

# LogistEC

## Logistics for Energy Crops' Biomass

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**Priority: Food, Agriculture and Fisheries, and Biotechnology**

### **Deliverable D3.2**

***Preliminary quality characteristics of commoditized energy crops***

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## Glossary and Definitions

ExCom	Executive Committee
BP	Bourgogne Pellets France
CRL	Coppice Resources Ltd
INRA	Institut national de la recherche agronomique
CENER	The National Renewable Energy Centre
RRes	Rothamsted Research
ECN	Energy research Centre of the Netherlands
CFN	C.F.Nielsen A/S
Risø DTU	Technical University of Denmark Risø Campus

## Summary

**Objectives:** To test the pelletizing and briquetting properties of different biomass species at different conditions in order to decide the optimal operation parameters for bench-scale pelletizer.

**Rationale:** a pellet model previously developed by Risø DTU is used to screen the influence of different parameters, and a theory of correlating the single pellet model with a bench-scale rotating pellet mill is proposed and tested.

The different species of biomass are then tested in a briquetting press, to determine if briquetting is a viable choice of densification technique, and to verify that the operation parameters with the most significant impact on production are equally important in briquetting.

**Teams involved:** Risø DTU, CFN, ECN

**Geographical areas covered:** France, Spain, UK, and Denmark

In total twelve biomass samples (from seven different species) have been received from partners. All samples were ground using the same hammer mill after receiving, particle size distribution and moisture content were determined for each sample. Single-pellet press was used to study the pelletizing properties of different biomass. First, each biomass has been adjusted to three different moisture contents, and the friction between pellet and press channel during pelletizing and pellets' densities were determined to choose the optimal moisture content for each biomass. Second, the influence of pellet press temperature on the friction during pelletizing was measured for each kind of biomass in order to find the optimal die temperature in real pellet production. It was found different biomass has different desired temperature for pelletizing. In the end, three kinds of biomass (fresh miscanthus, triticale, and fescue) have been pelletized in a bench-scale pelletizer to test the proposed theory of die temperature influence. The results showed good consistency with our theory, a steady pellet production requires die temperature reaching a certain level, which is dependent on biomass species.

More experiments will be run in a bench-scale pelletizer, and the results will be included in D3.3 (Improved densification recipes for raw energy crops). The work on pelletizing torrefied and torwashed biomass will be described and carried out in D3.5 (Improved densification recipes for thermally pretreated energy crops). Teams involved in D3.2 are Risø DTU and CFN. The WP leader from ECN validates the table of contents, and approves the final version.

# 1. Pelletizing

## 1.1. Material and Methods

### 1.1.1. Materials

Biomass samples were supplied by different partners in this project, below is the table of all samples received by the time this report was prepared (November 2013). All samples from INRA were dried after harvesting to remove “free water”. Green miscanthus means the fresh miscanthus, which has a green color. They were ground and pelletized within 10 days after collecting.

**Table 1 : Details of biomass samples received from partners, and pelletizing tests conducted in this deliverable.**

Species	Supplied by	Region	Harvest	Moisture content (w.b.)	Single-pellet press	Bench-scale pelletizer
Triticale	INRA	Versailles, France	10-07-2013	11%		
	CENER	Extremadura, Spain	2013	10%	✓	✓
Fescue	INRA	Versailles, France	22-07-2013	9%	✓	✓
Alfalfa	INRA	Versailles, France	22-07-2013	9%	✓	
Sorghum	INRA	Versailles, France	15-09-2013	10%	✓	
	CENER	Spain	2013	11%		
Miscanthus	BP	Burgundy, France	2013	15%	✓	
Green miscanthus	RRes	Woburn, Bedfordshire, UK	10-10-2013	50%	✓	✓
Willow ( <i>salix viminalis</i> )	CRL	Retford, Nottinghamshire, UK	28-09-2013	12%	✓	

All biomass samples were ground using a hammer mill (J. N. Jensen & sønner APS, type 55, Denmark) before the pelletizing tests. The hammer mill is equipped with a 4 mm sieve and is powered by a 7.5 kW electric motor. Biomass particles in the range of 0.25 – 1 mm were used for single-pellet tests. Moisture content of the biomass was measured using a moisture analyzer (Halogen moisture analyzer, Mettler Toledo, Switzerland).

