KAUNAS UNIVERSITY OF TECHNOLOGY

LITHUANIAN ENERGY INSTITUTE

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# INVESTIGATION OF PELLETIZED BIOMASS THERMAL DEFORMATIONS DURING PYROLYSIS

Summary of Doctoral Dissertation

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LIETUVOS ENERGETIKOS INSTITUTAS

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# PIROLIZĖS METU ATSIRANDANČIŲ GRANULIUOTOS BIOMASĖS TERMINIŲ DEFORMACIJŲ TYRIMAS

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# THE CONTENT OF THE SUMMARY OF DOCTORAL DISSERTATION

## INTRODUCTION

Increasing requirements for environmental protection due to global climate change is forced to reduce the usage of fossil fuels and replacing it by the renewable fuel sources for electricity and heat production. One of technological ways to convert biomass into heat or electrical energy is gasification process in which the solid fraction is converted into fuel gas. As biomass gasification is a complex process (supply of fuel, drying, pyrolysis, gasification, combustion, ash disposal), it is confronted with the fuel bridging problems and ash adhesion problems. Large scale (> 10 MW) gasification plants are confronted with biofuel bridging where fuel particles clump together to form a solid fraction due to poorly designed fuel supply system or due to different fuel particle size, moisture content. Also bridging problems occur in ash removing system, where ashes form slag due to wet fuel and too high temperature. However, these problems are avoided by improving the fuel transportation system using a nitrogen flow to prevent fuel clogging or installation of a vibration system for moving clogged fuel. Ash slag formation is avoided by reducing the temperature of the oxidation zone blowing steam to ash layer or installing additional ash removal system.

Increasing usage of renewable energy results in increased production of medium and small scale gasification reactors. These reactors are usually adapted to the granular biomass due to easier and comfortable fuel transportation, simpler production of fuel feed system, simpler gasification process management. Furthermore, gasification gives opportunity to use not only high-quality biomass (wood, straw, peat), but lower quality fuels, such as forest floor and wood waste after harvesting, water waste, chicken manure and others. However, studies of granular biomass gasification reveals that often there occur fuel bridging problems, even when using high quality wood pellets. During gasification, biomass pellets are moving from the pyrolysis zone to the oxidation zone clumps together possibly due to the expansion of pellets and stops the entire process. Such pelletized fuel bridging is described in scientific works, but ongoing biomass thermal deformation is not known and bridging causes remain undisclosed.

Performed review on researches of biomass thermal deformations shows that granular biomass thermal deformation taking place during thermal decomposition are not fully investigated and their causes and regularities are not identified. The object of this research – study of granular biomass thermal deformations performing experiments of biomass size and mass changes during pyrolysis from 300 °C to 900 °C temperature.

**Aim of the work**

Investigation of pelletized biomass thermal decomposition to determine the regularities of thermal deformations and create mathematical equations that define the biomass thermal deformations dependence on temperature.

**Tasks of the work**

For implementation of the scientific goal, the following main tasks should be performed:
1.  Research biomass thermal deformations performing an experimental study of wood particle, wood pellet and wheat straw pellet pyrolysis.
2.  Determine the cause of pelletized biomass thermal deformations.
4.  Determine regularities of biomass thermal deformations and their dependence on temperature.

**Relevance of the work**

The solid biofuel is converted into valuable fraction of fuel as gas during gasification process. Obtained gasses are used to generate electricity or heat. However, gasification is a complex process in which there is a wide range of fuel adhesion problems affecting the entire process. One of the fuel sticking problems caused by use of pelletized biomass, when pellets slide from the pyrolysis zone to the oxidation zone and stick together into lumps causing to stop further process. In order to improve the gasification reactor, first of all the ongoing fuel thermal deformations in pyrolysis zone should be investigated, causes of thermal deformations should be identified and its dependence on temperature and type of biomass should be determined.

**Novelty of the work**

By analysing swelling and shrinking of granular biomass (wood pellets and straw pellets) during thermal decomposition, by establishing the regularities and presenting the equations describing the thermal deformations of biomass, a deeper understanding of the pyrolysis process was presented and the mechanism of biomass thermal deformations was revealed.

**Significance of research results**

Estimated regularities and cause of biomass thermal deformations upon pyrolysis could be used to improve the existing numerical models of biomass pyrolysis and offer technological solutions to develop more efficient biomass gasification equipment.

**Statement presented for defense**

1. During pyrolysis thermal deformations of biomass take place where pelletized biomass firstly expands and after it - shrinks.
2. During pyrolysis pelletized biomass expands due to complicated emission of water vapor and volatiles, and intensified emission of water vapor and volatiles cause shrinkage of pellets.
3. During pyrolysis formed char expands at high temperature due to intensive emission of volatiles.

**Author's contribution in analysed problem**

The author R. Paulauskas performed analysis of scientific information on biomass thermal conversion and ongoing thermal deformation of biomass, designed and constructed experimental stand for investigation of biomass thermal deformations during pyrolysis. In order to determine the cause of biomass thermal deformation, author carried out surface analysis of pyrolysed biomass samples. In accordance with the results of biomass thermal deformation, R. Paulauskas developed mathematical equations, which describe dependence of biomass size changes on the heating temperature.

**Scientific approbation of dissertation**

Research results presented in the dissertation were published in 1 scientific article in publications with a citation index at the "Thomson Reuters" database "Web of Science Core Collection" and 1 article published in journal referred in international scientific database. Research results were also presented at 6 conferences, 5 of them international ones.

# 1. LITERATURE REVIEW

Biomass biochemical composition, thermal decomposition properties of biomass constituent components and works of biomass thermal deformations, using different thermal decomposition technology are presented in literature review. It mainly focuses on thermal deformation of wood particles and pellets analysis. This choice was made due to a gasification process, where using pelletized biomass is encountered the problem of fuel sticking in lumps caused by ongoing thermal deformation of pellets. To author's knowledge, openly available literature mostly contains works where thermal deformations of wood particles are investigated and just a few works have analyzed thermal deformations of pelletized biomass (wood, straw and refuse-derived fuel (RDF) pellets) at high temperatures. In these works are indicated that RDF pellet and straw pellet expands at high temperature and RDF pellet expands due to containing plastic in pellets. However, the cause of RDF pellet swelling not clarify the swelling of straw or wood pellet, which biochemical composition and structure are different comparing with RDF pellet. Besides, in reviewed works is not defined the temperature at which swelling of pellets starts and ends. According to literature review, it was decided to perform experimental study of thermal deformations of various properties biomass samples. Wood pellets, straw pellets and wood particles were selected for this research. Different properties of biofuels will allow to determine how biochemical composition and structure influence biomass thermal deformation during thermal decomposition.

In accordance with literature review, experimental study of biomass thermal deformations was divided into three stages:

• Research of wood particle, wood pellet and straw pellet thermal deformation during pyrolysis from 300 °C to 900 temperature where the changes of samples diameter are captured over time and measured the center temperature of the samples. This study will determine the regularities of biomass thermal deformations.

• Research of wood particle, wood pellet and straw pellet mass loss from 300 °C to 900 temperature along measuring the temperature of the samples center to be compared with obtained results of biomass thermal deformation and determine the impact of volatile emission during biomass thermal deformation.

• Analysis of pyrolyzed samples surface to determine the cause of ongoing biomass thermal deformations.

As the ongoing biomass thermal deformations cause fuel bridging problems during gasification, mathematical equations will be developed according to obtained results from investigation of biomass thermal deformations. These equations describe the evolution of biomass thermal deformations with increasing heating temperature.

## 2. EXPERIMENTAL SETUP AND METHODOLOGY

### 2.1. Material characterization

Wood pellets, wheat straw pellets and cylindrical wood particles were used for experimental investigation of biomass thermal deformations. The diameter of all samples was 8±0.5 mm, length 20±1 mm. Before the investigation of biomass thermal deformations, the ultimate, proximate and biochemical analyses of samples were acquired. The ultimate and proximate analysis were performed using an IKA C5000 calorimeter, a Flash 2000 CHNS analyzer and a NETZSCH STA 449 F3 Jupiter thermogravimeter (TG, DTG,) in accordance with: LST EN 14774-1 (moisture content), LST EN 14918 (HHV), LST EN 14775 (ash content), LST EN 15148 (volatile content) and LST EN 15104 (CHNS content). Biochemical composition of samples was obtained by NDF and ADF analyzes using an ANKOM procedure with a polyester bag (PBT) and the method of Van Soest (VST). The obtained characteristics are shown in Table 1.

**Table 1.** Characteristics of biomass samples.

| Parameter | Wood pellet | Wheat straw pellet | Wood particle |
|---|---|---|---|
| Ultimate analysis wt% (dry basis) | | | |
| Carbon | 51.22 | 45.84 | 49.59 |
| Hydrogen | 5.56 | 5.16 | 5.36 |
| Oxygen (diff.) | 43.2 | 47.67 | 45.03 |
| Nitrogen | 0.01 | 1.12 | 0.01 |
| Sulphur | 0.01 | 0.21 | 0.01 |
| Proximate analysis | | | |
| Moisture content, % | 6.68 | 8.28 | 5.83 |
| Ash, % | 0.7 | 4.9 | 0.2 |
| HHV, MJ/kg | 18.77 | 16.57 | 17.49 |
| LHV, MJ/kg | 17.47 | 15.33 | 16.25 |
| Biochemical analysis wt% (dry basis) | | | |
| Hemicellulose | 14.2 | 29.9 | 19.6 |
| Cellulose | 51.7 | 43.5 | 61.3 |
| Lignin | 25.4 | 6.22 | 14.4 |

### 2.2. The methodology for experimental investigation of biomass thermal deformations during pyrolysis

Investigation of biomass thermal deformations was carried out at 300 – 900 °C heating temperature using the electrically heated horizontal tubular furnace Nabertherm RS 80/500/13. A scheme of this experimental setup is presented in Fig. 1.
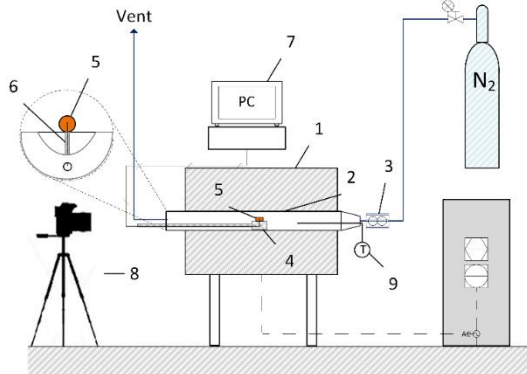
**Fig. 1**. Scheme of experimental rig for biomass thermal deformations:
1) electrical furnace Nabertherm RS 80/500/13; 2) a heating tube; 3) a flow meter; 4) a pad for sample; 5) biomass particle; 6) thermocouple installed into pad; 7) a computer to collect data; 8) a photo camera; 9) thermocouple to measure nitrogen temperature.

