



UNIVERSITY OF MISKOLC
FACULTY OF EARTH SCIENCE & ENGINEERING
Institute of Raw Material Preparation and Environmental Processing



INVESTIGATION OF PRESSURE AGGLOMERATION PROCESS OF BIOMASS

Theses of Doctoral (PhD) Dissertation

Author

Trinh Van Quyen

Scientific supervisor

Dr.habil. Barnabás Csőke
Professor

Co-Scientific supervisor

Dr. Sándor Nagy
Associate Professor

MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCES

Head of the Doctoral School: Prof. Dr.habil. Péter Szűcs

1. Aims of the dissertation, scientific preliminaries

The use of biomass is a good option for domestic heating systems and power plants to reduce net CO₂ emissions. Agglomeration of biomass such as briquetting, extrusion, tableting, pelletizing can increase bulk density, improves storability, reduces transportation costs, makes easy the handling and increase the quality of products. The globally installed pellet production capacity for 2011 was estimated to be about 30 million tonnes. All studies suggest strong growth for both the European and North American pellet markets. The Finnish Pöyry Industry consulting company has predicted growth in global wood pellet production capacity to 46 million tonnes by 2020 (Pöyry 2011) and 65 million tonnes by 2025 (Strauss 2017).

The parameters of biomass agglomerate production are especially important in aspects related to product quality and economics. On the one hand, the reduction of moisture content and increasing temperature usually results in better quality agglomerates; it is possible to achieve higher density and strength. On the other hand, moisture content reduction (drying) and increasing temperature have a large energy demand. To find the optimal production parameters, the exact relation between moisture content, temperature and briquetability (applied pressure agglomerate density) should be known. The grinding of raw materials also demands a large amount of energy. Optimal particle size should be determined for economical production and for good agglomerate quality. For the purpose of compacting very fine powders without any binding material an experimented pilot-scale technological set up with press-roll have been built with systematical tests have been carried out by Csöke and Faitli (2003).

Pelletizing is currently one of the most frequently used methods for producing agglomerates, using either a ring die or a flat die pelletizer. This process can increase bulk density, reduce storage and transportation costs and makes easy handling of biomass. Briquettes and pellets are often in the stock with a long time before use, so their biological stability is important. While which are increasing in the case of higher density and strength. Raw materials for fuel pellets production can be different types of biomasses from various resources. Also, composition and structural properties of these materials are diverse. This has the consequence that different types of biomass require different processing conditions such as press channel length, moisture content, particle size and temperature. Nowadays, the process optimization is mainly based on expensive and time-consuming “trial and error” experiments and personal experience (Holm et al. 2011). Pressure is an important process parameter that greatly influences the density of biomass pellets.

Previous research in the press channel model

The press channel model in experimental piston press has recently been studied by many research groups, such as Adapa et al. 2002; Mani et al. 2004; Holm et al. 2006; Nielsen et al. 2009a; Zafari et al. 2012. The studies (Adapa et al. 2002; and Mani et al. 2004) have focused on the compaction behavior of different biomass types by conducting a pelletizing test in a single pellet unit and fitting the obtained data to the different mathematical model found in the literature. The models having the best fit to the experimental data were identified and used to explain the compaction mechanism of biomass. According to Holm et al. 2006, the major force acting on the biomass in the press channel is the friction force between the press channel wall and the biomass. Assuming that the biomass is an orthotropic material, where the fibers align perpendicular to the direction of the press channel, and that the biomass also is an elastic material, the pressure build-up in the press channel of a pellet mill, can be described as a function of the friction coefficient, the Poisson ratio, press channel length and its radius.

Nielsen et al. 2009a presented tools and methods that separate the pelletizing process into compression, flow and friction components to measure the importance of raw material properties for the energy requirements of pelletizing, and pellet strength. Experimental materials were sawdust from European beech and Scots pine. Zafari et al. 2012 introduced in their research work about the experimental design to evaluate the effect of moisture content, the speed of piston, die length, and particle size on pellet density, and obtained maximum density. Experimental material was composted municipal solid waste, press channel had 10 mm diameter, a total length of the active part was 100 mm. Statistical analyses confirmed that the moisture content, speed of piston and particle size significantly affected the pellet density.

Previous research in the applied pressure and backup pressure

Maximum applied pressures ranged from 50 MPa (Odogherty and Wheeler 1984) to 600 MPa (Stelte et al. 2011b). The pressure typically used in most studies was above 50 MPa in the case of open form (Adapa et al. 2009; Mani et al. 2006; Stelte et al. 2011b). Holm et al. 2007 measured the backup pressure needed to press pellets of different lengths out of the press channel, it is shown that the pelletizing pressure does increase exponentially as a function of the pellet length. The backup pressures were shown to be dependent on biomass species for all tested pellet lengths. Stelte 2011b showed that the pelletizing pressure increases exponentially with the pellet length. An increasing pelletizing pressure is resulting in an increasing pellet density.