### 1.1.2. Single-Pellet Press

The biomass was pelletized using a single-pellet press (designed and constructed at the biomass gasification group of DTU) as shown in Figure 1. The unit consisted of a cylindrical die 8 mm in diameter, made of hardened steel, packed with heating elements and thermal insulation. The temperature was controlled using a thermocouple connected to a control unit. The end of the die was closed using a removable backstop. Force was applied by a hydraulic press. The pellet unit is placed on a load cell stand to measure the forces needed to make the pellet and the static friction (force that is required to extrude the pellet from the press channel, also referred as 'back pressure'). For each test, at least two replicates have been made. The die was rinsed with acetone when changing biomass species.

In order to simulate the pelletizing process in a real pellet mill, the pellet should be built up in sequential layers. Biomass was loaded in sequential steps and with 0.25 g per layer. For determining the optimal moisture content of the biomass, 4 layers per pellet was chosen; for investigating the die temperature influences and simulating the real pelletizing process, pellet was first made by 11 layers to reach the same length (about 34 mm) of channels in the bench-scale pelletizer, and then backstop was removed and one more layer of biomass was added before pellet was pressed out.

The biomass samples were compressed with a maximum pressure of 200 MPa. The pressure was released after 10 s, then the piston was removed and a new amount of sample was loaded and compressed until the pellet had the desired length/weight. In the end the backstop of the unit was removed and the pressure on the top of the pellet is increased from 0 until the pellet starts moving downward in the press channel. The minimum pressure necessary to start the movement is measured by the load cell. The pressure needed to make the pellet move is equal to the back pressure ( $P_x$ ) arising from friction along the pellet walls. This is a model of the process taking place in the rotating pellet mill, where the magnitude of the pressure exerted on the pellet by the roller is equal to the back pressure under steady-state conditions [1].



Figure 1 : Picture of single-pellet press

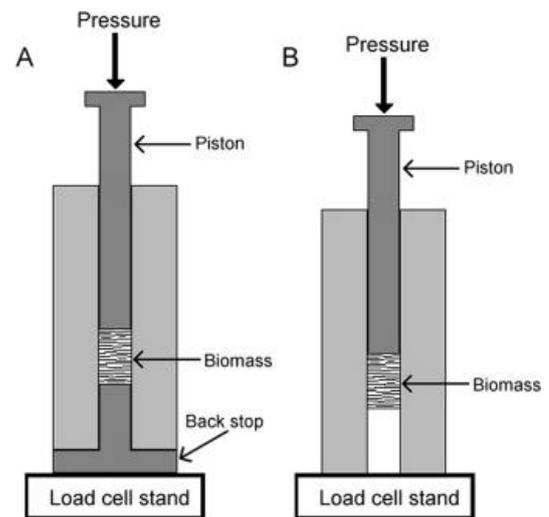


Figure 2 : Setup for pellet production (A). Setup for the measurement of back pressure (B) [1].

### 1.1.3. Bench-Scale Pelletizer

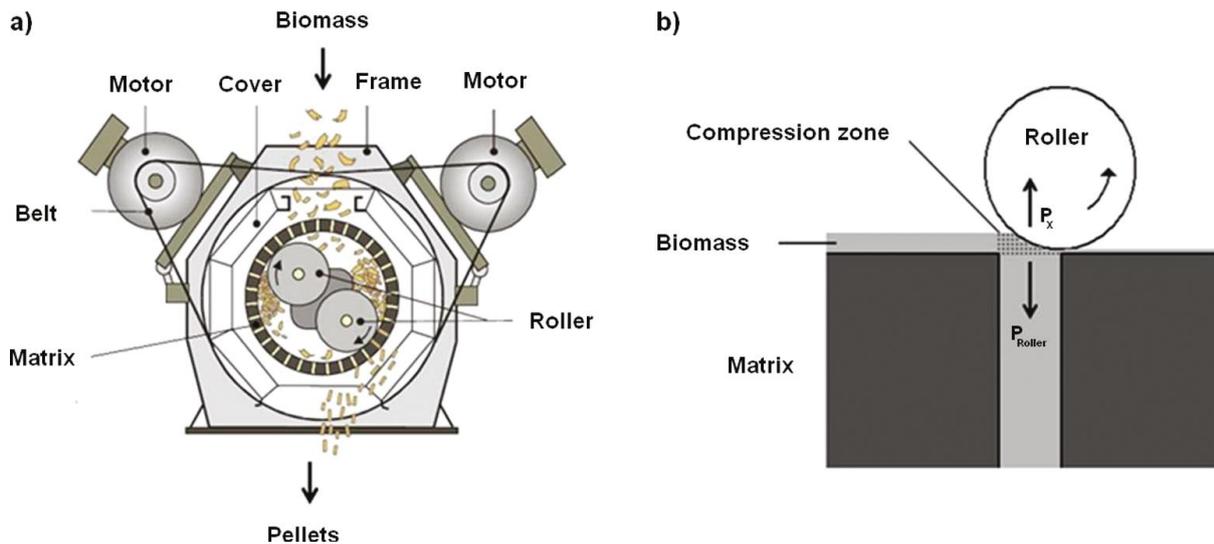
The bench-scale pellet mill (California Pellet Mill Co.) is based on extrusion for the pellet production, as shown in Figure 3. It is equipped with a vertical ring-type matrix, and the biomass is forced outward through the cylindrical holes by the action of an eccentrically mounted roller. A data-logger electric meter of the pellet mill motor functions as a load indicator. There are several sets of ring dies with different compression ratios, and each ring matrix has 40 holes. The ring matrix used in this report has a diameter of 7.8 mm and a length of 33.5 mm, giving a ratio of compression for the pellet mill of 4.3.