A heating tube (2) with outer diameter of 80 mm and length of 850 mm was mounted inside the furnace (1) and heated on both sides. One end of the tube was supplied by a nitrogen flow, which temperature was measured by an installed K-type thermocouple. The other end of the tube was left open for placement of a special pad with pelletized fuel particle and for capturing the sample size changes. The nitrogen flow of 8 l/min controlled by the flow meter (3) was fed into the furnace when the temperature reached the desired level. The special pad (4) with the sample (5) was inserted in the middle of the working tube through the open end. K-type thermocouple (6) was installed in the pad for sample center temperature measurement during the pyrolysis process. The measured center temperature values were collected by data logger PICO TC-08 and sent to the computer (7). The pelletized fuel particle was captured with the digital camera Fuji HS25 EXR (8) through the open end of the tube during the pyrolysis process until the pellet stopped shrinking. The initial diameter of the sample was measured with a varnier caliper, which provided precision to 0.05 mm before the sample load. The measurements were performed with wood particle, wood and straw pellets at constant heating temperature from 300 °C to 900 °C by 50 °C step repeating 5 times. The high-resolution (1920 x 1080 pixels) recorded videos of pelletized fuel particles changes were analyzed using GIMP software. Each 150th recorded video frame was converted into a photo and the pelletized fuel particle diameter was measured with a digital ruler in pixels. Obtained data in pixels was expressed as the relative units.

## 2.3. The methodology for experimental investigation of biomass devolitilization during pyrolysis

Using the electrically heated horizontal tubular furnace Nabertherm RS 80/500/13, mass loss of biomass samples was determined during pyrolysis. The furnace was assembled vertically and a working sillimantin tube (2) was mounted inside the furnace (1). One end (lower) of the working tube was supplied by a nitrogen flow, the other end (upper) of the tube was left open for placement of a bracket with sample. A special frame with moving tray was mounted on the top of furnace and a digital scale Kern Kern EW 420-3NM was placed on the tray. A special rod with bracket of stainless steel mesh with K-type thermocouple for measurement of sample center temperature, was attached to measurement point at scale bottom. One additional thermocouple (9) was installed in the working tube and used to measure heating temperature affected by nitrogen flow. Nitrogen flow of 8 l/min, controlled by the flow meter (3) was maintained into the furnace when the heating temperature reached the desired level. From this point, the sample was placed in the bracket, fed into heating tube through open end and measurements started. Mass loss data was collected over software Kern BC2006 and the center temperature values were collected by data logger PICO and all data sent to the computer (7). When mass loss of sample was no more observed, bracket with sample was extracted from heating tube and left to cool down. Measurements were repeated 3 times in range of 300 – 900 °C heating temperature by 50 °C step.
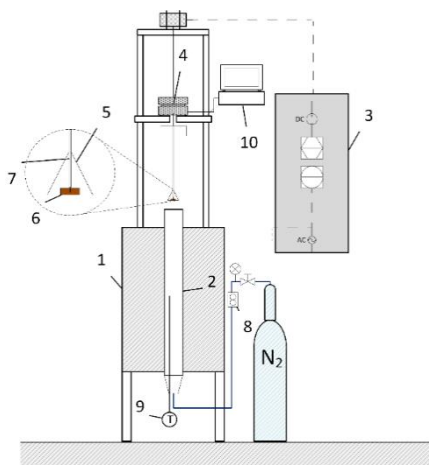


**Fig. 2.** Experimental scheme of mass loss determination of biomass sample
1) electrical furnace Nabertherm RS 80/500/13; 2) the heating tube; 3) the furnace controller; 4) digital scales Kern Kern EW 420-3NM; 5) a sample holder; 6) biomass particle; 7) thermocouple installed into sample; 8) the flow meter; 9) thermocouple to measure nitrogen temperature; 10) a computer to collect data.

# 3. RESULTS AND DISCUSSION

## 3.1. Thermal analysis of used samples

In order to identify ongoing thermal conversion processes during pyrolysis, thermal analysis of samples was acquired. Ongoing thermochemical processes inside cylindrical wood particle, wood pellet and wheat straw pellet can be divided into several stages:

- The first endothermic peak at 100 °C shows the first stage – the heat absorption due to water evaporation which lasts till 115 °C and influences slight mass loss in all samples.

- The second stage occurs with hemicellulose and cellulose decomposition and lignin heating, which takes place in 180 – 340 °C temperature for wheat straw pellet, 210 – 365 °C for wood pellet and wood particle (see Fig. 3). Slight exothermic peak around 300 °C indicates hemicellulose of wood pellet and particle decomposition. With growing temperature hemicellulose decomposition is completed and endothermic peak appears due to cellulose devolatilization. Endothermic peak of wood particle is more intensive than wood pellet, what indicates higher heat absorption. According to the greater mass loss rate in Fig. 3 and higher amount of cellulose (Table 1) in wood particle, it is related to cellulose structure braking and volatile release. The straw pellet decomposition differs from wood samples, as it can be seen from DSC curves: emission of heat starts at 180 °C and the exothermic peak appears at 340 °C, while DSC curves of wood samples indicate endothermic reactions. Though decomposition of cellulose is high endothermic, in this way endothermic peak at 370 °C is neglible, comparing with endothermic peaks of wood samples (see Fig. 3). It could be related with biochemical composition (see Table 1) and crossover of hemicellulose and cellulose decomposition: emitted energy amount from hemicellulose is approximately enough to heat up cellulose, brake bounds and start to release volatiles and only slight amount of energy is absorbed from the environment. According to heat flow rate, presented in Fig. 3, the most intensive decomposition of hemicellulose occurs at 285 °C (the maximum of heat flow rate) and after that heat flow rate decreases due to emitted heat absorption. The decrease of heat flow rate below zero indicates lack of energy –
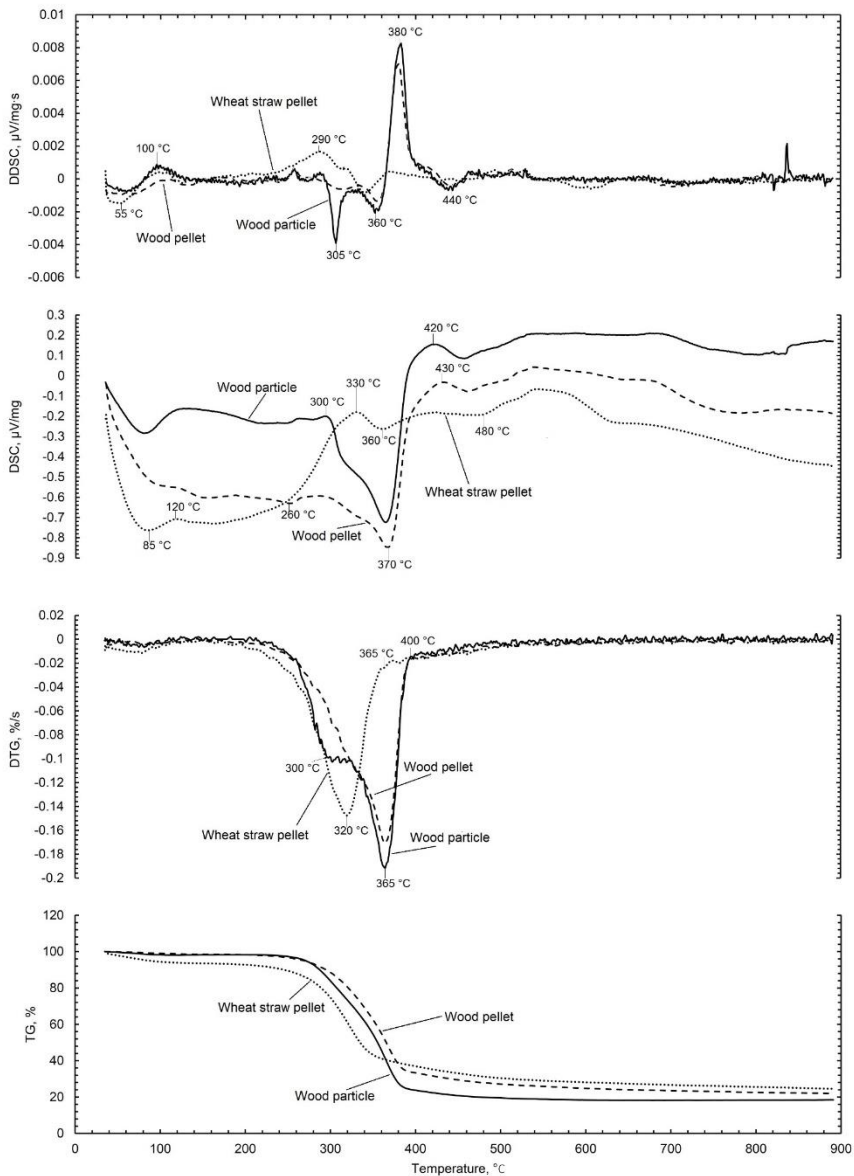
**Fig. 3.** Thermal analysis of biomass samples in nitrogen atmosphere

the absence of energy from hemicellulose to devolatilize fully cellulose – and the deficit is gained absorbing energy from environmental heating, resulting in endothermic peak (Fig. 3).

- The third stage consists of lignin decomposition. According to literature (Beall ir Eickner, 1970), the heat flow increase from 340 °C till 380 °C for straw pellet and from 370 °C till 400 °C for wood samples resulting in exothermal process (Fig. 3) is due to volatile emission. At the end of lignin devolitilization, char formation takes place.