During continuous pelletization of biomass, a backup pressure is needed to initiate the process of pelletization. The backup pressure is created by the buildup of material in the press channel, which sets the requirement for a pressure (pre-stressing pressure) to overcome the friction within the channels. The initial pelletization pressure depends on die dimensions such as die hole, die length, friction coefficient and pre-stressing pressure (Holm et al. 2006; Tumuluru et al. 2010).

Previous research in the binding by lignocellulose

Kaliyan and Morey (2010) described the creation of “solid bridge” type bonding between particles in corn stover and switchgrass pellets. According to their results, the potential natural binding component in these materials are water-soluble carbohydrates, lignin, protein, starch and fat.

Stelte et al. 2011 studied the bonding and failure mechanisms of pellets from beech, spruce and straw. In the case of wood pellets, they signalled Van der Waals forces and hydrogen bonds being mainly responsible for agglomeration; solid bridge formation was observed in beech pellets but not in spruce one. The presence of waxes on the straw surface produced a weak waxy boundary layer, resulting in a lower strength. They found that temperature played an important role in the bonding mechanisms, obtaining in every case higher compression strength for pellets produced at higher temperatures.

Biomass raw materials with high percentages of lignin and low extractives contents reached higher durability values. Extractive has a double effect in the pelletization process: they form a weak boundary layer preventing particles from bonding strongly together and they also produce a lubricating effect resulting in a lower friction inside the die channels. Lignin seems to favor the agglomeration of the biomass particles due to its thermoplastic behavior (Castellano et al. 2015).

The aims and novelty of research

The investigated materials are diverse with different kinds of biomass types, which have viscoelastic material properties. Their behavior against mechanical stress are more complex and diverse even between various biomasses than other non-viscoelastic materials. Composition (cellulose, lignin and starch content) and material structure of them are quite various. To demonstrate similar and different behavior according to the above mentioned features, the experimental test is very important, which requires proper experimental procedure and equipment as well as evaluation method.

The development of equipment (single pelletizer units include comparison of laboratory results (SPU1) and measuring backup pressure distribution on the wall of a flat die pelletizer (SPU2)) are one of the main aims of this scientific research work. It was also an objective to use a pilot test method that directly provides data for the process engineering design of biomass pelletization in relation to biomass with granular properties and phase composition. Introduction of examination and evaluation methods (the effects of moisture content, temperature, particle size and main components on biomass agglomeration process) are generally suitable for investigation of behavior of biomass during pressure agglomeration. The equipment of this experimental test was a larger (25 mm diameter) heated pressure channel.

Although many researchers worked on the effect of compaction pressure during the pelletizing process, only few of them, such as Holm et al. 2007 and Stelte 2011b discussed the backup pressure in the press channel. Hence the objective of the present research is to supplement the measurement method of backup pressure distribution during fuel pellet production from biomass. To achieve this, the backup pressure values along the direction of the applied force was measured at three different positions along the active part length.

There are studies regarding the compaction equation between density and applied pressure, however no equation was found in the literature that includes the moisture content as a parameter of biomass raw materials. The objective of the current work is also to supplement the original Johanson equation with the parameter moisture content. I also tested the spring-back ratio (SBR) and biomass components because they are related to the density and quality of the tablets.

2. Experimental equipment and methods of research work

2.1. Hydraulic piston press at the University of Miskolc

The hydraulic piston press (Figure 1) was designed by the University of Miskolc. The press is supported by a pump motor unit with a pressure limiter and a heatable load cell (20-140°C). The maximum force is 200 kN and the maximum velocity of the piston is 30 mm/s. The measuring of the piston position is done with an incremental encoder.



Figure 1. Hydraulic piston press

2.2. Flat die pelletizer at the University of Miskolc

Flat die pelletizer (Figure 2): the system contains a press, mixing container, conditioning screw and a steam generator (Theobald TJ-Extra II, 19 kg/h steam). The capacity of the pelletizer is 50...100 kg/h depending on feed, during my experiment it was 60 kg/h. The diameter of the flat die is 200 mm, hole length is 28 mm and diameter is 8 mm.

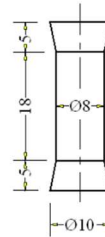


Figure 2. Flat die pelletizer (left); hole length (right)

2.3. Materials

Biomasses (including spelt chaff raw material, ground post-agglomerated spelt chaff, beech sawdust, *Acacia mangium* sawdust one-month seasoned wood, *Acacia mangium* saw dust six-months seasoned wood and rice straw) were used for my experiments.