Figure 3 : Bench-scale California pellet mill at DTU [2].

The basic principle of pelletization is shown in Figure 4. When the matrix is rotating, the adjacent roller forces the raw material into the channels of the matrix. The actual compression of the material is likely only taking place in a minor compression zone at

the beginning of the channel. The compressed material in the matrix then functions as a continuously moving backstop, set up by the friction with the channel walls.



**Figure 4 : (a) Working principle of a ring matrix pellet press and (b) magnification of a press channel showing how biomass is compacted by the roller in the compression zone and subsequently flowing into the press channel where it is further compacted due to high friction between the biomass and the walls of the press channel [3].**

## 1.2. Results

### 1.2.1. Particle size distribution

All biomass samples were ground in the same hammer mill after reception. Figure 5 shows the particle size distribution of ground samples for different biomass species. It is interesting to see that green miscanthus had a much higher fraction of small particles (<0.5 mm) compared to dry miscanthus. Compared to other biomass, fescue had much finer particles after grinding. Sorghum, miscanthus, and willow showed similar particle size distribution, and had the biggest particles (ca. 50% particles with diameter less than 1 mm) after grinding. Three intercropping showed the similar particle size as their main species. After grinding there were more than 90% of particles less than 2 mm for all biomass.

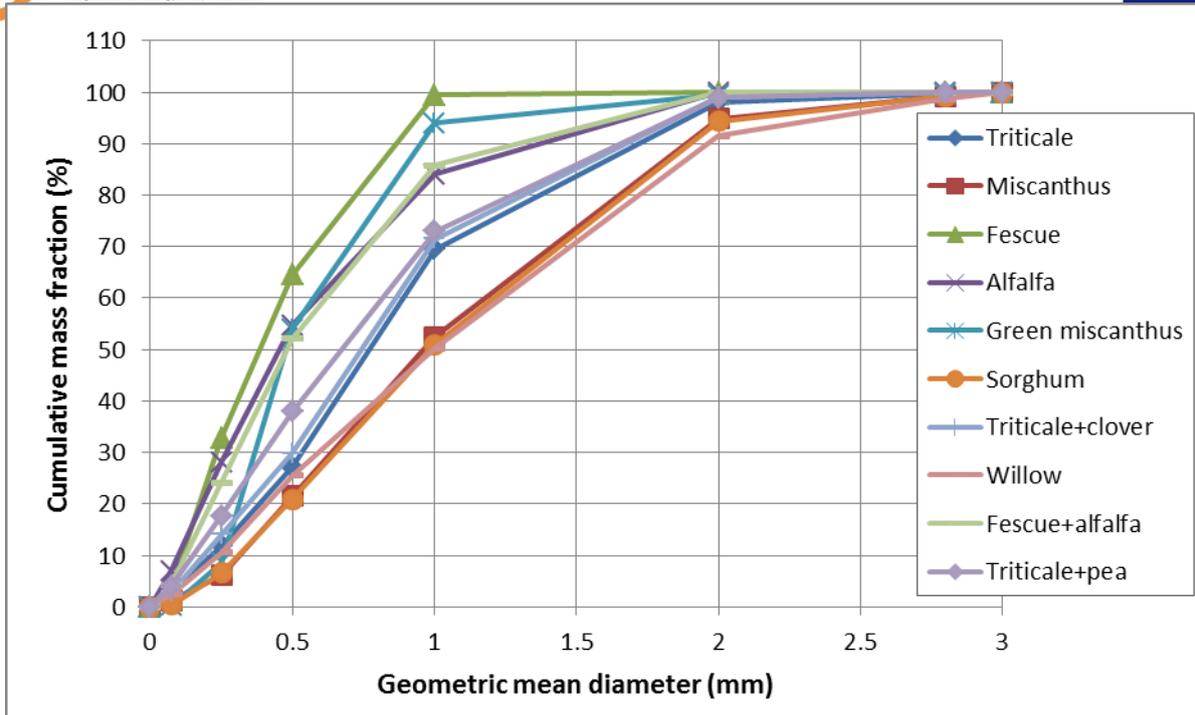


Figure 5 : Particle size distribution of different biomass materials after grinding in a hammer mill with Ø-4mm sieve

### 1.2.2. Optimal moisture content of different biomass materials

Single-pellet press was used to decide the optimal moisture content of each biomass for pelletizing. Biomass samples in the size range of 0.25 - 1 mm were adjusted to different moisture contents by drying at 100 °C or adding distilled water, and kept in a tightly closed plastic box for several days before pelletizing. During pelletizing, the die was heated to 90 °C. Biomass samples were loaded stepwise in amounts of 0.25 g into the die, and then compressed until a maximum pressure of 200 MPa was reached. The pressure was released after 10 s, this step was repeated for 4 times for each pellet resulting that every pellet weighs around 1 g. In the end, the pellets were removed from the die by removing the backstop and pushing out the pellet. The applied maximum force was logged, and it corresponds to the static friction ( $P_x$ ) in the press channel.

Table 2 shows the test results of different biomass at adjusted moisture contents. Density of the pellet was determined by weighing the pellet and using a digital caliper measuring the pellet dimensions right after pelletizing. The moisture contents selected for tests were based on the literature study conducted in the Deliverable D3.1. The optimal moisture content was chosen based on the static friction ( $P_x$ ) of the pellet and pellet density. For same material, the higher static friction usually results in more dense pellet and pellet with higher durability. But on the other hand, higher static friction also means higher energy consumption during pelletizing.

**Table 2 : Experimental results from single-pellet press at die temperature of 90 °C, and pelletizing pressure at 200 MPa (except for fescue, which was pressed at 100 MPa). Number in the bracket is the standard deviation of measured samples.**

Species	Moisture content	Px (N)	Density (kg/m <sup>3</sup> )	Optimal moisture content for pelletizing
Triticale	10%	1016 (53)	1126 (11)	10%
	15%	745 (20)	969 (17)	
	20%	637 (-)	760 (-)	
Miscanthus	10%	1610 (81)	1145 (8)	10%
	15%	1565 (32)	1046 (17)	
	20%	946 (15)	918 (16)	
Fescue	9%	309 (25)	1178 (5)	9%
	15%	186 (10)	1126 (12)	
	20%	137 (-)	-	
Alfalfa	10%	593 (15)	1233 (26)	10%
	5%	500 (10)	1230 (40)	
	3%	500 (-)	1237 (-)	
Sorghum	10%	287 (12)	1213 (18)	10%
	15%	260 (5)	1215 (37)	
	20%	206 (5)	1173 (-)	
Green miscanthus	50%	167 (-)	Didn't form dense pellet	x
Willow	5%	353 (10)	1153 (1)	10%
	10%	436 (44)	1190 (2)	
	12%	343 (20)	1168 (5)	

Green miscanthus, which has been pelletized within short time after harvesting, has been found not suitable for pelletizing. This is mainly due to the high moisture content in the fresh miscanthus.

### 1.2.3. Die temperature influence

Another series of tests have been done on the single-pellet press to simulate the real pelletizing process in the bench-scale pelletizer. The test procedure is similar as for determining the optimal moisture content, the difference being that 11 layers of biomass with 0.25 g per layer were compressed stepwise at 200 MPa while closing the end of the channel with a backstop, and the twelfth layer was compressed without the backstop. The static friction determined is also the pelletizing pressure of the last layer. In this way, it is possible to check if the static friction in the channel is high enough to form the pellet.

