Effect of moisture content and particle size on beech biomass agglomeration

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Abstract. The utilisation of biomass agglomerates is an attractive option for heat and power production to reduce greenhouse gas emissions. Agglomeration of biomass, such as tableting, can increase bulk density, improve storability, reduce transportation costs and increase the quality of products. The purpose of this study is to analyse the effects of moisture content and particle size on beech biomass agglomerates and also to identify the optimal conditions of the process for producing tablets with high density. Beech biomass was compressed in a load cell by a hydraulic piston press with 25 mm diameter. Effects of the independent variables, including moisture content (4.71 wt.% to 19.55 wt.% with particle size < 1 mm, and 4.74 wt.% to 19.45 wt.% with particle size < 2 mm) were investigated. Results showed that at the same pressure, increasing moisture content resulted in lower density of the tablet, tablets made from raw material with the smaller particle size have lower strength than tablets made from material with the larger particle size, and a new compression equation can be introduced, which contains moisture content as parameter.

Key Words: agglomeration, tableting, compressibility, biomass, beech sawdust.

Introduction. Agglomeration is the mechanical process in which the particle size of solid disperse materials (bulk materials, fine particles of slurry) is increased by bonding forces between the particles. Agglomeration has three main types: pressure agglomeration (briquetting, extrusion, tableting, pelletising), growth agglomeration, and sintering (Tarján 1986). In this paper pressure agglomeration is considered applied to biomasses.

Environmental issues, the increasing demand for energy and the decreased availability of fossil fuels have all encouraged the development of sustainable technologies based on renewable raw materials. One of the main advantages of biomass as a source of energy is that it is a clean and renewable product, which contributes to the reduction of greenhouse gas emissions and fossil fuel dependence. Biomass manipulation requires great energy consumption due to its elevated moisture, irregular shapes and sizes and low bulk density, which makes it difficult to transport, store and use the material in its original form. The processing of biomass into pellets significantly reduces the costs of storage and transportation; moreover, the pelleted biomass possesses higher density, homogeneity and energy potential (Moya et al 2015).

Raw materials of biomass pellets include agricultural wastes, by-products and forestry residues. According to Mustelier et al (2012), it is possible to produce pellets even from undergrowth with physical and thermochemical characteristics similar to the pellets sold on the market. A wood pellet is a densified fuel with homogeneous physical properties suitable for use at various scales in domestic and industrial furnaces (Lee et al 2011).

Wood pellet production in Europe was estimated to be 10 million tonnes (Sikkema et al 2011) and 6.2 million tonnes for North America (Spelter & Toth 2009) for the year 2009. Another study by Cocchi et al (2011) estimated between 2009 and 2010 global installed production capacity of the pellet industry has recorded 22% increase reaching over 28 million tonnes. 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 pellet production capacity to 46 million tonnes by 2020 (Pöyry 2011).

More than one fifth of Europe's heating consumption is expected to come from renewable energy sources (RES) in 2020. The share of RES in heating and cooling will increase from 10.2% in 2005 to 21.3% in 2020. Biomass is expected to make up 17.2% of heating and cooling consumption, heat pumps from aerothermal and hydrothermal energy 1.6%, solar thermal energy 1.2% and geothermal energy 1.3% (EREC 2011).

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 usually results in better quality agglomerates; it is possible to achieve higher density (also higher calorific value) and strength. On the other hand, moisture content reduction (drying) has a large energy demand. To find the optimal production parameters, the exact relation between moisture content and briquettability (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.

This study investigates the effects of moisture content and particle size on the density and strength of European beech (*Fagus sylvatica*) biomass tablets.

Theoretical background. Five different compaction equations for describing briquetting processes, are introduced here. The stress-strain behaviour of biomass and the relationship between moisture content and porosity are also introduced.

Pressure agglomeration principle. During pressure agglomeration, new, enlarged entities (tablets, briquettes, etc.) are formed by applying external forces to particulate solids in more or less closed dies that define the shape of the agglomerated product (Figure 1) (Pietsch 2005).

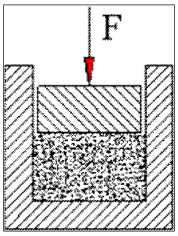

Figure 1. Pressure agglomeration.

Compaction equations. Different approaches have been taken in order to calculate the connection between applied pressure and agglomerate density.