## 3.2. Thermal deformations of biomass

### 3.2.1. Heat effects of biomass samples during pyrolysis

Along the experiment of biomass thermal deformations, center temperature of wood particle, wheat straw pellet and wood pellet was measured and center heating rates were calculated by the first order derivative ($\Delta T_{center}/\Delta t$) at 300 °C, 400 °C, 550 °C, 650 °C and 900 °C heating temperature to identify thermochemical processes determined by thermal analysis.
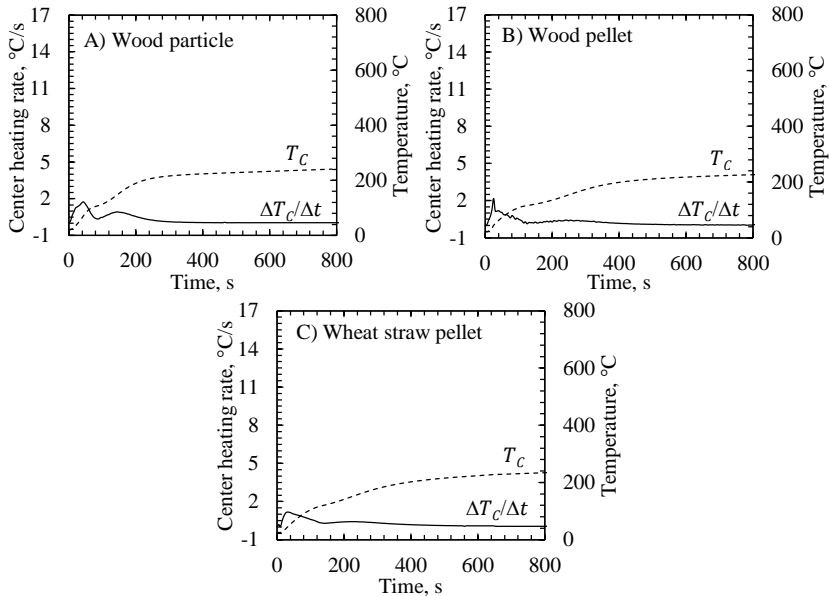


**Fig. 4.** Center temperature profiles of wood particle, wood pellet and wheat straw pellet at 300 °C temperature

The obtained center temperature graphs of samples show that the center temperature profiles changes become more intensive with the heating temperature increase.

At low heating temperature (300 °C) only water evaporation and a slight decomposition of biomass samples dominates. Dehydration process starts after 20 seconds from the beginning of the experiment. According to the sample center heating rate curve in Fig. 4, the intensity of the dehydration process identifies peak of center heating rate at 40 s, which disappears when samples' centers heat up over 150 °C. Variation of samples' center heating rate associated with dehydration process since the water specific heat (cp = 420 kJ/kg) is less than the water evaporation specific heat (cp = 2250 kJ/kg), and evaporation of water requires a higher heat amount than the temperature increase up to 100 ° C, thus the center of the sample heating rate is decreasing (Fig. 4).
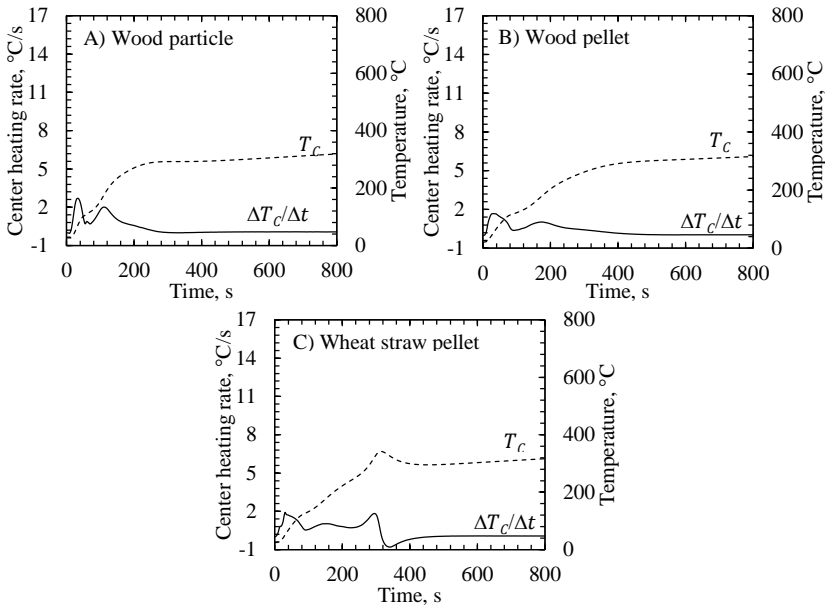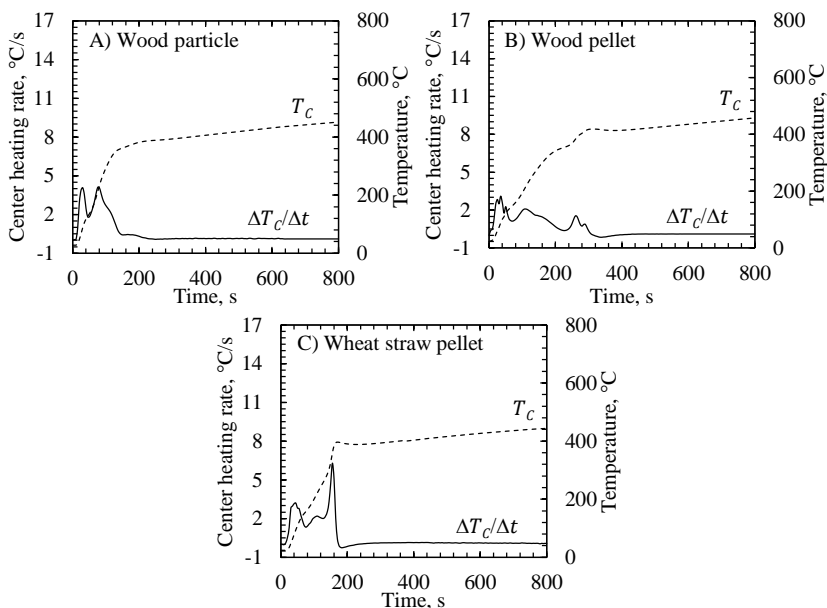


**Fig. 5.** Center temperature profiles of wood particle, wood pellet and wheat straw pellet at 400 °C temperature

At higher temperature (400 °C), partly hemicellulose, lignin and cellulose conversion takes place in the end of evaporation process (center temperature of samples - 150 °C) (Fig. 5). According to the profiles of samples center heating rate (see Fig. 5) wood particle overheats faster than pelletized samples after the dehydration process and thermal conversion of hemicellulose starts earlier. Conversion of hemicellulose in wood particle starts when center heats up over 170

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°C and in wood pellets - over 190 °C temperature. Observed peaks in center heating rate profiles of samples indicates that thermal conversion of wood pellet is slower than wood particle. It is possible that slower decomposition is affected by higher content of lignin in wood pellet, which the thermal degradation is more complex than that of cellulose or hemicellulose.

It is seen from Fig. 5 that wheat straw pellet overheats slower than the wood samples. Peaks of center heating rate are wide, indicating a slower sample overheating. But from 240 s is observed intensive increase of center heating rate of wheat straw pellet and peak of center temperature occurs at 315 °C temperature (Fig. 5 C). According to biochemical composition of the wheat straw pellet (see. Table 1) and thermal analysis data (Fig. 3) this effect is due to higher hemicellulose content of straw pellet and tendency of thermal degradation, where the released heat is sufficient to heat the sample. Furthermore, the thermogravimetric curve (Fig. 3) reveals that most intensive mass loss of wheat straw pellet coincide with the center temperature peak of wheat straw pellet (see Fig. 5 C), what indicates decomposition of hemicellulose and cellulose.



**Fig. 6.** Center temperature profiles of wood particle, wood pellet and wheat straw pellet at 550 °C temperature

With growing heating temperature (550 °C), water evaporation process shifts towards higher center temperatures for all samples till 160 °C. The wood particle dehydrates faster than pelletized samples and after this stage occurs

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exothermic reactions of hemicellulose decomposition causing peaks of center heating rate (Fig. 6). Decreasing heating rate of wood particle indicates cellulose decomposition. The tendency of thermal conversion of pelletized samples is different to wood particle: dropped heating rates of wheat straw and wood pellets (Fig. 6) slowly increase after water evaporation. From wheat straw pellet center temperature point of 300 °C occurs peak of center heating rate indicating devolitilization process, while peak of heating rate of wood pellet occurs from 400 °C center temperature point. According to thermal analysis (Fig. 3) and biochemical composition (Table 1) devolitilization of wheat straw pellet at lower temperatures is caused by crossover of hemicellulose and cellulose decomposition where emitted energy amount from hemicellulose is used to heat up cellulose, brake bounds and start to release volatiles.
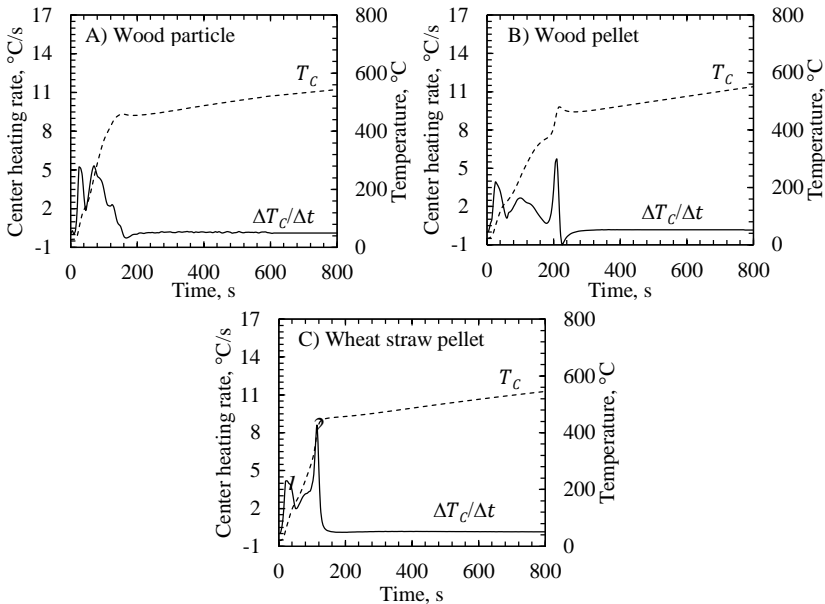


