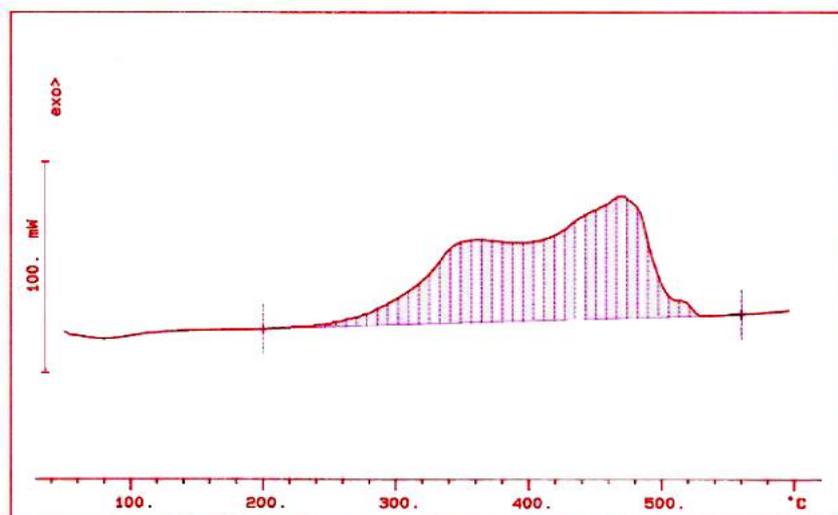
*Fig. 3 DSC curve of thermal decomposition in air – sample A*



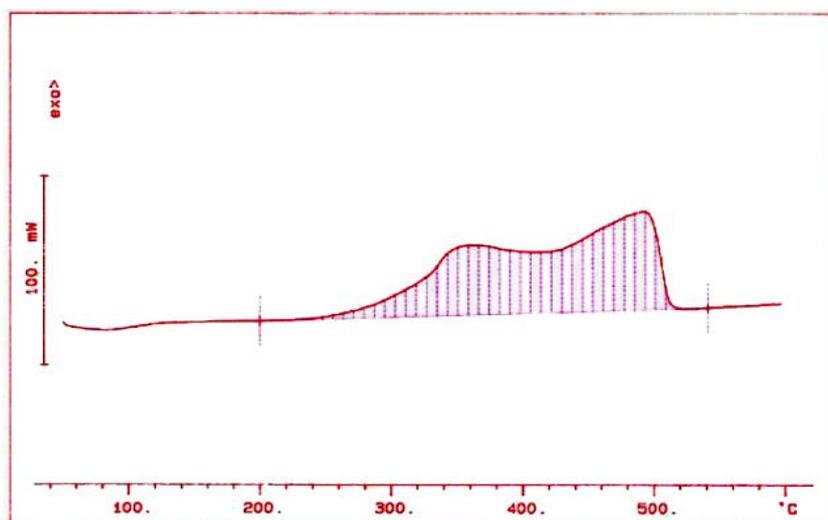
*Fig. 4 DSC curve of thermal decomposition in nitrogen – sample A*



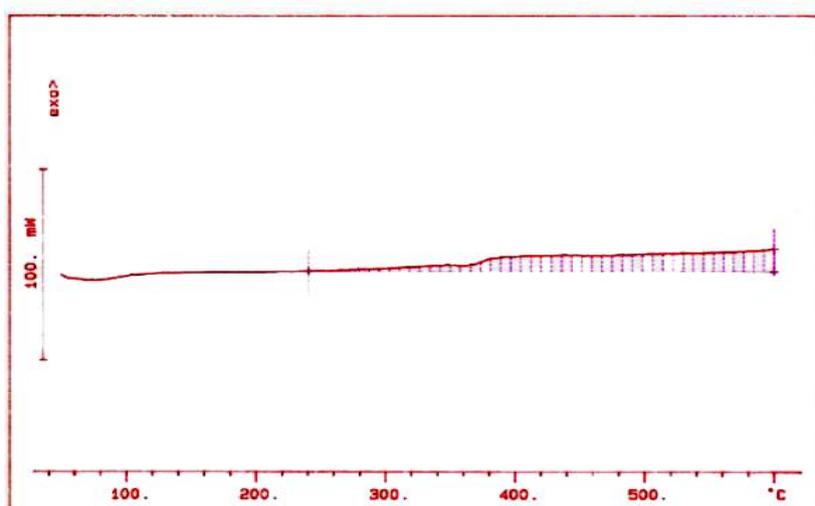
*Fig. 5 DSC curve of thermal decomposition in air – sample B*