The Johanson equation (1965) can take two forms:

$$\frac{\rho}{\rho^*}=\left(\frac{p}{p^*}\right)^{1/\kappa}; \quad \frac{F}{F_o}=\left(\frac{V_o}{V}\right)^{\kappa} \qquad (1)$$

where κ is compressibility factor, ρ is agglomerate density, p is tableting pressure, F is tableting force, V is tablet volume, and p^*, ρ^*, F_o, V_o are reference values (if surface perpendicular to force and mass of tablet are constant) (Stieß 1997).

Liu & Wassgren (2016) modified the Johanson model for improved relative density predictions:

$$\frac{P}{P_{initial}}=\left(\frac{\eta}{\eta_{initial}}\right)^{\kappa} \qquad (2)$$

where $\eta_{initial}$ is the inlet relative density, $P_{initial}$ is the corresponding pressure according to the fit data; η is the powder's relative density.

Walker (1923) reported a series of experiment on the compressibility of powder. He expressed the volume ratio V as a function of applied pressure P:

$$V = a_1 - K_1 \ln p \qquad (3)$$

where a_1 and K_1 are constants (Walker 1923; Adapa et al 2009).

Heckel (1961) proposed a model to express the compaction behaviour of compressed powder. The equation expresses the density of powdered materials in terms of packing fractions as a function of applied pressure:

$$\ln\frac{1}{1-\rho_f}=mp+n \quad \text{and} \quad \rho_f=\frac{\rho}{\rho_1X_1+\rho_2X_2} \qquad (4)$$

where ρ_f is packing fraction or relative density of the material after particle rearrangement, P is applied pressure (MPa); m, n are Heckel model constants, ρ is bulk density of compacted powder mixture (kg m^{-3}); X_1, X_2 are mass fraction of components of the mixture (Heckel 1961; Mani et al 2004).

Kawakita & Lüdde (1971) performed compression experiments and proposed an equation compaction of powders based on an observed relationship between pressure and volume:

$$\frac{p}{C}=\frac{1}{a_2b_2}+\frac{p}{a_2} \qquad (5)$$

where P is applied pressure, a_2 and b_2 are constants, and C is relative volume decrease or engineering strain given by the equation:

$$C=\frac{V_o-V_P}{V_o} \qquad (6)$$

where V_o is the initial volume and V_P is volume measured at any given pressure (Kawakita & Lüdde 1971; Chevanan et al 2010). The model was further developed by Sone (1969) and is used for understanding the compaction characteristics by tapping. Sone's model has close resemblance to the Kawakita and Lüdde model and the pressure term in the Kawakita and Lüdde model is replaced with number of tappings. Equation (6) can be rewritten in the form:

$$\frac{n}{\gamma_n}=\frac{1}{a_3b_3}+\frac{n}{a_3} \qquad (7)$$

where γ_n is volume reduction ratio, n is number of tapping and a_3 and b_3 are constants. The volume reduction ratio γ_n calculated using:

$$\gamma_n=\frac{V_o-V_n}{V_o} \qquad (8)$$

where V_o is initial volume, and V_n is volume after n taps.

A compression equation was proposed by Panelli & Filho (2001) given as:

$$\ln\frac{1}{1-\rho_r}=A\sqrt{p}+B \qquad (9)$$

where ρ_r is the relative density of compactness, A is a parameter related to densification of the compacted agglomerate by particle deformation and B is a parameter related to powder density at the start of compression (Panelli & Filho 2001).

Some papers have reported about the relationship between moisture content and porosity. For instance, an increase in moisture content has been reported to increase the bonding area of plastically deforming materials during compaction due to plasticising and lubricant effects (Pande et al 1995; Steendam et al 2001). Moisture facilitates particle rearrangement and deformation during compaction resulting in tablets with a lower porosity at constant compaction pressure, and therefore increased area over which interparticulate bonding can take place. This effect has been reported for pharmaceutical powders as a reduction in the mean yield pressure derived from the Heckel equation (Nokhodchi et al 1996; Sun 2008; Malaj et al 2010) and also by an increase in the compressibility coefficient derived from the Walker equation (Walker 1923).

Stress-strain behavior of wood. Ahmad et al (2010) introduced results for tropical hardwoods. Stress-strain graphs for tensile strength of solid timber made from three types of tropical hardwoods were determined. The graph shows that strain increases with load and is approximately linear until the point the specimen fails. The slope of the graph represents the Modulus of Elasticity (MOE). The ultimate stress then occurs at the highest point when the load reaches its maximum value. The samples break at that final stage and the reading indicates an abrupt drop in strength. Therefore, solid timber is a brittle material. The grade stresses for timber in structural size are relatively high compared to the grade stresses based on a small clear specimen.