The results are shown in Figure 6. It can be seen most biomass experienced a friction increase when die temperature increased from room temperature to 60-90 °C, and then friction decreased when die temperature increased further. The influence of die temperature on static friction during pelletizing has been studied earlier in our group for woody biomass [4], and it was observed that there's a sudden decrease of friction at 100 °C for beech and about 70 °C for spruce. This was suggested as an indication of thermal softening of wood polymers (lignin) resulting in greater plasticity and flow

ability and the leaching of extractives to the surface. Low molecular weight hydrocarbons, such as oils and waxes are known to reduce the friction and their content on the pellet surface has been observed to increase (using infrared spectra) on pellets produced at higher die temperatures [4]. In order to form a good bonding between adjacent particles in the pellet and produce a mechanically strong pellet, high die temperature beyond the softening point (also referred as glass transition temperature) of wood polymers (lignin) is necessary. Furthermore, pelletizing in the bench-scale is a dynamic process. The compressed material in the press channel functions as a moving backstop, set up by the friction with the channel walls. The friction builds up when more material enters the channel, and the die temperature increases correspondingly. This step lasts until the die reaches certain temperature that the friction decreases to certain level, which allows the pellet to be pushed out. Based on this theory, a satisfactory pelletizing most likely happens in the region where  $P_x$  decreases with die temperature as shown in Figure 6. This means sorghum can be pelletized at the lowest die temperature (below 60 °C), a steady production of triticale and fescue pellets is possible when die temperature is higher than 60 °C, and a higher die temperature (> 90 °C) will be required to produce pellets from miscanthus, alfalfa, and willow.