Spelt chaff raw material originated from Szendrő, Hungary (Natur Gold Farms Ltd.). It was dried and then ground using a cutting mill (Retsch SM2000) in one step (screen size 2 mm).

Ground post-agglomerated spelt chaff (GPA-spelt chaff) was produced from agglomeration spelt chaff raw material at a temperature of 100°C (particle size < 1.6 mm) and then ground with screen size 1 mm.

Beech sawdust originated from Miskolc, Hungary (Borsodwood Ltd.). It was dried and then ground using a cutting mill (Retsch SM2000) on two steps (screen sizes: 2 mm and 1 mm).

Acacia mangium (*A.mangium*) originated from Quang Ninh province, Vietnam. It was seasoned to reduce the moisture content. Half of it was seasoned for 1 month by being covered outdoors, while the other half was seasoned for 6 months.

2.4. Methods of research work

The spelt chaff pellets were produced by flat die pelletizer, and the results were compared with those of single pelletizer unit 1. During this modelling, the parameters could be changed easily. Single pelletizer unit 2 was developed and used for three positions of backup pressure measurement in the press channel.

Biomasses (including six types of materials above) were compressed in load cell by hydraulic piston press with 25 mm diameter and compressibility was determined.

Effects of independent variables, including temperature (20 - 120°C), pressure (50 - 300 MPa), moisture content and particle size of various raw materials (1 mm and 2 mm) with different cellulose, lignin, starch content and morphology were investigated for pellet properties, pellet densities, strength tablets and expansion of tablets (spring-back ratio).

The particle size distribution is calculated based on mass distribution by sieving. The moisture content is defined as the weight loss of mass that occurs as the material is heated (at 105°C) per initial mass of material (wet basic).

The quality of tablets can be described easily by their density. The diameters and heights of the tablets product were measured by Vernier caliper (a tablet can be extended after agglomeration). The mass was measured and density was calculated for each test.

The springback ratio (SBR) of a tablet can be determined by

$$\text{SBR} = \frac{H_t - H_{tp}}{H_{tp}} 100 \%$$

where H_t is the height of the produced tablet, H_{tp} is a minimum height of the tablet under pressure. The minimum height of tablets under pressure was measured by the incremental distance measurement method.

The cross-sectional surfaces of tablets were investigated with an optical microscope Zeiss AXIO Imager.M2m. The determination of tablet strength was carried out by the well-known falling test method. Tablets were released by freefall from a height of 2 m onto a concrete floor repeatedly until they broke. The falling number is the number of falls the sample survived undamaged. In each experiment three tablets were tested, the results were calculated from the average of three independent tests.

3. Scientific results, theses

The new scientific results of my dissertation can be summarized as follows:

THESIS 1. Development of single pelletizer unit 1 (SPU 1)

I have developed and implemented a pelletizing channel device (SPU 1) that is suitable for modeling the pelletizing process in the pressure channels of the flat pelletizer in the case of biomass with different properties in relation to the applied pressure and temperature.

THESIS 1.a.) The design of new press channel and piston of single pelletizer unit 1

The single pelletizer unit 1 design showed in Figure 3. Length of the active part was 55 mm, the diameter of the hole was 8 mm. A piston (diameter: 8 mm, length: 15 mm limited by a cylinder) can force the raw material into the chamber. Above the piston, a force meter was built in to measure the actual force.

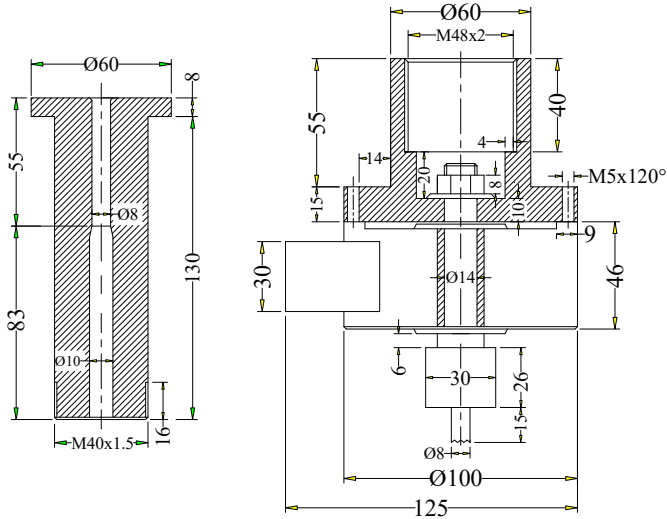


Figure 3. Design of new press channel (left) and piston (right) of single pelletizer unit 1

THESIS 1.b.) Modelling of pellets production by single pelletizer unit 1

Modelling with single pelletizer unit 1 at the different behavior of different biomasses and the importance of experimental studies are shown in Figure 4. It is observed that at the same production conditions, pellets made from GPA-spelt chaff had a higher density than those made from spelt chaff raw material and have a maximum value at 20 wt.% moisture content. The increasing temperature has a higher density of pellets values.