*Fig. 6 DSC curve of thermal decomposition in nitrogen – sample B*



*Fig. 7 DSC curve of thermal decomposition in air – sample C*



*Fig. 8 DSC curve of thermal decomposition in nitrogen – sample C*

**Table 2** Characteristics of temperature changes depending on the reaction enthalpies changes by DSC analysis in the atmosphere of air and nitrogen

<i>Atmosphere</i>	<i>Sample</i>	<i>Temperature interval [°C]</i>	<i>Reaction enthalpy change [J.g<sup>-1</sup>]</i>	<i>Peak temperature [°C]</i>
Air 10 [°C.min <sup>-1</sup> ]	A	200 - 560	8760.50	327.00
				475.30
	B	200 - 560	9184.90	364.00
				427.90
	C	200 - 540	8340.90	364.00
				498.10
Nitrogen 10 [°C.min <sup>-1</sup> ]	A	54 - 143	3805.30	597.20
	B	200 - 600	2416.80	599.20
	C	240 - 600	2655.20	599.40

The decomposition of the sample A was observed in air at the temperature range of 50 °C to 600 °C. The results of DSC are shown in Figure 3. The exothermic decomposition of the sample A at heat rate of 10 °C min<sup>-1</sup> occurred from 200 °C and continued up to 560 °C. The temperature at which occurred the maximum heat release rate of sample A in air was 328 °C. The second peak occurred at 476.3 °C. The change in reaction enthalpy of the sample A was 8760.5 J.g<sup>-1</sup> and  $\Delta H$  was 46.78 J.

The changes in reaction enthalpy of sample A were measured by the DSC method of thermal decomposition under nitrogen. The decomposition was carried out in the temperature range of 50 °C – 600 °C. The results of the DSC are shown in Figure 4. The exothermic decomposition of the sample A at a heat rate of 10 °C.min<sup>-1</sup> occurred at 200 °C and continued to 600 °C. The thermogram of the measurements does not contain any peaks which characterize the maximum decomposition temperature, similarly to the case of earlier measurements of the same sample in air at the same heating rate. The change of the enthalpy of reaction of the sample A under nitrogen was 3805.3 J.g<sup>-1</sup> and  $\Delta H$  is 20.09 J.

In the sample B changes in reaction enthalpy were measured by DSC method of thermal decomposition in air. The decomposition was carried out in the temperature range 50 °C – 600 °C. The results of the DSC are shown in Figure 5. The exothermic decomposition of the sample B at a heat rate of 10 °C min<sup>-1</sup> occurred at 200 °C and continued to 560 °C. The temperature at which the maximum heat release rate of sample B in air occurred was 364 °C. The second peak occurred at 427.9 °C. The change in the reaction enthalpy of the sample B is 9184.9 J.g<sup>-1</sup> and  $\Delta H$  is 48.772 J.

DSC thermograms results of the sample B in the atmosphere of nitrogen are in figure 6. The exothermic decomposition of the sample B at a heat rate of 10 °C.min<sup>-1</sup> occurred at 200 °C and continued up to 600 °C. There were no significant exothermic reactions in both samples and therefore not visible exothermic peaks. The reaction enthalpy change of the sample B in a nitrogen atmosphere was 2416.8 J.g<sup>-1</sup> and  $\Delta H$  was 13.05 J.

DSC thermograms results of the sample C in the atmosphere of air ranged in temperature interval 50 °C – 600 °C and are shown in figure 7. The exothermic decomposition of the sample C at a heat rate of 10 °C.min<sup>-1</sup> occurred at 200 °C and continued up to 540 °C. The temperature at which a maximum heat release rate occurred in the sample C (air atmosphere) was 364 °C. The second peak occurred at 498.1 °C. The reaction enthalpy change of the sample A was 8340 J.g<sup>-1</sup> and  $\Delta H$  was 44.87 J.