Moisture content dependence, novelty. Although many works have been devoted to the study of a compaction equation between density and pressure, 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 to supplement the original Johanson's equation with the parameter moisture content. The spring-back ratio (SBR) was also investigated because it has an effect on the density of tablets.

Material and Method. The biomass for experiment, hydraulic piston press and experimental procedure are introduced. The study was carried out from the period of February 2016 to April 2017.

***Materials**.* Beech sawdust was chosen as raw material for our experiments. It originated from Miskolc, Hungary (Borsodwood Ltd.). It was dried and then ground using a cutting mill (Retsch SM2000) in one step (screen size 2 mm) and in two steps (screen sizes: 2 mm, 1 mm). The biomass was stored at room temperature (25°C), in closed plastic bags. The moisture contents of beech biomass were determined to be 1.47 wt.% in the case of particle size < 1 mm, and 1.44 wt.% (particle size < 2 mm).

The raw material of beech sawdust can be seen in Figure 2. It can be observed that beech sawdust is a homogeneous material.

Figure 2. Beech sawdust with particle size < 2 mm; (left) optical camera; (right) optical microscope: Zeiss AXIO Imager.M2m.

***Hydraulic piston press**.* The hydraulic piston press (Figure 3) 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 feedrate is 30 mm s^{-1}. The measuring of the piston position is done with an incremental encoder.

Figure 3. Hydraulic piston press.

Experimental procedure. The hydraulic piston press with diameter 25 mm was used for two different kinds of tests and each tablet was made by the compression of 5 g sawdust. Applied pressures on the surface of tablets were 50, 100, 150, 200, 250 and 275 MPa, temperature 20°C.

In the first test, the applied moisture contents were 4.71, 9.27, 14.9, and 19.55 wt.% with particle size < 1 mm and 4.74, 9.68, 14.6, and 19.45 wt.% with particle size < 2 mm.

In the second test, spring-back ratio experiments were carried out with particle size < 2 mm, moisture content 4.9, 9.6, 13.9 and 18.8 wt.%.

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

$$SBR = \frac{H_t - H_{tP}}{H_{tP}} 100\% \qquad (10)$$

where H_t is the height of the produced tablet, H_{tp} is minimum height of the tablet under pressure.

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 minimum height of tablets under pressure was measured by the incremental distance measurement method.

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.

Raw material particles and cross sectional surfaces of tablets were investigated with an optical microscope Zeiss AXIO Imager.M2m.

Results and Discussion. Tablet density, spring-back ratio, tablet strength, and the structure of tablets were determined in our investigations.

Tablet density and compressibility. Tablets produced by processes with different parameters are shown in Figure 4. The tablet density values are recorded as an average of five measurements with particle size < 1 mm and also with particle size < 2 mm (T = 20°C).

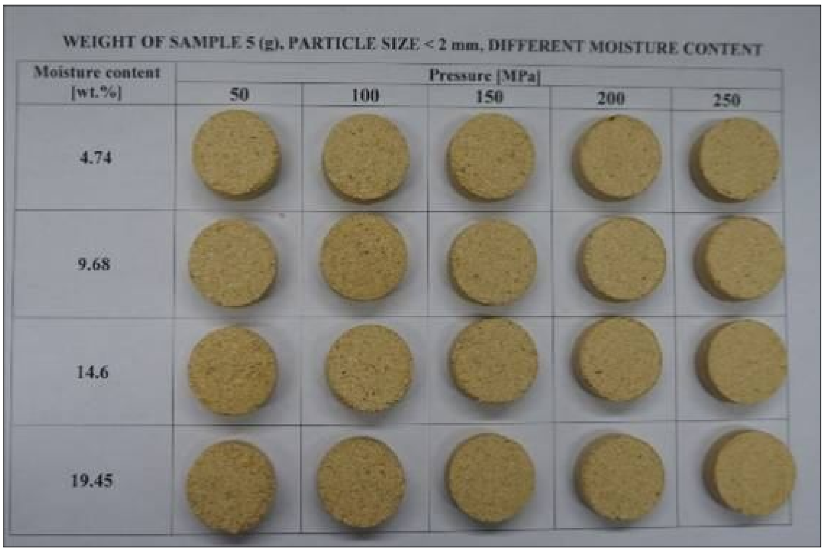

Figure 4. Tablets made from particle size < 2 mm.