**Fig. 7.** Center temperature profiles of wood particle, wood pellet and wheat straw pellet at 650 °C temperature

At higher temperatures (650 °C, 900 °C ), the trend of center overheating in the biomass samples remains the same as determined at 550 C temperature (see Fig. 6), however, the decomposition is about 1.65 time faster at 650 °C and about 2.2 time faster at 900 °C heating temperature due to the received higher amount of heat. According to obtained biomass decomposition proceeding and duration depend on the heat transfer, which is determined by the heating temperature.
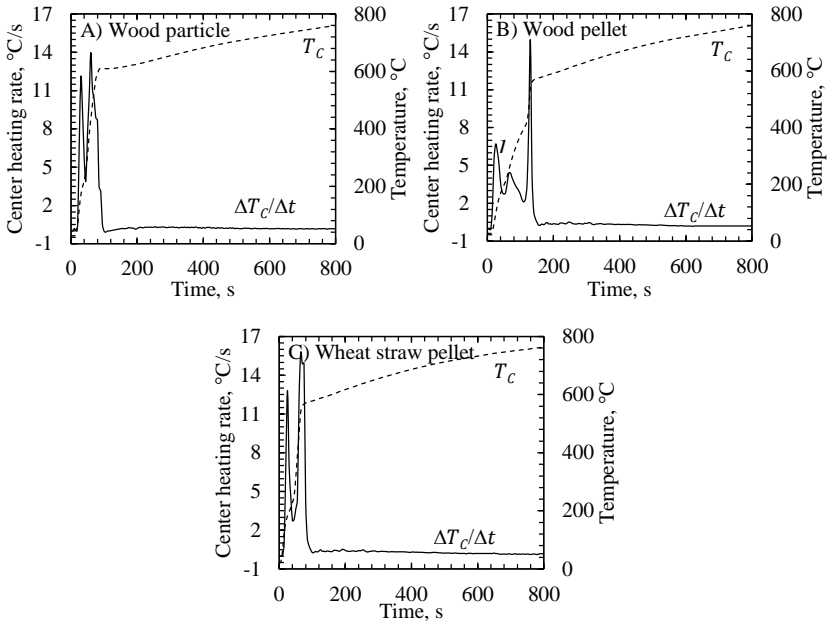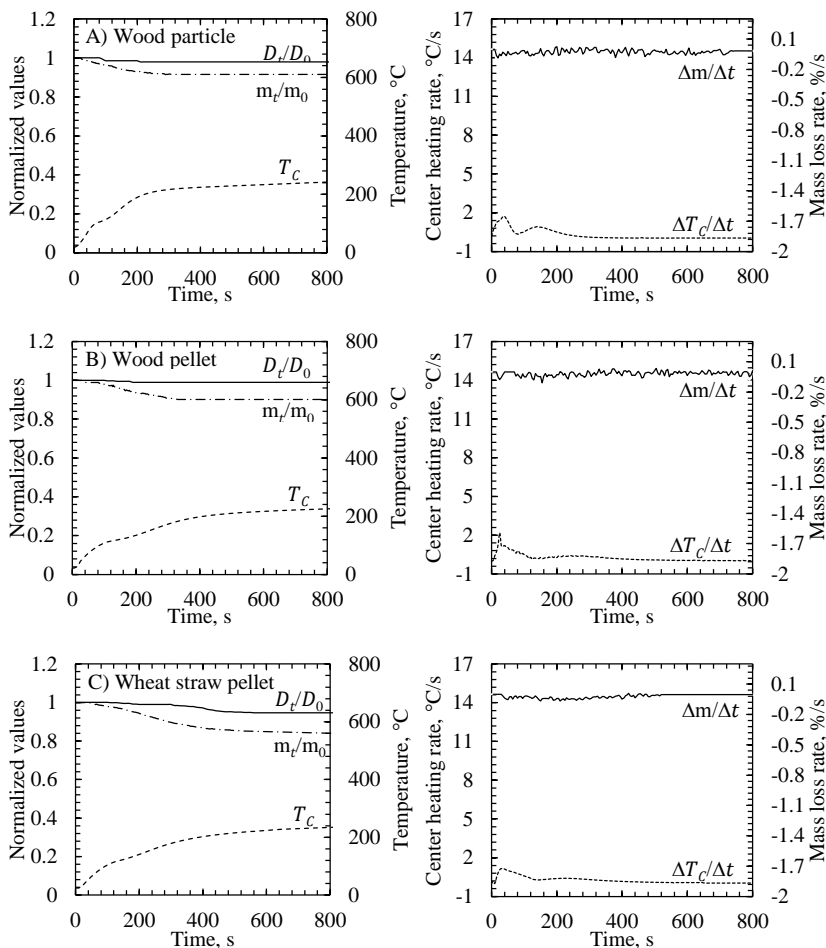
**Fig. 8.** Center temperature profiles of wood particle, wood pellet and wheat straw pellet at 900 °C temperature

### 3.2.2. *Diameter changes and mass loss of biomass samples during pyrolysis*

In order to obtain a deeper knowledge about thermal deformations of pelletized biomass which cause fuel agglomeration in pyrolysis zone during gasification, wood particle, wood pellet and wheat straw pellet diameter changes and mass loss has been studied during pyrolysis at various temperatures. Obtained data from experiments was analysed and diameter changes and mass loss at 300 °C, 400 °C, 650 °C and 900 °C from both experiments are presented in this chapter.
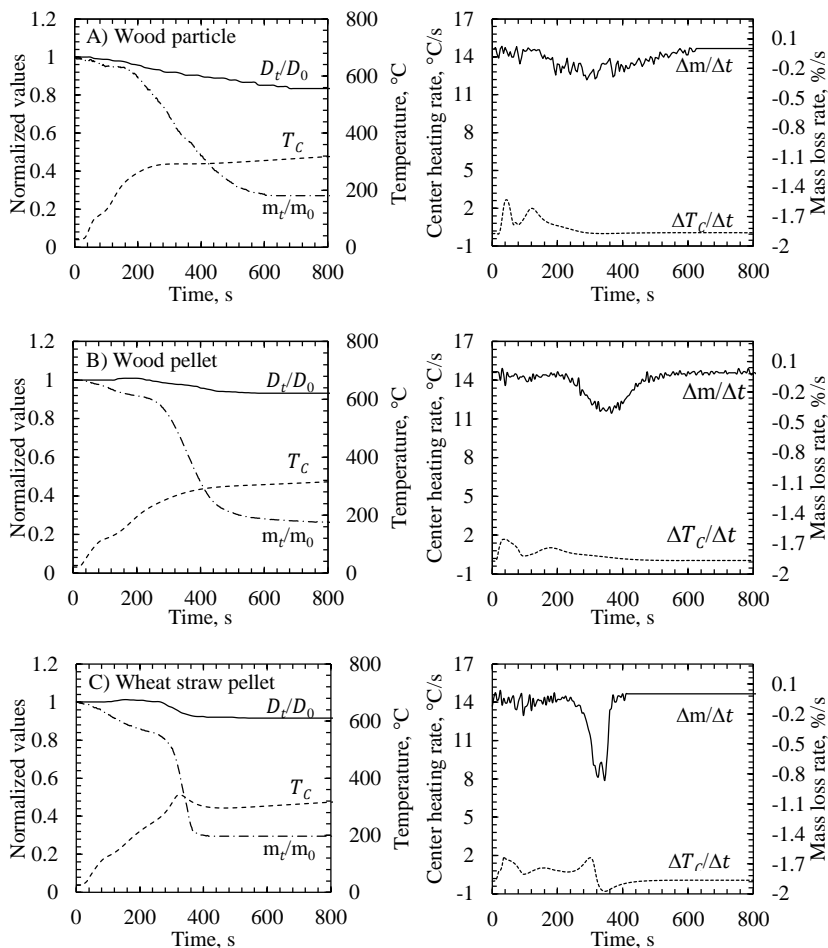
The analysis of wood particle, wood pellet and straw pellet thermal deformations at low temperature (300 ° C) provides that during pyrolysis mainly dominates the dehydration process and a slight release of volatiles from biomass samples (Fig. 9). Dehydration process in all biomass samples is recorded after 20 seconds from the start of the experiment. In view of the moisture content of the samples (see Table 1), wood particle dehydration process ends when center heats up to 170 ° C, whreas wood pellet – to 210 °C. During dehydration process, wood

**Fig. 9.** Diameter changes and mass loss of wood particle, wood pellet and wheat straw pellet at 300 °C temperature

particle diameter shrinks to 98.5% of the original diameter (Fig. 9 A). Water evaporation from pelletized biomass is slower. According to mass loss of samples, water removal ends after 210 s in wood pellet, after 190 s in wheat straw pellet (Fig. 9). It was found that a slight volatile emission occurs after dehydration process. Wood particle diameter shrinks to 97.9% of the original diameter, losing 8.3% of initial mass, and further thermal deformation is no more observed. Wood pellet diameter remains unchanged with increasing volatile emission. The wood pellet mass loss (see. Fig. 9 B) reveals that the release of volatile content stopped

or remains negligible when wood pellet center heats over 220 °C temperature. At 300 °C temperature wood pellet loses 10% of the initial mass. Wheat straw pellet diameter shrinks 5.4% of the initial diameter, losing 15% of the initial mass.
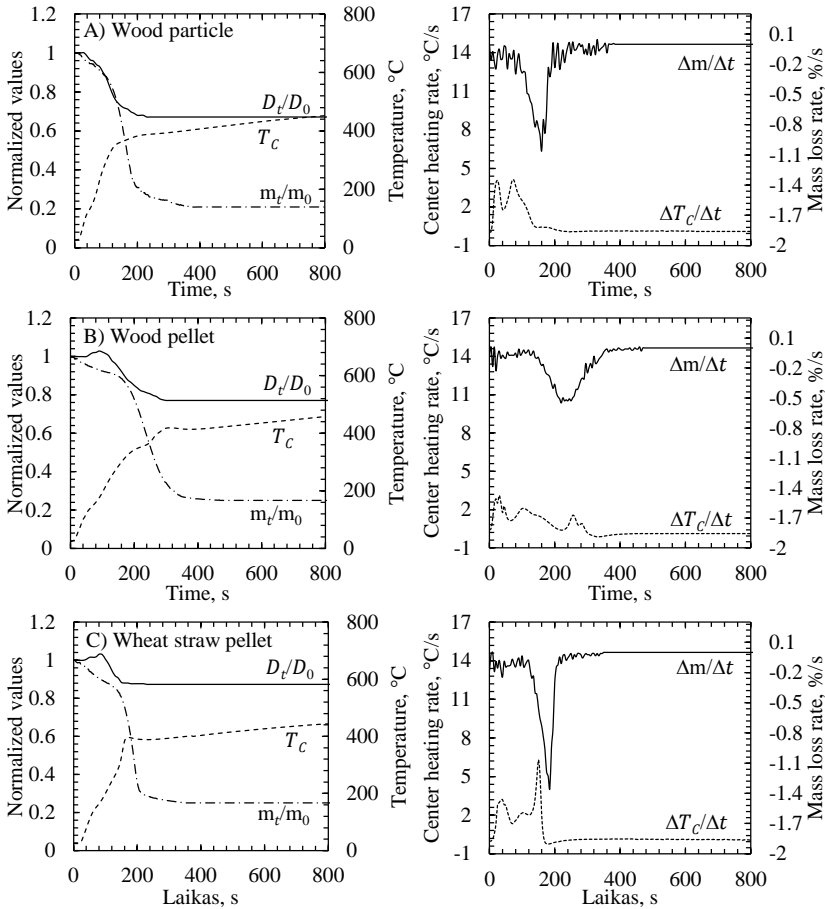