DSC thermograms results of the sample C in the atmosphere of nitrogen are in figure 8. The decomposition was conducted in the temperature range of 50 °C – 600 °C. The exothermic decomposition of the sample C at a heat rate of 10 °C.min<sup>-1</sup> occurred at 200 °C and continued up to 600 °C. There were no peaks which characterize the maximum decomposition temperature as in the previous measurement of the same sample C in the air atmosphere at a heat rate of 10 °C.min<sup>-1</sup>. The reaction enthalpy change of the sample A was 2655.2 J.g<sup>-1</sup> and ΔH was 13.70 J.

## CONCLUSION

Thermal properties of lignocellulose pellets were measured by Differential Scanning Calorimetry method. The heat rate during experiments was 10 °C min<sup>-1</sup> in the atmosphere of air and inert gas. During measurements in air two peaks of maximum weight loss at each test sample were observed. The first peak of decomposition occurred at 320 °C in sample A and in sample B and C similar decomposition peak occurred at 364 °C. The largest amount of the reaction enthalpy change was observed for the sample B – 9184.9 J.g<sup>-1</sup> and the ΔH was 48.772 J. Peaks reached their maximum decomposition in the atmosphere of nitrogen at a temperature of 600 °C. The highest amount of the reaction enthalpy change was observed in the sample A – 3805.3 J.g<sup>-1</sup> and the ΔH was 20.092 J.

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## References:

1. AEBIOM. 2008. *A Pellet Road Map for Europe*. European Biomass Association.
2. OLLSON, M. 2002. *Wood pellets as low-emitting residential biofuel*. Göteborg, Sweden: CHALMERS UNIVERSITY OF TECHNOLOGY.
3. OVEISI, E. et al. 2013. Breakage behavior of wood pellets due to free fall. *Powder Technology*.
4. MALAŤÁK, J., VACULIK, P. 2008. *Biomasa pro výrobu energie. (Biomass for energy generation.)* Prag: Powerprint s.r.o. ISBN 978-80-213-1810-6
5. LEHTIKANGAS, P. 2000. Storage effects on pelletized sawdust, logging residues and bark. *Biomass Bioenergy*, 19, pp. 287–293.
6. NILSSON, D., BERNESSON, S., HANSSON, P. 2011. Pellet production from agricultural raw materials – a systems study. *Biomass Bioenergy*, 35, pp. 679–689.
7. VERMA, V. K., BRAM, S., DELATTIN, F., LAHA, P., VANDENDAEL, I., HUBIN, A. et al. 2012. Agro-pellets for domestic heating boilers: standard laboratory and real life performance. *Appl. Energy*, 90, pp. 17–23.
8. BEALL, F.C. 1971. Differential calorimetric analysis of wood and wood components. *Wood Sci Technol*, 5, pp. 159–175.
9. RATH, J., WOLFINGER, M. G., STEINER, G., KRAMMER, G., BARONTINI, F., COZZANI, V. 2003. Heat of wood pyrolysis. *Fuel*, 82, pp. 81–91.
10. STENSENG, M., JENSEN, A., DAM-JOHANSEN, K. 2001. Investigation of biomass pyrolysis by thermogravimetric analysis and differential scanning calorimetry. *J Anal Appl Pyrolysis*, 58–59, pp. 765–780.
11. GOMEZ, E., VELO, C., BARONTINI, F., COZZANI, V. 2009. Influence of secondary reactions on the heat of pyrolysis of biomass. *Ind Eng Chem Res*, 48, pp. 10222–10233.
12. TELMO, C., LOUSADA, J. & MOREIRA, N. 2010. Proximate analysis, backwards stepwise regression between gross calorific value, ultimate and chemical analysis of wood. *Bioresource Technology*, Vol. 11, 101, pp. 3808-3815.

13. HORVÁTH, J., BALOG, K. 2013. Teplota vznietenia usadeného a rozvíreného prachu z priemyselne vyrábaných drevených peliet. (Ignition temperature of the adhering and turbid dust from the industrially produced wood pellets.) Žilina: STRIX, Vol. 1., pp. 101-105. ISBN 978-80-89281-90-9

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