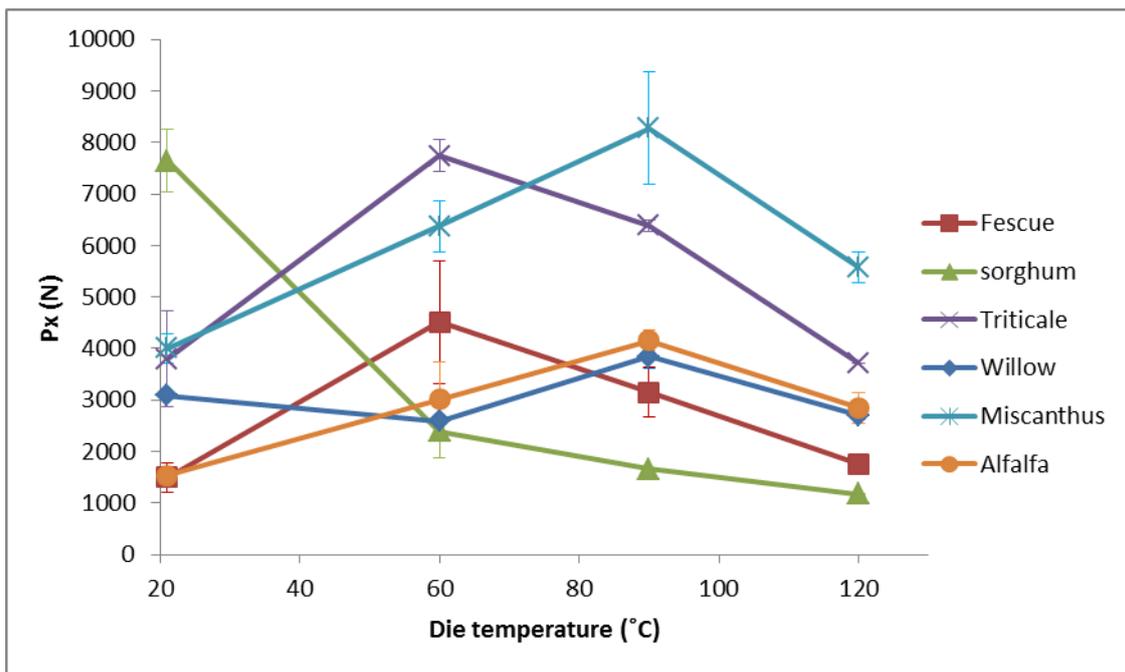


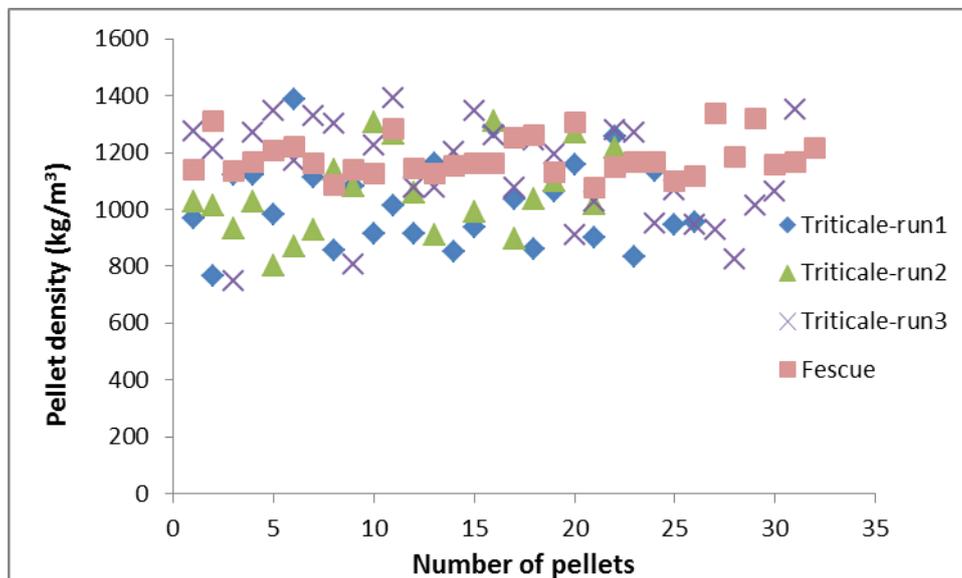
Figure 6 : Static friction of pressing out a long pellet (ca. 3 g, with diameter of 8 mm) at different die temperatures. Error bars are standard deviation of measured samples.

#### 1.2.4. Bench-scale pelletization for selected biomass

Triticale (from CENER), fescue and green miscanthus have been tested in the bench-scale pelletizer. In agreement with the single-pellet press results, it was not possible to produce dense pellets from green miscanthus. The pellets were very loose and expanded right after leaving the pelletizer. Therefore, green miscanthus was used as a start-up material for the pelletizer to achieve the high die temperature before adding other biomass. Triticale pellets have been produced in three runs, and

pellet density (as shown in Figure 7) has been determined by measuring 22-31 pellets from each run. The average density of triticale and fescue pellets are  $1073 \pm 56 \text{ kg/m}^3$  and  $1182 \pm 70 \text{ kg/m}^3$  respectively, which are very close to the density of pellets made from single-pellet press at same moisture content in Table 2.