**Fig. 10.** Diameter changes and mass loss of wood particle, wood pellet and wheat straw pellet at 400 °C temperature

At higher (400 °C) heating temperature dehydration process shifts towards higher temperature till 130 °C, whereas with the end of water evaporation starts hemicellulose, lignin and cellulose decomposition. According to center temperature profiles, wood particle overheats faster than pelletized samples and only shrinkage of particle is determined. Particle shrinks to 82.5% of the initial

diameter, thus losing 74% of the initial mass. Meanwhile, at the end of dehydration process pelletized biomass is affected by neglible swelling phenomenon – diameter of pellets expands around 1% (see Fig. 10 B, C). With the beginning of hemicellulose decomposition, expanded pellets start to shrink and in the end of thermal conversion residual diameter is 93.2% and 91.4% for wood and straw pellet respectively with mass loss of 74% and 72% of initial mass. Besides center temperature peak with maximum at 315 °C appears (Fig. 10 C) during thermal conversion of wheat straw pellet, what indicates full thermal conversion of straw pellet. According to thermal analysis (Fig. 3) and biochemical composition (Table 1), devolitilization of wheat straw pellet at lower temperatures is caused by crossover of hemicellulose and cellulose decomposition, where emitted energy amount from hemicellulose is used to heat up cellulose, brake bounds and start to release volatiles, while wood pellet is affected only by thermal resistance decrease and inception of charring.

Thermochemical processes of biomass samples intensify with increasing heating temperature (550 °C). Dehydration process of wood particle starts 10 seconds before thermal deformation of the particle (see. Fig. 11 A). During dehydration process, wood particles shrink 3.7% of its initial diameter and lose around 6% of the initial mass. With increasing center temperature of wood particle, volatiles' content emission starts due to thermal degradation of hemicellulose and cellulose. Cellulose thermal decomposition identifies the formed mass loss rate peak at 160 second (see. Fig. 11 A). After cellulose decomposition, wood particle loses 46% of the initial mass and shrinks to 71.6% of its initial diameter. During residual lignin decomposition of wood particle, mass loss rate is decreasing to 0%/s. Wood particle shrinks overall to 67.1% of the initial diameter.

Obtained thermal deformation of biomass results at 550 °C heating temperature shows that increasing of heating temperature intensifies the expansion of biomass pellet. During dehydration process, wood pellet expands to 2% of the initial diameter and losses 4% of the initial mass (see Fig. 11 B). At the end of the dehydration process (7% of the initial weight loss), the expanded pellet starts to shrink. In view of the pellet mass loss rate increase from 160 s to 220 s (see. Fig.11 B), the shrinkage of wood pellet is due to hemicellulose and cellulose thermal decomposition. Devolatilization process (total weight loss of 65%) affects the wood pellet structure - diameter shrinks to 82.5% of the initial diameter (see. Fig. 11 B). Residual lignin decomposition and carbon formation stage in wood pellet is not as intense as in wood particle, but this process can be assessed in terms
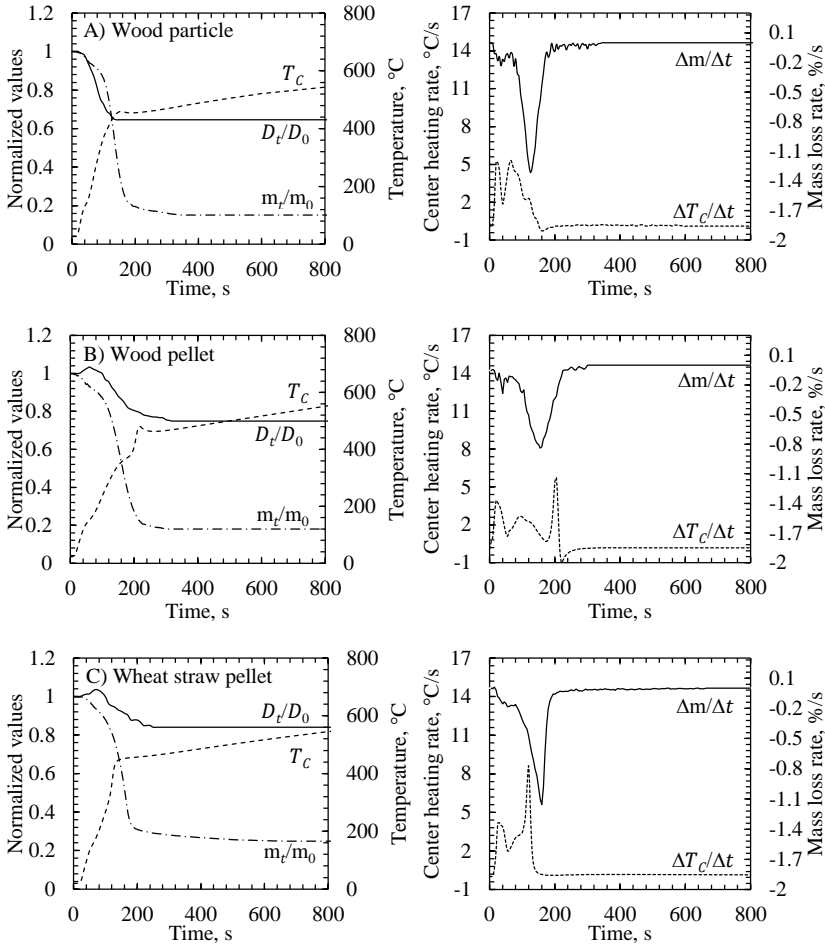
**Fig. 11.** Diameter changes and mass loss of wood particle, wood pellet and wheat straw pellet at 550 °C temperature

of mass loss rate decrease and the center heating rate peak at 260 second (see Fig. 11 B). When center of wood pellet heats over 420 °C temperature, thermal deformations of pellet is no longer observed. Wood pellet shrinks 23.3% of the initial diameter, thus losing 75% of the initial mass.

According to mass loss rate of biomass samples at 550 °C the pyrolysis temperature, thermal decomposition of wheat straw pellet is more intensive than the wood pellet (see Fig. 11 C). During water evaporation stage, wheat straw pellet diameter expands to 3.2% of the initial diameter. The beginning of expanded pellet shrinkage match the peak in mass loss rate graph at 120 s, which indicates the

thermal decomposition of the hemicellulose, cellulose and lignin (Fig.11 C). Wheat straw pellet shrinks to 87.6% of the initial diameter, thus losing 75% of the initial mass.
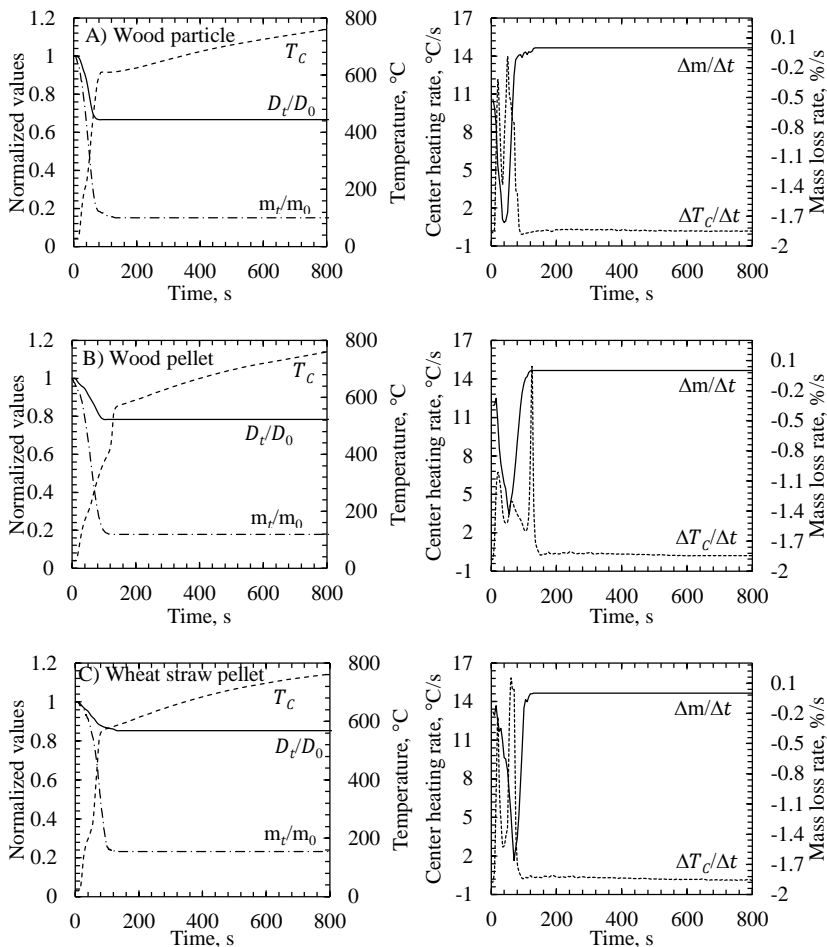


**Fig. 12.** Diameter changes and mass loss of wood particle, wood pellet and wheat straw pellet at 650 °C temperature

With growing heating temperature (650 °C), thermal conversion of samples is more intensive, water evaporation process shifts towards higher center temperatures for all samples till 150 °C (Fig. 12). Wood particle is affected by fast thermal conversion (80% mass loss over 200 s), resulting in shrinkage of 34.3% of the initial diameter. At this temperature point, a swelling effect of pelletized

samples takes place earlier (after 30 s from the beginning of experiment), whereas wheat straw pellet and wood pellet diameter expands by 4.2% and 3.4% of the initial diameter, respectively (Fig. 12).



**Fig. 13.** Diameter changes and mass loss of wood particle, wood pellet and wheat straw pellet at 900 °C temperature

Pelletized samples start to shrink after dehydration process ends and devolitilization starts. Wood pellet diameter shrinks linearly to 75.6% of the initial diameter, wheat straw pellet diameter – to 85.8% of initial diameter, loosing overall 82% and 75% of mass, respectively.