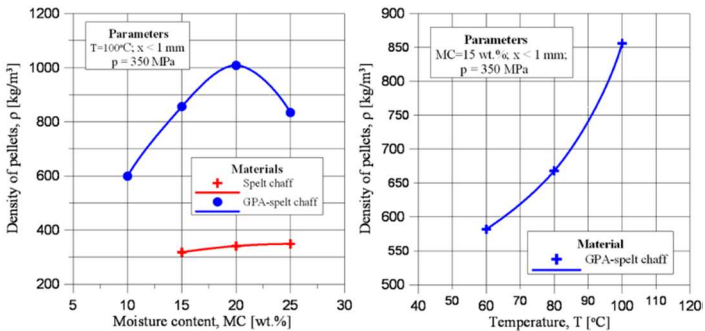


Figure 4. Relationship between density of pellets and moisture content (left) and temperature (right)

THESIS 2. The new design press channel of single pelletizer unit 2 (SPU 2)

I have designed and built a pelleting channel (SPU 2), which is divided into segments, one of which is a measuring segment, and I have developed a measurement method suitable for modeling pelletization in flat pelletizer pressure channels for biomasses with different properties, revealing the correlation between pellet density and channel wall pressure at different depths and temperatures.

THESIS 2.a) The single pelletizer unit 2 design (Figure 5)

The length of the active part was 55 mm, including three parts made from carbon steel, one part made from polyoxymethylene (POM), the length of each part was 13.75 mm and the diameter of the hole was 8 mm. Above the single pelletizer unit 2 a pressure transducer was built in to measure the backup pressure (solvent between membrane and pressure transducer: glycerin and alcohol). A piston (diameter: 8 mm, length: 15 mm limited by a cylinder (height: 26 mm)) can force the raw material to the chamber.

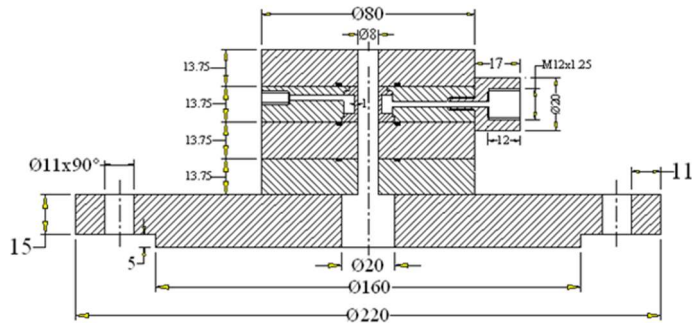


Figure 5. New design press channel of single pelletizer unit 2

THESIS 2.b) The test methodology measuring of backup pressure distribution and pellet density

I developed a test methodology for the 2 pelletizing units as follows: the backup pressure distributions can be determined in the chamber by changing the pressure measurement position. Each measurement was made until the process of agglomeration stopped. The quality of pellets can be described easily by their density. The diameters and heights of the pellet sections were measured by Vernier caliper. The mass was measured and density was calculated for each test.

The relationship between the density of pellets and backup pressure - in different position of backup pressure measurement disc at 20°C, 60°C temperature are shown in Table 1.

Table 1. Relationship between density of pellets and backup pressure

Temperature [°C]		20°C		60°C	
Density/backup pressure		ρ_1 [kg/m ³]	p_B [bar]	ρ_1 [kg/m ³]	p_B [bar]
Position of backup pressure measurement disc	2	1080	21.1	1093	23.7
	3	959	9.9	987	14.9
	4	631	8.4	773	9.6

THESIS 2.c) Backup pressure and pellet density distribution

Systematic measurement with SPU 2 has shown that wall pressure (backup pressure) and pellet density decrease in the channel in the direction of pellet progression (Figure 6). The quality of the pellet leaving the pressure channel is determined by the pressure in the last section (segment) of the channel.

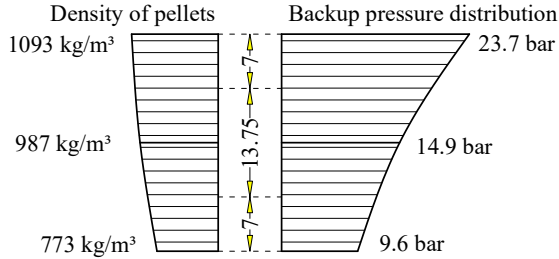


Figure 6. Illustration density of pellets and backup pressure distribution values along the active part at temperature of 60°C

THESIS 2.d) Determination of compressibility of GPA- spelt chaff

Tablets were produced to determine the compressibility of the raw material. The hydraulic piston press (25 mm diameter) was used. The feed material was produced from GPA- spelt chaff. Figure 7 shows the relationship between applied pressure and density, described by the linear function. According to the equation in Figure 7, using the average pellet density, the applied pressure of flat die pelletizer can be calculated.