Figure 5 (left) shows the pressure density values and the fitted Johanson curves in the case of particle size < 1 mm raw material at 4.71, 9.27, 14.9 and 19.55 wt.%. Table 1 shows the constants of the fitted curves and coefficient of determination (R^2), residual mean square (σ) and calculated deviation (V_s). Results for particle size < 2 mm are introduced in Figure 5 (right), and Table 2.

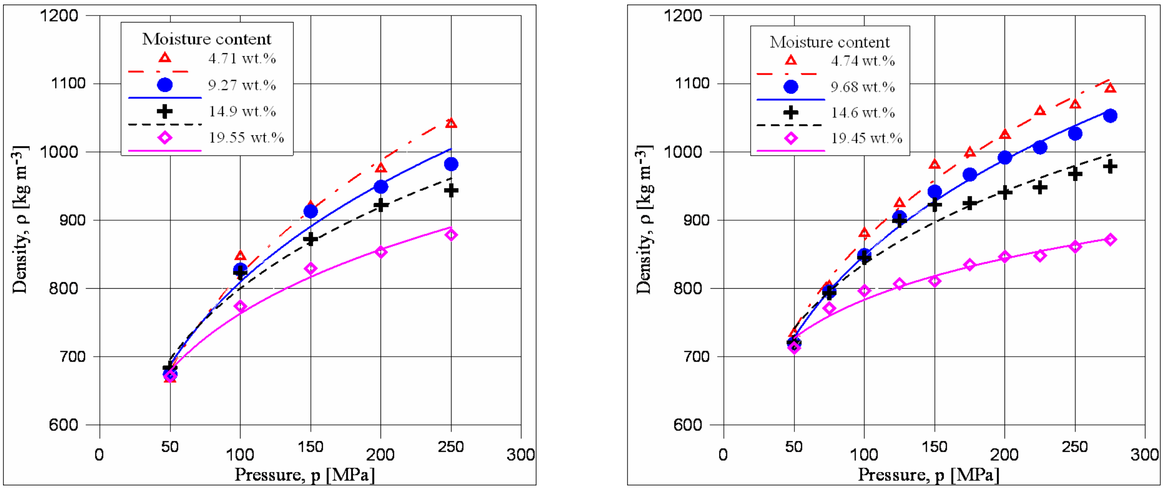

Figure 5. Compressibility data for biomass with different moisture content; (left) particle size < 1 mm; (right) particle size < 2 mm.

Table 1

Constants of Johanson's equation for different moisture content (particle size < 1 mm)

Moisture content [wt. %]	*Constant α*	*Constant 1/κ*	*Spread deviation: V_s Coefficient of determination: R^2 Residual mean square: σ*
4.71	237.8374	0.2687	R^2 = 0.9858; σ = 0.00055; V_s = 1.9 %
9.27	273.9956	0.2352	R^2 = 0.9782; σ = 0.00066; V_s = 3.0 %
14.9	318.1958	0.2002	R^2 = 0.9775; σ = 0.00049; V_s = 2.3 %
19.55	352.8749	0.1674	R^2 = 0.9835; σ = 0.00025; V_s = 1.9 %

Table 2

Constants of Johanson's equation for different moisture content (particle size < 2 mm)

Moisture content [wt. %]	*Constant α*	*Constant 1/κ*	*Spread deviation: V_s Coefficient of determination: R^2 Residual mean square: σ*
4.74	293.8512	0.2360	R^2 = 0.9919; σ = 0.00015; Vs =1.1 %
9.68	307.3354	0.2205	R^2 = 0.9934; σ = 0.00011; Vs =2.2 %
14.6	377.0277	0.1729	R^2 = 0.9588; σ = 0.00044; Vs =2.6 %
19.45	478.6041	0.1070	R^2 = 0.9660; σ = 0.00013; Vs =1.4 %

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 (in the case of < 1 mm raw material at 100 MPa tablet densities: 847.2 kg m^{-3} (MC = 4.71 wt.%) and 773.76 kg m^{-3} (MC = 19.55 wt.%)). The reason for that could be the increasing SBR. Moisture content may have an effect on the strain-stress behaviour of biomass particles, and on the volume of particles, both of which affect SBR.