Swelling effect of pelletized samples is no more observed at 900 °C heating temperature and only shrinkage dominates for all samples (Fig. 13). thermal conversion of wood particle is most intensive (85% mass per 100 s) and particle starts to shrink immediately after placement into heated tube: shrinks to 66% of the initial diameter. Thermal conversion of pelletized samples is identical to each other (ends after 120s with 83% and 77% mass loss for wood pellet and wheat straw pellet, respectively) and 1.2 time slower than that of wood particle. Measured shrinkage values are 21.6% for wood pellet and 14.6% of initial diameter for wheat straw pellet (Fig. 13).

### 3.2.3. *Mechanism of pelletized biomass swelling*

According to obtained results, shrinking and swelling of biomass samples is related to pyrolysis temperature and heat transfer inside the particles. With growing heating temperature, the absorbed heat amount increases, causing more intensive devolitilization process.
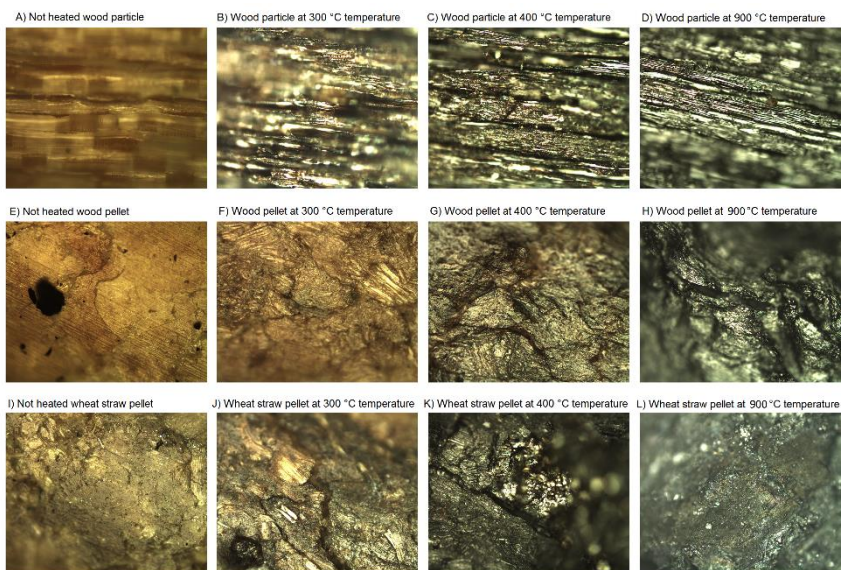


**Fig. 14.** Images of wood particle, wood and wheat straw pellets surface at different heating temperature acquired by optical microscope at 10X magnification.

For better understanding of thermal deformation mechanism, microscopy analysis of pyrolysed samples at various temperatures was performed using optical Olympus BX51 microscope with high-resolution digital color camera Go-21 controlled by QCapture software. Acquired pictures with 10X optical magnification are presented in Fig. 14.

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Not heated wood particles' surface is a uniform structure where mostly fibers are directed in one line (Fig. 14 A), while surfaces of pelletized samples consist of chaotically located pressurized sawdust or straw scobs with occurred vessels (Fig. 14 E, I). The surface of wood particle affected by 300 °C heating temperature starts charring (Fig. 4 B), which indicates intensive thermochemical processes. With growing heating temperature these processes cause wide cracks on surface and volatiles are emitted easily without any structure expansion (Fig. 14 C, D). As it is shown in Fig. 14 F, J, pelletized biomass surface is less affected by thermochemical processes at 300 °C temperature. Due to slower heat transfer according to profiles of heating rates, only micro cracks and neglible tar melting occur on the surface. On the surface, affected by 400 °C heating temperature, tar melting is more intensive (Fig. 14 G, K) covering cracks. Besides, vessels are formed on melted surface zones due to complicated volatiles emission. Expansion of wood and wheat straw pellets occurs due to formed water vapour and volatile compounds inside pellet which fail to evaporate from the pellet center due to tars condensation on the surface, which influence the pellet porosity decrease – melted surface in Fig. 14 F, G, J, K. In this way, water and volatile substances are emitted from deeper layers of pellet, causing increment of internal pressure near the pellet's surface, which destroys the surface structure of the pellet (swelling occurs) and spreads cracks on surface (Fig. 14)) by freeing water vapour and volatiles. Shrinkage of expanded pellets starts after evaporation of volatile compounds. The swelling phenomenon is no longer observed when the heating temperature is over 900 °C. The pellets overheat so quickly that the melted compounds from the surface layer are decomposed by high amount of heat and the evaporation of volatiles becomes uncomplicated from the deeper layers without eruption (Fig. 14 H, L).

## 3.3. Regularities of thermal deformations of biomass

Final swelling and shrinking values and residual mass of biomass samples, increasing heating temperature, are presented in Fig. 15. Expansion of wood pellet depends on heating temperature and maximum values in range of 0.9 – 3.93% are proportional to heating temperature from 400 °C to 600 °C. With growing heating temperature over 600 °C swelling effect linearly diminishes and over 900 °C heating temperature it disappears. However, the tendency of wheat straw pellet expansion depends less on heating temperature: expansion values fluctuate in the range of 2.93 – 3.68% from 450 to 850 °C heating temperature, and from 850 °C swelling phenomenon disappears (Fig. 15).
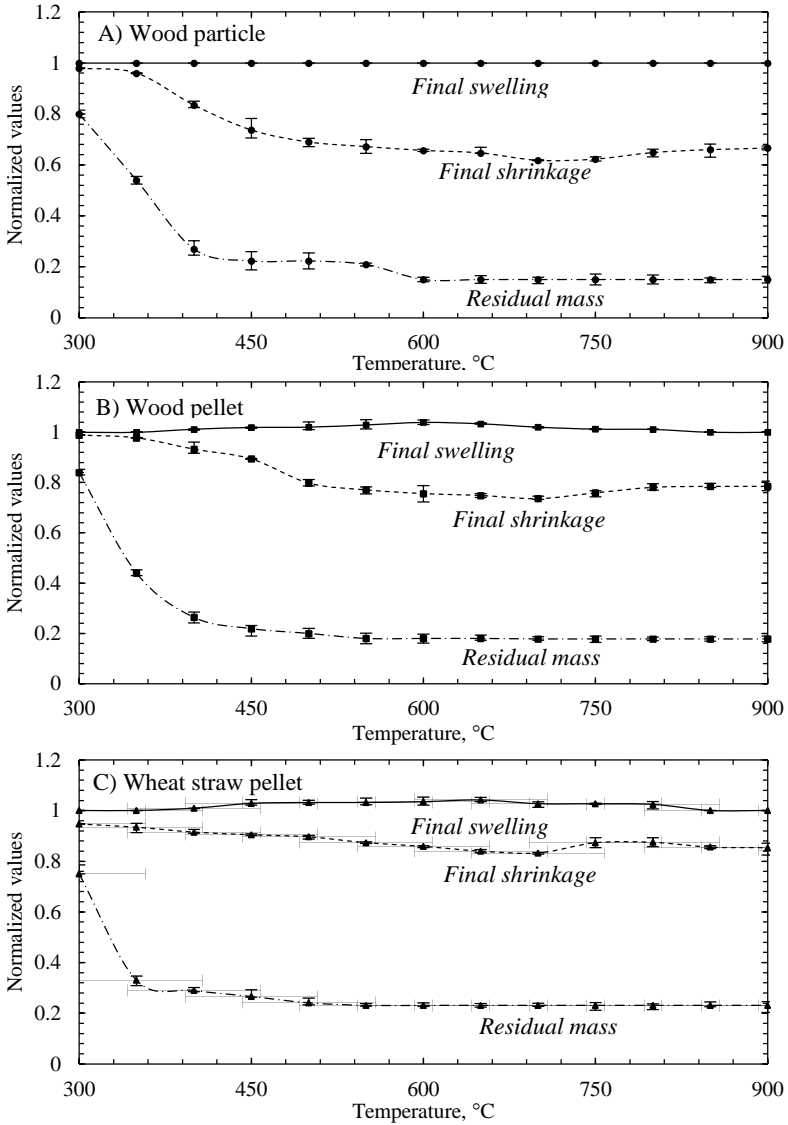
**Fig. 15.** Tendency of biomass thermal deformations at different heating temperature

Observed final radial shrinkage intensifies exponentially from 2.1% to 38.2% for wood particle, from 1.1% to 26.4% for wood pellet and from 5.3% to

16.7% for wheat straw pellet with mass loss increase respectively, from 300 – 750 °C heating temperature (Fig.15).

In the range of 700 – 900 °C heating temperature, it is observed that final shrinkage of biomass samples starts to decrease logistically from 38.2% to 33.4% for wood particle, from 26.4% to 21.5% for wood particle and from 16.7% to 14.7% for wheat straw pellet (Fig. 15). Experimental results reveal that at high heating temperature formed char starts to expand during shrinking process, what influences final shrinkage decrease. According to thermal analysis of used samples, it is considered that during thermal conversion of lignin, char formation takes place and high temperature results in rapid emission of volatiles from formed char. Increased inner pressure stretches the overall char structure, resulting in char swelling.

The mathematical formulas, describing regularities of biomass thermal deformations were created using established data from performed experiments of biomass thermal deformations. As the proceeding of biomass thermal deformations depends on the heating temperature, and can be divided into three phases (swelling, shrinkage and swelling of the residual char), its ongoing processes are described by three mathematical formulas.

The first mathematical equation evaluates pelletized biomass expansion increasing temperature. An assumption that expansion of pellets depends on two competing processes was made for creating formula:

1) The first process when the volatiles inside the pellet expands its diameter. The intensity of the process can be described by the first approach with logistic function:

$$f_{D1}(T_P) = D_0 - \frac{(D_{D1} - D_0)}{\left(1 + \exp\left(-c_{D1} \cdot (T_P - T_{D1})\right)\right)}; \qquad (1)$$

here $D_0$ – initial diameter of biomass particle, AU; $D_{D1}$ – theoretical biomass expansion value, AU; $C_{D1}$ – coefficient describing the expansion intensity depending on the type of biomass; $T_P$ – temperature of pyrolysis, °C; $T_{D1}$ – temperature of pellet expansion saturation, °C.

2) The second process when the condensed tars on pellet surface are decomposed due to high temperature and water vapor and volatiles are more easily emitted from pellet center resulting in final swelling decrease. The intensity of this process is described by a reverse function:

$$f_{D2}(T_P) = D_0 - \frac{(D_0 - D_{D2})}{\left(1 + \exp\left(-c_{D2} \cdot (T_P - T_{D2})\right)\right)}; \qquad (2)$$

here $D_{D2}$ – theoretical biomass particles maximum expansion value, AU; $C_{D2}$ – coefficient describing the intensity of expansion decreasing and depends on the type of biomass; $T_{D2}$ – temperature of pellet expansion saturation, °C.