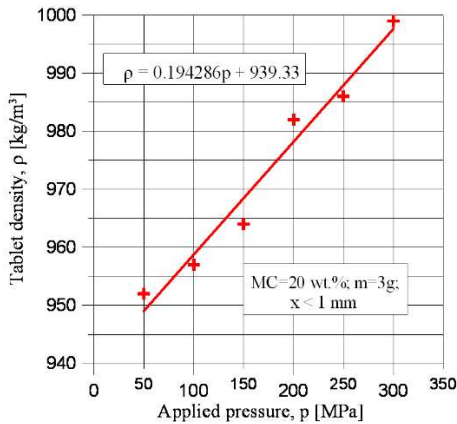


Figure 7. Applied pressure-tablet density diagram for GPA- spelt chaff

THESIS 3. Modelling of volume reduction and process optimization in closed form agglomeration equipment

I have further developed the method of testing and evaluating tablet pelletization. Thereby, the relationship between the dispersive properties, moisture content of biomass and the properties of the pellets produced were determined at different pressures and temperatures, as a basis for the pelleting process engineering design.

The most important results of this method and its application are:

THESIS 3.1 Effect of moisture content and particle size on biomass agglomeration

The Johanson relation expresses opposition to a granular set against volumetric change. The most common is the relationship between specific force and specific volume, i.e. pressure and agglomerate density. The following measurements demonstrate the effect of particle size and moisture content on the density of the agglomerate under increasing load, for a given size, the modified Johanson, which also contains moisture content, is directly suited to describe the process.

THESIS 3.1.a) Tablet density and new equation based on the original Johanson equation

Figure 8 (left) shows the pressure density values and the fitted Johanson curves in the case of beech sawdust with $x < 1$ mm. Results for $x < 2$ mm are introduced in Figure 8 (right).

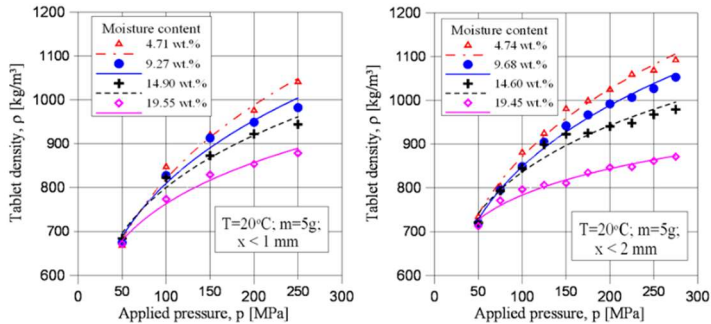


Figure 8. Compressibility data for beech sawdust with different moisture contents; (left) particle size < 1 mm; (right) particle size < 2 mm

Tablets compressed at lower pressure have lower density. If pressure and particle size are kept constant, an increasing moisture content resulted in lower tablet density. Tablets made from particle size < 2 mm had higher density than those made from particle size < 1 mm, at constant pressure, temperature and similar moisture content.

To describe the compressibility of beech sawdust, the Johanson equation was used. As the original equation for describing compressibility, it is universal. It is possible to insert other parameters in it, such as moisture content.

I have improved the Johanson equation. The compression can be described by an equation based on the original Johanson equation and containing moisture content as a parameter.

Johanson's equation: $\rho = ap^{1/k} = ap^b$

The new equation for pressure density function for a given particle size:

$$\rho = (a_1 \cdot c_w + a_1) p^{(-b \cdot c_w + b_2)}$$

c_w is the moisture content [wt.%]

THESIS 3.1.b) Springback ratio of tablets

Figure 9 shows the relationship between applied pressure and SBR in the case of different moisture contents of beech. This relationship can be described by the following function: $SBR = c$ the constants c and b are corresponding to each moisture content. Tablets made from the raw material with larger moisture content had larger SBR at the same pressure, temperature and particle size.