Tablets made from particle size < 2 mm have higher density than those made from particle size < 1 mm, at constant pressure, temperature and similar moisture content. For instance, at 200 MPa and T = 20°C, the tablet densities were 975 kg m^{-3} (particle size < 1 mm, MC = 4.71 wt.%) and 1024 kg m^{-3} (particle size < 2 mm, MC = 4.74 wt.%).

To describe the compressibility of beech sawdust Johanson's 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.

The spread deviation values (V_s) of fitted Johanson's equations are calculated (Table 1) and shown to have a value smaller than 3%. At the same temperature the higher moisture content results in higher constant a, and constant κ. A linear equation can be found, that describes the moisture content dependence of values a, and κ, and so compression can be described by an equation based on the original Johanson's equation and containing moisture content as a parameter.

Johanson's equation: $\rho = a.p^{1/\kappa} = a.p^{b}$

Where $a = f(m) = a_1 m + a_2 = 7.763917m + 201.724288$ and

$$b = f(m) = b_1 m + b_2 = \frac{1}{\kappa} = -0.006749m + 0.299591$$

The new equation for particle size < 1 mm beech sawdust:

$$\rho = (7.763917m + 201.724288)p^{(-0.006749m + 0.299591)} \quad (11)$$

where m is moisture content.

Spread deviation values (V_s) were calculated and found to have a value smaller than 2.6% (Table 2).

The new equation for particle size < 2 mm beech sawdust:

$$\rho = (12.704515m + 210.257636)\,p^{(-0.0088514m + 0.291352)} \quad (12)$$

The processes were well described by the applied Johanson functions at each moisture content.

The modified Johanson's equation, which contains moisture content m as a parameter, provides only a small deviation of density of approximately 0.7%.

Thus, we conclude that the modified Johanson's equation gives accurate results, particle size < 1 mm, MC = 4.71 wt.%, p = 50 MPa, density of Johanson's equation 680 kg m^{-3}; density of modified Johanson's equation 679 kg m^{-3} and also for particle size < 2 mm, MC = 19.45 wt.%, p = 75 MPa, density of Johanson's equation 765 kg m^{-3}, density of modified Johanson's equation 759 kg m^{-3}.

Spring-back ratio. Figure 6 shows the relationship between applied pressure and SBR in the case of different moisture contents of raw materials (4.9, 9.6, 13.9 and 18.8 wt.%).

This relationship can be described by following function: $\mathbf{SBR = ap^c}$. The constants c and a are corresponding to each moisture content.

Tablets made from raw material with larger moisture content had larger SBR (at the same pressure, temperature and particle size). In the case of 18.8 wt.% moisture content SBR of over 50% was measured. Tablets with 4.9 wt.% moisture content had only 20.1 to 35.8% SBR depending on pressure, in the examined pressure range.

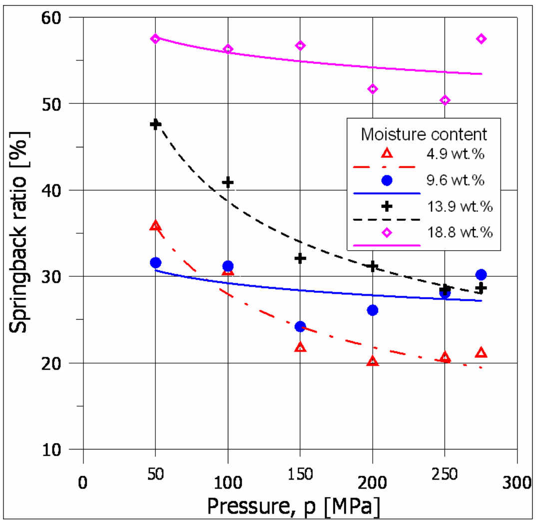

Figure 6. Relationship between pressure, spring-back ratio and moisture content (particle size < 2 mm).

Tablet strength. Falling number values are shown in Figure 7 as a function of moisture content at different pressures for both particle size < 1 mm and < 2 mm raw materials. Increasing moisture content resulted in lower tablet strength at the same pressure and with same particle size. Adsorbed moisture on the particle surface can also interfere with intermolecular forces, thus reducing the bond strength and resultant tablet strength (Herrmann 1971; Lordi & Shiromani 1983; Nyström et al 1993). Tablets made from particle size < 2 mm form tablets with higher strength (falling number: 5.66 at 250 MPa and 4.74 wt.%) than tablets made from particle size < 1 mm biomass (falling number: 4.33 at 250 MPa and 4.71 wt.%) 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 under the same experimental conditions.