Since these two processes compete with each other, final expansion of biomass pellets increasing the heating temperature from 300 °C to 900 °C is described by arithmetic average of both functions (1, 2):

$$f_D(T_P) = \frac{1}{2} \cdot \big(f_{D1}(T_P) + f_{D2}(T_P)\big); \qquad (3)$$

Theoretically calculated and experimentally determined final expansion values of wood pellet and straw pellet with increasing pyrolysis temperature are presented graphically in Fig. 16.
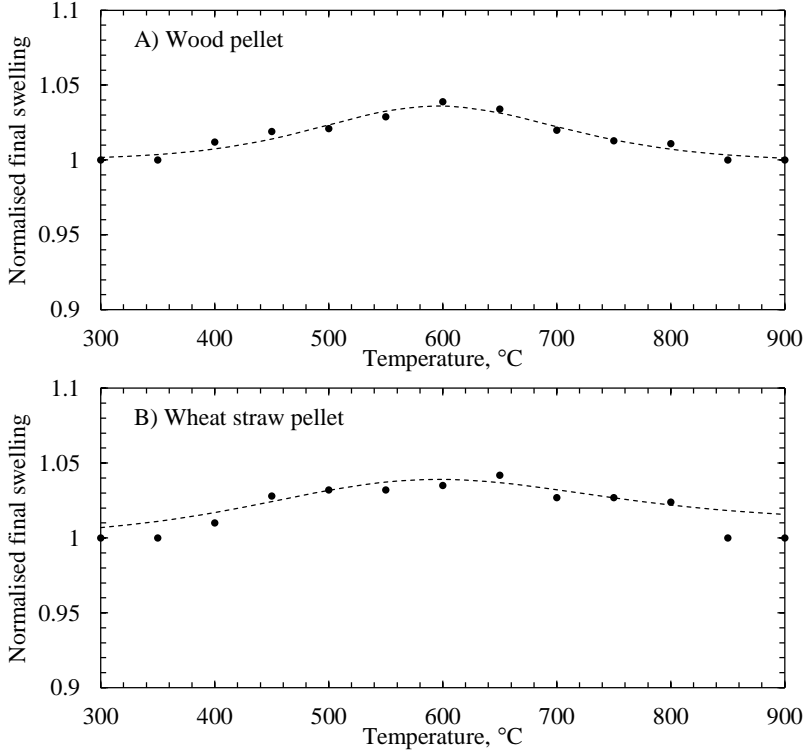


**Fig. 16.** Experimentally determined (dots) and a theoretical calculated (dash line) final expansion values of wood pellet and wheat straw pellet increasing pyrolysis temperature from 300 °C to 900 °C

The assumption is that each moment of time biomass shrinkage progress depends on the current diameter until it reaches the final smallest possible diameter. Therefore, final shrinkage of the diameter can be described as follows:

$$f_{M1}(T_P) = D_0 - (D_0 - D_{M1})/\big(1 + \exp^{(-c_{M1} \cdot (T_p - T_{M1}))}\big); \qquad (4)$$

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here $f_{M1}(T_P)$ – function describing biomass final shrinkage in the range of 300-750 °C pyrolysis temperature, AU .; $D_{M1}$ – the final diameter of the biomass shrinkage not including residual char expansion and depends on the type of biomass, AU; $C_{M1}$ –coefficient describing the intensity of shrinkage and depends on the type of biomass; $T_{M1}$ – pyrolysis temperature at which diameter shrinks to the smallest possible value, °C.

A second assumption is that if the pyrolysis temperature is higher than 700 °C, residual char begins to expand in the end of biomass shrinkage, and the intensity of char expansion at each moment of time depends on the current size of residual char. The higher the temperature, the more intensive char expansion resulting in shrinkage decrease of final biomass diameter. In this case, biomass final diameter dependence on the pyrolysis temperature above 700 °C, including the influence of char swelling, could be described as:

$$f_{M2}(T_P) = D_{M1} + (D_{M1} - D_{M2})/(1 + \exp^{(-c_{M2}\cdot(T_p-T_{M2}))}); \quad (5)$$

here $f_{M2}(T_P)$ – the function describing the final diameter of the shrinkage above 700 °C pyrolysis temperature, AU; $D_{M2}$ - maximum expansion value of residual char at 900 °C temperature, AU; $C_{M2}$ – coefficient describing the intensity decreasing of shrinkage and depends on the type of biomass; $T_{M2}$ – the temperature at which expansion of residual char starts and depends on the type of biomass, °C.

Experimentally determined and theoretically calculated shrinkage values of wood particle wood pellet and wheat straw pellet, increasing pyrolysis temperature from 300 °C to 900 °C, are presented in Fig. 17.
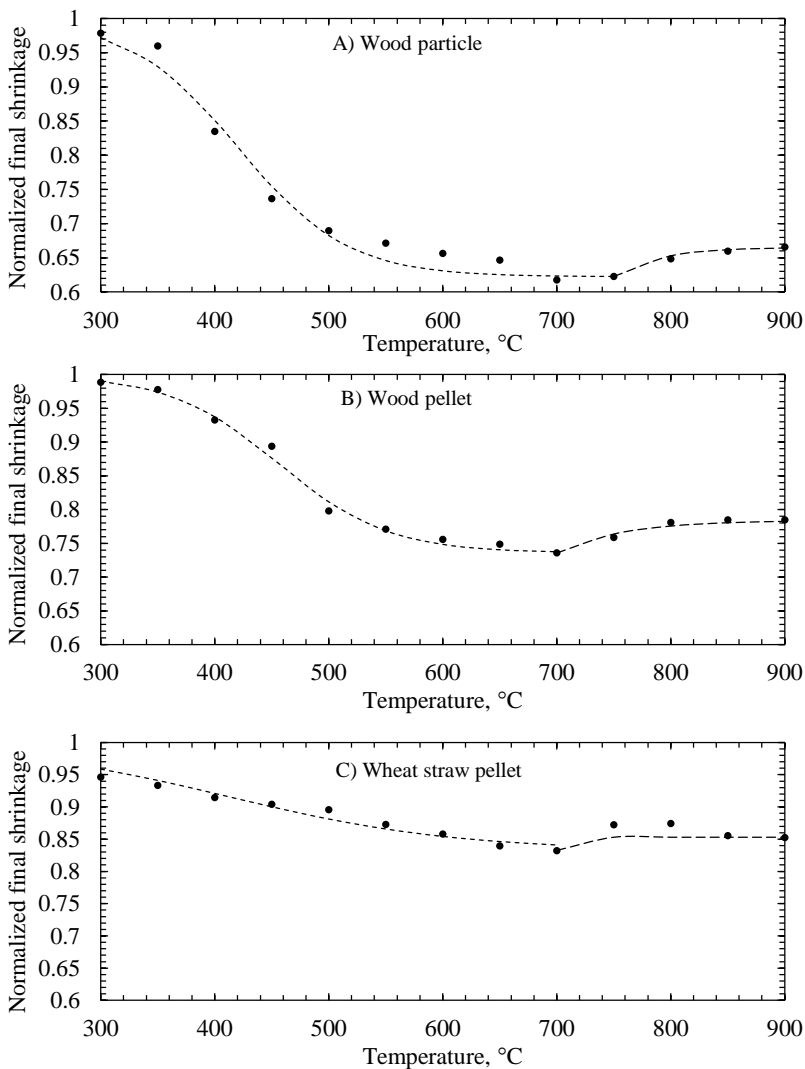
**Fig. 17.** Experimentally determined (dots) and theoretically calculated (dash line) final shrinkage values of wood particle, wood pellet and wheat straw pellet increasing pyrolysis temperature from 300 °C to 900 °C

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**CONCLUSIONS**

Thermal deformations of wood particle, wood pellet and wheat straw pellet were studied during pyrolysis in the range of 300-900 °C. According the obtained results, the main conclusions are the following:

1.  It has been determined that the biomass pellets, contrary to the case of wood particles, expand at the beginning of the pyrolysis process. The pelletized biomass begins to expand at the heating temperature of 400°C as the dehydration process is going on and the diameter increases by approximately 1% of the initial value. As the temperature increases, swelling of the pellets intensifies in the beginning of the pyrolysis process and the largest swelling, amounting to 3.9% of the initial diameter, has been identified at the temperature of 600°C, and the largest swelling of the straw pellet, equal to 4.2% of the initial diameter, was identified at the temperature of 650°C. At the temperatures higher than 650°C, swelling of the pellets diminishes and starting from the temperatures of 850°C, the swelling phenomenon disappears.

2.  It has been determined that at the start of the pyrolysis process, the pelletized biomass expands due to obstructed emission of water vapour and volatile species:

    ✓  At low temperature (from 400°C to 600–650 °C), evaporated tars condense on the pellet surface which obstruct evaporation of water vapour and volatile species, therefore, the volatile species create internal pressure that expands the pellet.

    ✓  As the heating temperature increases to over 600 °C, due to more intense decomposition of evaporated and condensed tar, water vapour and volatile species evaporate easier from the pelletized biomass, therefore, the pressure inside the pellet created by volatile species decrease and the swelling phenomenon diminishes.

    ✓  After the temperature of 850°C is exceeded, swelling of the pelletized biomass does not occur anymore because of intense thermochemical processes during which the water vapour and volatile species evaporate without producing the internal pressure inside the pellets.

3.  It has been determined that expanded biomass pellets shrink after the hemicellulose starts to break down, i.e., as the emission of volatiles intensifies. Shrinkage of a wood particle, contrary to the case of the biomass pellets, starts during the dehydration process.

    ✓  As the heating temperature increases from 300 °C to 700 °C, the wood particle shrinks by 2.1% to 38.2% of its initial diameter, wood pellets and straw pellets shrink by 1.1% to 26.4% and by 5.3% to 16.7% of the initial

diameter, respectively. As the heating temperature increases from 700 °C to 900 °C, the biomass samples shrink less than at the temperature of 700 °C, i.e., the difference between the initial and residual diameters decreases. The final shrinkage of a wood particle decreases from 38.2% to 34% of the initial diameter, the final shrinkage of a wood pellet decreases from 26.4 % to 21.5 % of the initial diameter, and that of the straw pellet – from 16.7% to 14.7% of the initial diameter.

✓ An assumption was taken that the biomass samples shrink less, i.e., the final shrinkage decreases, because expansion of char formed at high temperature. This effect is induced by high temperature influencing sudden overheat of the produced char and emission of volatiles. The volatiles damage the structure of the produced char as they evaporate and expand the sample. Besides, it has been determined that during expansion of the produced char, during which the difference of the sample diameter decreases, the density of the samples decreases as well, because the density of the emitted volatile species does not change as the pyrolysis temperature increases from 700 °C to 900 °C.