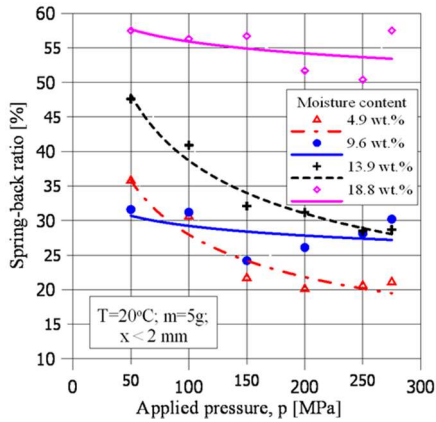


Figure 9. Relationship between spring-back ratio and pressure for different moisture contents of beech sawdust ($x < 2$ mm)

THESIS 3.1.c) Tablet strength

Falling number values in the case of tables made from beech sawdust are shown in Figure 10 as a function of moisture content at different pressures for both $x < 1$ mm and $x < 2$ mm raw materials. Increasing moisture content resulted in lower tablet strength at the same pressure and with same particle size. Tablets made from larger particle size have higher strength than tablets made from smaller particle size if moisture content and pressure are constant. The reason for this may be that the larger particles size connects together better than small particles size with the same experimental conditions.

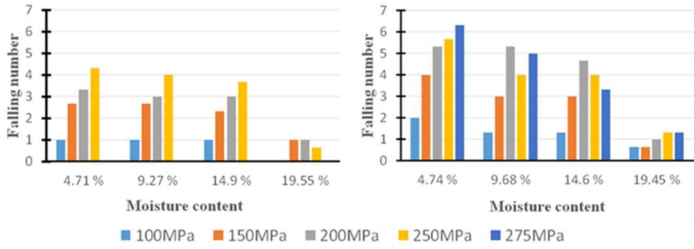


Figure 10. Falling number values in the case of tables made from beech sawdust with different moisture contents and pressures; (left) $x < 1$ mm; (right) $x < 2$ mm

THESIS 3.2. Temperature, particle size and seasoning effects on biomass agglomeration

THESIS 3.2.a) Tablet density

Figure 11 (a) and (b) show the pressure-density values and the fitted Johanson curves in the case of *A. mangium* one month seasoned wood ($x < 0.8$ mm and $x < 1.6$ mm) raw material. Tablets compressed at lower pressure have lower densities. If pressure and particle size are kept constant, an increasing temperature resulted in higher tablet density. Tablets made from material particle size < 1.6 mm had a higher density than tablets made from particle size < 0.8 mm, at constant pressure, temperature and moisture content.

Results for particle size < 1.6 mm, *A. mangium* six months seasoned wood are introduced in Figure 11 (c). If the pressure is kept constant, an increasing temperature resulted in higher tablet density.

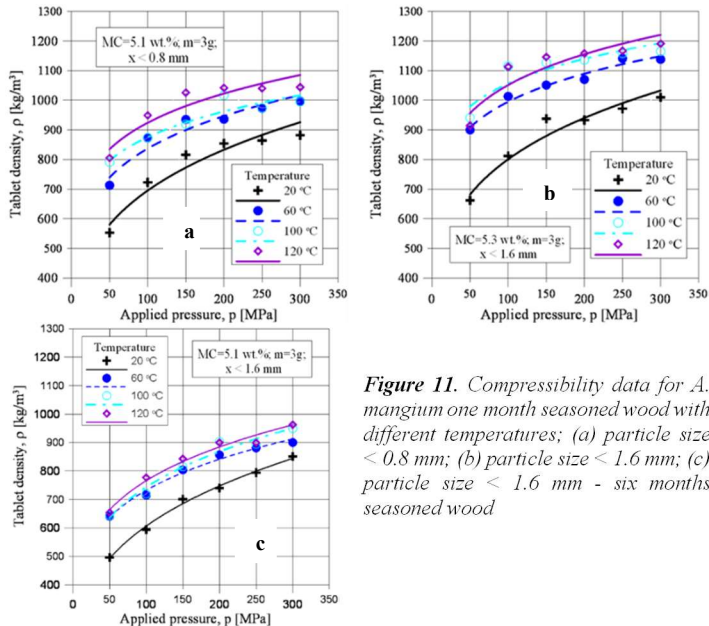


Figure 11. Compressibility data for *A. mangium* one month seasoned wood with different temperatures; (a) particle size < 0.8 mm; (b) particle size < 1.6 mm; (c) particle size < 1.6 mm - six months seasoned wood

THESIS 3.2.b) Tablet strength

Falling number values in the case of $x < 0.8$ mm and $x < 1.6$ mm raw materials (one month seasoned wood) are shown in Figure 12 (a) and (b) as a function of temperature on different pressures. Increasing temperature resulted in higher tablet strength at the same pressure and particle size. Tablets made from raw materials $x < 1.6$ mm had a higher strength than those made from $x < 0.8$ mm biomass, if moisture content and pressure are kept constant.