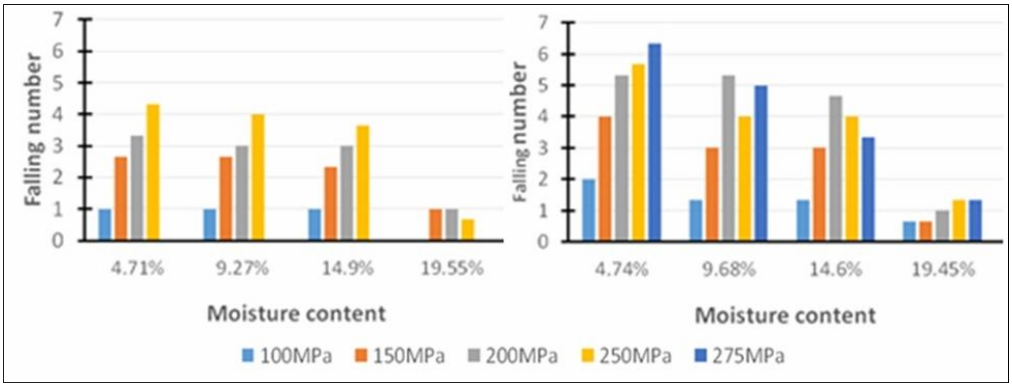

Figure 7. Relationship between falling number, moisture content and pressure; (left) particle size < 1 mm; (right) particle size < 2 mm.

Structure of tablets. The cross sectional surfaces of tablets are shown in Figure 8. The tablet made at pressure 50 MPa had more space between particles (porosity is higher) than the tablet made at 250 MPa, with the same moisture content 4.71 wt.%. The same effect can be recognised in the case of MC = 19.55 wt.%. In the optical microscopy pictures no relevant differences can be seen between tablets made with 4.71 wt.% and 19.55 wt.% moisture content. The reasons for that are that generally, with increasing external forces acting upon the particulate matter during size enlargement, porosity and characteristics related to this parameter decrease while density and strength increase (Pietsch 1991).

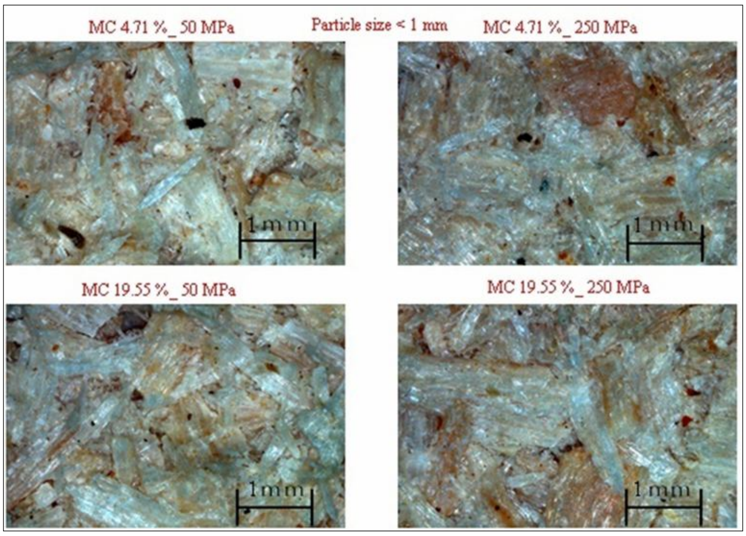

Figure 8. Cross sectional surface of tablets (optical microscope: Zeiss AXIO Imager.M2m).

Conclusions. This paper has presented tools and methods to evaluate the effect of moisture content, pressure and particle size on tablet density in the case of beech sawdust. The description of the processes is essential to be able to determine the optimal production parameters.

While the applied Johanson functions describe the processes well (at 9.27 wt.%, in the case of < 1 mm raw material V_s = 3.0 %, when using < 2 mm raw material V_s = 2.2 %) the modified Johanson's equation for compression introduced here contains also moisture content as a parameter.

Experiments found that if pressure and particle size are kept constant, increasing moisture content results in lower density tablets.

Increasing moisture content resulted in lower tablet strength at the same pressure and with the same particle size. An effect of particle size was also identified: tablets made from sawdust with particle size < 2 mm form tablets with higher strength than those made from particle size < 1 mm biomass when moisture content and pressure are kept constant.

The introduced experimental method can be used for other materials as well to find the optimal conditions of pressure, moisture content and particle size during agglomeration.

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