# PUBLICATIONS RELATED TO THE DISSERTATION

### Articles in journals from Thomson Reuters "Web of Knowledge" list

1  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Experimental investigation of wood pellet swelling and shrinking during pyrolysis. *Fuel*. ISSN 0016-2361. 2015. Vol. 142, p. 145–151.

### Articles in journals, referred in international scientific databases

1  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N., GARŠVINSKAITĖ L., MISIULIS E. Experimental and theoretical investigation of wood pellet shrinkage during pyrolysis. *Energetika*. ISSN 0235-7208. 2014. T. 60, Nr. 1, p. 1-11.

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1.  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Numerical models of wood pellet shrinking on pyrolysis. 10th annual international conference of young scientists on energy issues (10 CYSENI anniversary). Kaunas, Lithuania, May 29-31, 2013. Kaunas: LEI, 2013. ISSN 1822-7554, p. 543-547.

2.  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Experimental investigation of wood pellet shrinking during pyrolysis. 11th international conference of young scientists on energy issues (CYSENI 2014), Kaunas, Lithuania, May 29-30, 2014. Kaunas: LEI, 2014. ISSN 1822-7554, p. 373-378.

3.  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Experimental research on swelling and shrinking of wood and straw pellets during pyrolysis. 12th Annual international conference of young scientists on energy issues (CYSENI 2015), Kaunas, Lithuania, May 27–28, 2015. Kaunas: LEI, 2015. ISSN 1822-7554. p. 98-103.

4.  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. The cause and mechanism of pelletized biomass swelling uppon pyrolysis. 13th Annual international conference of young scientists on energy issues (CYSENI 2016), Kaunas, Lithuania, May 26–27, 2016. Kaunas: LEI, 2016. ISSN 1822-7554. p. 260-265

### Publications at the national conferences

1.  PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Medžio granulės pokyčių tyrimas pirolizės metu. Šilumos energetika ir technologijos-2014: konferencijos pranešimų medžiaga, Kauno technologijos universitetas, 2014 m. sausio 31. Kaunas: Technologija, 2014. ISSN 2335-2485, p. 31-36.

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1. PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Numerical models of wood pellet shrinking on pyrolysis. 10th annual international conference of young scientists on energy issues (10 CYSENI anniversary). Kaunas, Lithuania, May 29-31, 2013. Kaunas: LEI, 2013. ISSN 1822-7554, p. 543-547.
2. PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Comparison of experimental measurements and numerical model for wood pellet shrinking during pyrolysis. International Bioenergy conference 2014, Manchester central convention Complex, Manchester, UK 11th-13th March, 2014.
3. PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Experimental investigation of wood pellet shrinking during pyrolysis. 11th international conference of young scientists on energy issues (CYSENI 2014), Kaunas, Lithuania, May 29-30, 2014. Kaunas: LEI, 2014. ISSN 1822-7554, p. 373-378.
4. PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. Experimental research on swelling and shrinking of wood and straw pellets during pyrolysis. 12th Annual international conference of young scientists on energy issues (CYSENI 2015), Kaunas, Lithuania, May 27–28, 2015. Kaunas: LEI, 2015. ISSN 1822-7554. p. 98-103.
5. PAULAUSKAS R., DŽIUGYS A., STRIŪGAS N. The cause and mechanism of pelletized biomass swelling uppon pyrolysis. 13th Annual international conference of young scientists on energy issues (CYSENI 2016), Kaunas, Lithuania, May 26–27, 2016. Kaunas: LEI, 2016. ISSN 1822-7554. p. 260-265

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# REZIUMĖ

Dėl griežtėjančių aplinkosaugos reikalavimų, atsinaujinantys kuro ištekliai vis plačiau naudojami elektros ir šilumos gamyboje. Išaugus biokuro poreikiui, ieškoma būdų, kaip panaudoti ir prastos kokybės atsinaujinančius kuro išteklius (miško paklotas, spygliai, vištų kraikas ir t.t.). Vienas būdų – granuliuotą biomasę naudoti dujinimo procesui, kurio metu kietos frakcijos kuras paverčiamas dujomis. Tačiau dujinimo procese naudojant granuliuotą kurą, susidaro sąlygos, kurių metu granulės, judėdamos reaktoriuje iš pirolizės zonos į dujinimo zoną, sulimpa dėl atsirandančių granuliuotos biomasės deformacijų ir sustabdo tolimesnę proceso eigą.

Atlikus pasaulinės mokslinės-techninės literatūros analizę pastebėta, kad moksliniuose darbuose plačiausiai analizuojamos įvairios medienos dalelių terminės deformacijos ir tik keliuose nagrinėti biomasės granulių (medžio, šiaudų ir perdirbtų atliekų) pokyčiai aukštoje temperatūroje. Šiuose darbuose nurodoma, kad perdirbtų atliekų ir šiaudų granulės plečiasi aukštoje temperatūroje, bei pateikiamos perdirbtų atliekų granulių išsiplėtimo priežastys dėl jose esančio plastiko. Tačiau perdirbtų atliekų granulių išsiplėtimo priežastys nepaaiškina šiaudų ar medžio granulių, kurių biocheminė sudėtis ir struktūra skiriasi, išsiplėtimo. Be to, apžvelgtuose darbuose nėra apibrėžta, kurioje temperatūroje prasideda granuliuotos biomasės išsiplėtimas ir kurioje temperatūroje šis reiškinys nebevyksta. Todėl būtina ištirti biomasės termines deformacijas, atliekant medžio dalelės, medžio granulės ir šiaudų granulės pirolizės tyrimą, nustatyti granuliuotos biomasės terminių deformacijų priežastis bei biomasės terminių deformacijų dėsningumus ir jų priklausomumą nuo temperatūros.

Darbo tikslas - granuliuotos biomasės terminio skaidymo tyrimas, siekiant nustatyti terminių deformacijų dėsningumus, paaiškinti jų priežastis bei matematiškai aprašyti biomasės terminės deformacijos priklausomumą nuo temperatūros.

Darbe išsamiai apibūdintas pirolizės procesas ir atskleistas granuliuotos biomasės terminių deformacijų mechanizmas ištyrus granuliuotos biomasės (medžio granulės ir šiaudų granulės) išsiplėtimą ir susitraukimą terminio skaidymo metu, nustačius biomasės terminių deformacijų dėsningumus ir pateikus lygtis, aprašančias biomasės termines deformacijas.

Pagrindiniai darbe gauti rezultatai, atlikus biomasės bandinių terminės deformacijos tyrimus pirolizės metu 300–900 °C temperatūroje, parodė, kad biomasės granulės, kitaip nei medžio dalelės, pirolizės pradžioje plečiasi. Granuliuota biomasė pradeda plėstis 400 °C kaitinimo temperatūroje vykstant dehidratacijos procesui ir skersmuo padidėja apie 1 % pradinio skersmens. Kylant temperatūrai, pirolizės pradžioje granulių išsiplėtimas intensyvėja ir didžiausias medžio granulės išsiplėtimas, siekiantis 3,9 % pradinio skersmens, nustatytas 600 °C temperatūroje, o didžiausias šiaudų granulės išsiplėtimas (4,2 % pradinio

skersmens) – 650 °C temperatūroje. Esant aukštesnėms temperatūroms nei 650 °C granulių išsiplėtimas slopsta ir nuo 850 °C temperatūros išsiplėtimo reiškinys išnyksta. Ištirta, kad pirolizės pradžioje granuliuota biomasė išsiplečia dėl apsunkinto vandens garų ir lakiųjų medžiagų išsiskyrimo, nes žemoje temperatūroje (nuo 400 iki 600–650 °C) granulės paviršiuje kondensuojasi išsiskyrusios dervos, kurios apsunkina vandens garų ir lakiųjų medžiagų išsiskyrimą, dėl to lakiosios medžiagos sukelia vidinį slėgį, kuris išplečia granulę. Kylant kaitinimo temperatūrai per 600 °C, dėl spartesnio išsiskyrusių ir susikondensavusių dervų skaidymo, vandens garai ir lakiosios medžiagos lengviau išsiskiria iš granuliuotos biomasės, todėl lakiųjų medžiagų sukeliamas slėgis granulės viduje mažėja ir išsiplėtimo reiškinys slopsta. Aukštesnėje nei 850 °C temperatūroje granuliuota biomasė nebesiplečia dėl intensyvių termocheminių procesų, kurių metu vandens garai ir lakiosios medžiagos išsiskiria nesukeldamos vidinio slėgio granulių viduje. Nustatyta, kad išsiplėtusios biomasės granulės traukiasi prasidėjus hemiceliuliozės skilimui, t. y. intensyvėjant lakiųjų medžiagų išsiskyrimui. Medžio dalelės susitraukimas, kitaip nei biomasės granulių, prasideda dehidratacijos proceso metu. Kylant kaitinimo temperatūrai nuo 300 iki 700 °C, medžio dalelė susitraukia nuo 2,1 iki 38,2 % pradinio skersmens, medžio granulės ir šiaudų granulės susitraukia atitinkamai nuo 1,1 iki 26,4 % pradinio skersmens ir nuo 5,3 iki 16,7 % pradinio skersmens. Kylant kaitinimo temperatūrai nuo 700 iki 900 °C, biomasės bandiniai susitraukia mažiau nei 700 °C temperatūroje, t. y. skersmens pokytis tarp pradinio ir likutinio dydžio mažėja. Skersmens pokytis sumažėja: medžio dalelės – nuo 38,2 iki 34 % pradinio skersmens, medžio granulės – nuo 26,4 iki 21,5 % pradinio skersmens ir šiaudų granulės – nuo 16,7 iki 14,7 % pradinio skersmens. Iškelta prielaida, kad biomasės bandiniai susitraukia mažiau, t. y. skersmens pokyčio mažėjimas, aukštoje temperatūroje dėl susiformavusios anglies išsiplėtimo. Šį efektą sukelia aukšta temperatūra veikdama susiformavusios anglies staigų perkaitimą ir lakiųjų medžiagų išsiskyrimą. Išsiskirdamos lakiosios medžiagos pažeidžia susiformavusios anglies struktūrą ir išplečia bandinį. Be to, nustatyta, kad susiformavusiai angliai plečiantis mažėja ir bandinių tankis, nes išsiskyrusių lakiųjų medžiagų kiekis nekinta, didėjant pirolizės temperatūrai nuo 700 iki 900 °C.