Falling number values in the case of *A.mangium* six months seasoned wood with the particle size < 1.6 mm are shown in Figure 12 (c). Increasing temperature resulted in higher tablet strength at the same pressure and particle size. Tablets made from *A.mangium* one month seasoned wood had a higher strength than tablets made from six months seasoned wood biomass, if moisture content and pressure are constant.

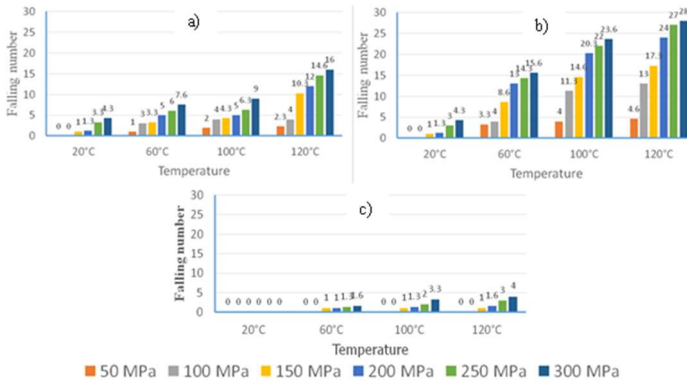


Figure 12. Relationship between falling number, temperature and pressure (a) $x < 0.8$ mm - one month seasoned wood; (b) $x < 1.6$ mm - one month seasoned wood; (c) $x < 1.6$ mm - six months seasoned wood

THESIS 4. Correlation of lignin, cellulose and starch with biomass agglomeration

I have proved the essential correlation between the properties of the produced agglomerate and the phase composition of the biomass.

THESIS 4.a) Correlation of cellulose with tablet density

The tablets compressed at higher pressure had a higher density, if the same production conditions (Figure 13). The tablet densities were determined by cellulose content of the biomass materials, tablets made from higher cellulose content of material had higher density in the case of without treatment materials and the same starch content (Figure 14).

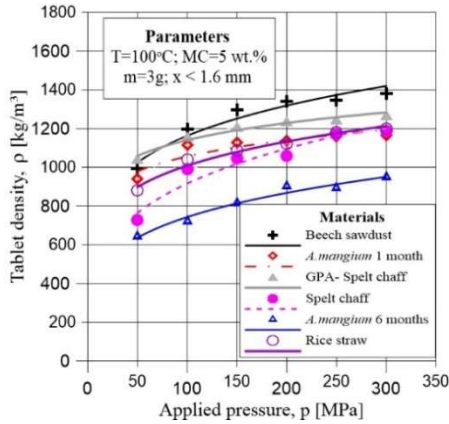


Figure 13. Compressibility data for various materials with the same production conditions

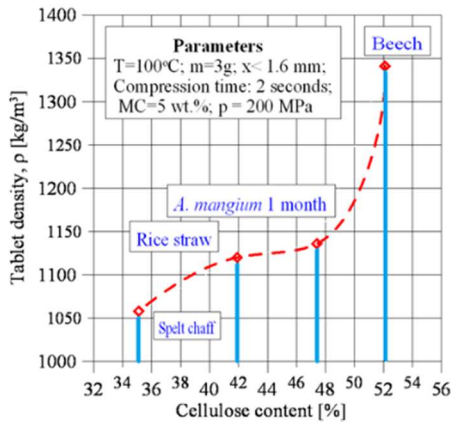


Figure 14. Relationship between cellulose content and tablet density

THESIS 4.b) Correlation of lignin with tablet strength

Falling number values in the case of tablets made from different materials at the same production conditions are shown in Figure 15. In the case of untreated material, the falling number of tablets made from *A. mangium* one month seasoned wood have the highest value and those made from rice straw had the lowest value. In the case of treated material, the falling number of tablets made from *A. mangium* six months seasoned wood was lower than those made from the GPA-spelt chaff.

The strength (drop strength) of tablets were determined by lignin content of the biomass materials, tablets made from higher lignin content of material had higher tablet strength in the case of without treatment materials and the same starch content (Figure 16).

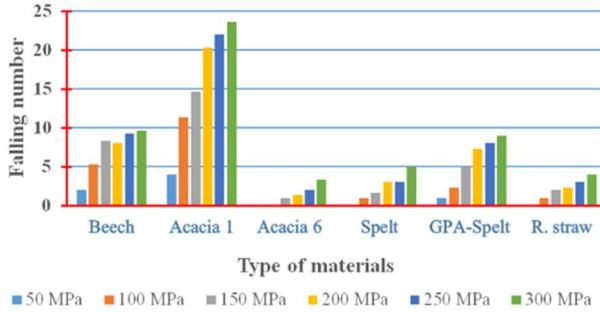


Figure 15. Drop strength (falling number) of tablets made from various materials with the same production conditions