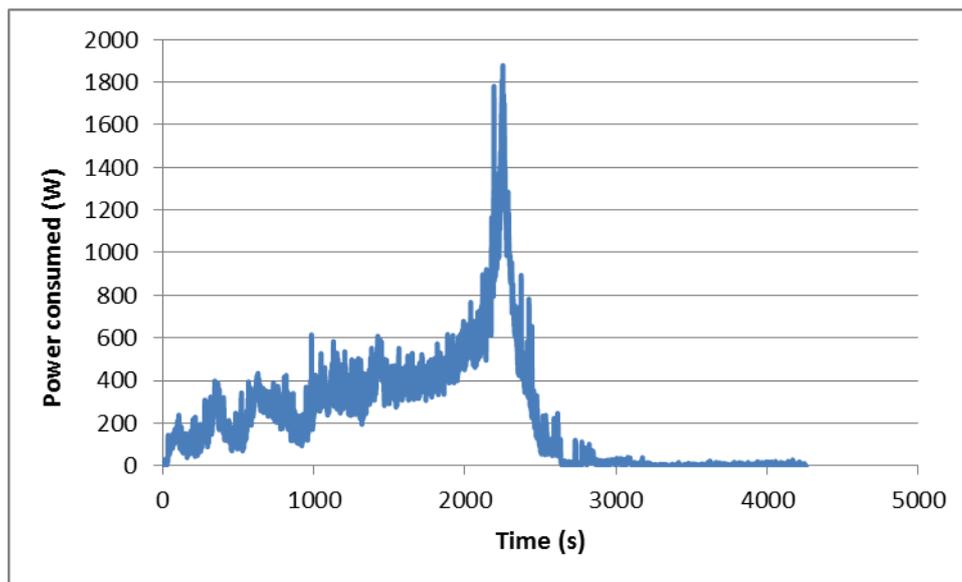
In the first two runs of pelletizing triticale, feedstock was added when die temperature was about  $40 \text{ }^\circ\text{C}$  and pellets were formed and come out of the machine when temperature reached  $50 \text{ }^\circ\text{C}$ . Then the temperature stabilized at about  $55\text{-}60 \text{ }^\circ\text{C}$ , but the pellet production yield was low (i.e. 6% of the feedstock was pelletized). In the last run, triticale was fed when die temperature reached  $76 \text{ }^\circ\text{C}$ , and it stabilized at  $87\text{-}90 \text{ }^\circ\text{C}$  during pelletizing. Afterwards fescue was added, and the die temperature decreased and stayed at about  $75 \text{ }^\circ\text{C}$ . The production yield of triticale and fescue pellets in run-3 reached 39% and 60% respectively. Pictures of produced pellets are shown in Figure 8 and the power consumption for pelletizing fescue is presented in Figure 9..