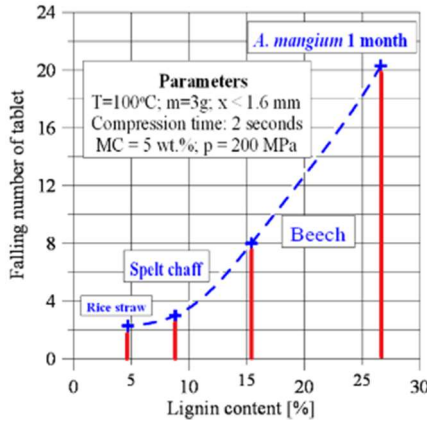


Figure 16. Relationship between lignin content and falling number of tablets

THESIS 4.c) Structure of tablets

The morphology of biomass granules after compression, in conjunction with their chemical composition and influence of tablet densities were introduced. The cross-sectional surfaces of tablets were investigated by an optical microscope as shown in Figure 17. In the case of untreated materials (Figure 17 a, b), tablets made from rice straw ($x_{50} = 0.63$ mm) with the lower lignin content (4.7 %) had the smaller granules, smoother, smaller pores and higher densities than those made from spelt chaff ($x_{50} = 0.8$ mm). Similar results can be observed in the case of treated materials (Figure 17 c, d).

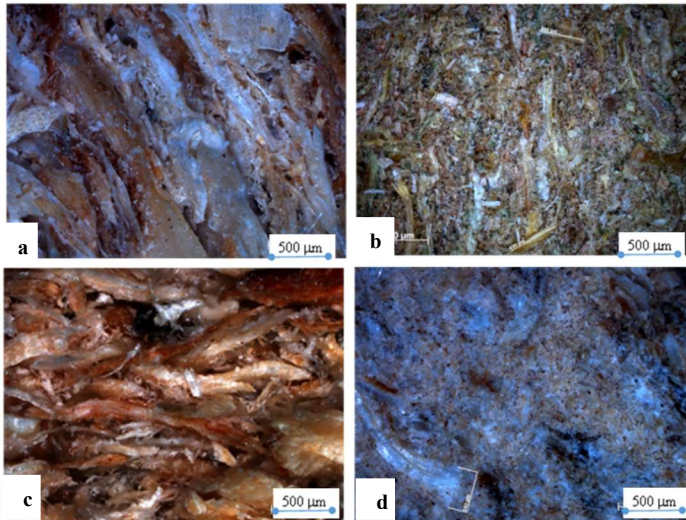


Figure 17. Cross sectional surface of tablets; (a) spelt chaff; (b) Rice straw; (c) *A. mangium* 6 months, $x_{50} = 0.75$ mm; (d) GPA-spelt chaff, $x_{50} = 0.55$ mm. (optical microscope: Zeiss AXIO Imager.M2m)

4. Opportunities for application and further development

The process optimization of pelletizing such as moisture content, particle size and temperature equivalent each type of biomass can be determined by single pelletizer unit 1.

The single pelletizer unit 2 can be used for other materials as well to find the optimal conditions of backup pressure, moisture content and particle size during the process of agglomeration. The membrane of the measurement disc should be made from a material the properties which are equivalent to carbon steel properties.

The new equation (modified Johanson's equation) allow the forecasting of the effect of various production parameters on the quality of agglomeration. The lignin content - falling number relationship and cellulose content - density relationship allows the expected tablet parameters to be calculated which can be used in industry (e.g. forecast mixtures behavior).

The experimental method can be used for other materials as well to find the optimal conditions of pressure, temperature, moisture content and particle size during an agglomeration process. The ground post-agglomerated raw material (spelt chaff) should be investigated in a new research work.

The determination of other components of biomass (for example hemicellulose, proteins, lipid and fat) and compression time are to be studied in a further work.

5. List of publications

Paper in international journal

[1] Trinh, V. Q., Nagy, S., Csöke, B.: *Effect of Moisture Content and Particle Size on Beech Biomass Agglomeration*. Advances in Agriculture & Botanic-International Journal of the Bioflux Society. Vol 9, issue 2 (2017), p79-89. Online ISSN 2067-6352; Print ISSN 2066-7639.

Paper in Hungarian Journal

[2] Trinh, V. Q., Nagy, S.: *Effect of Various Production Parameters on Biomass Agglomeration*. Journal of Geosciences and Engineering, Vol.4, No.7 (2015), p86-96. HU ISSN 2063- 6997.

Paper in Vietnamese Journal

[3] Trinh, V. Q., Nagy, S.: *Agglomeration of Acacia mangium Biomass*. Vietnam Journal of Science and Technology. 56 (2) (2018) p196-207. ISSN 2525-2518.

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