**Figure 7 : Pellet density of triticale and fescue pellets. Density was determined by weighing the pellet and using a digital caliper measuring the pellet dimensions at least one week after pelletizing.**



**Figure 8 : Triticale pellets (left) and fescue pellets (right) from the bench-scale pelletizer.**



**Figure 9 : Power consumed during the production of fescue pellets.**

### 1.3. Conclusion

A single-pellet press was used to study the pelletizing properties of seven different kinds of biomass (triticale, fescue, sorghum, miscanthus, fresh miscanthus, willow, alfalfa) and to determine the best production conditions to obtain high quality pellets. Results showed that Miscanthus at fresh state was not suitable for pelletizing due to its high moisture content. The quality of the pellet was measured by its density because this ensures a better durability. Densities between 1126 and 1233 kg/m<sup>3</sup> were obtained for the tested biomass samples at an optimum moisture content of 10% and at a die temperature of 90°C. The maximum pellet density that can be achieved is that corresponding to the plant cell wall density (1420-1500 kg/m<sup>3</sup>).

The influence of press channel temperature (also referred as 'die temperature') on the friction during pelletizing was measured for above mentioned biomass except fresh miscanthus. It was found all biomass experienced a sudden slope change when die temperature increased to certain levels. In order to produce mechanically strong pellet with a high density, it is necessary to heat the die above this temperature before pelletizing. Based on the results, sorghum can be pelletized at a die temperature below 60°C, triticale and fescue require temperatures above 60°C and miscanthus, alfalfa and willow require a die temperature over 90°C. When pelletizing triticale and fescue in a bench-scale rotating pellet mill, steady production was found at a die temperature which is in the region where friction decreased with die temperature. In this case pellets with a density of 1135 kg/m<sup>3</sup> were produced for triticale and 1182 kg/m<sup>3</sup> for fescue. These densities are slightly higher but in good agreement with those found using the single pellet press (1126 kg/m<sup>3</sup> and 1178 kg/m<sup>3</sup>).

## 2. Briquetting

### 2.1. Material and Methods

#### 2.1.1. Materials

For briquetting purposes, normally a larger particle size than for pelletizing is acceptable. This is due to the fact that the acceptable particle size corresponds directly with the size and shape of the die channel. Since a normal briquette for industrial purposes usually is around 75mm diameter, the die channel for making such briquettes have an inlet of 81,5mm diameter, and an outlet of for example 68mm diameter. This means that particle sizes of 20mm length (for woody materials) or 30mm (for grassy materials) would normally be suitable.

In this particular case however, the testing materials come from productions, made by test facilities around Europe. This means that the available volume is limited to around 300kg for each material. Our normal test press line is of an industrial scale, an as such has a capacity above 1.000 kg per hour in 75mm diameter. Therefore, a test run with such a small volume, would leave too little time to make changes to the process, according to preliminary results. To account for this, the decision was made to test the material on a smaller machine, making briquettes of 50mm diameter. This machine has a capacity of 200 kg per hour, making it possible to assess briquette quality and production parameters within each test, and make advantageous adaptations, before the material runs out.

The smaller briquette diameter made it necessary to run some of the material through a hammer mill, to achieve an appropriate particle size. The miscanthus and willow were ground in a hammer mill with a 10 mm screen, while the sorghum and triticale were already prepared to a sufficiently small particle size (also around 10mm).

The following material was available in a sufficient scale for briquetting tests :

Species	Suppl.	Region	Harvest	Moisture content (w.b.)	Particle size	Bulk density
Triticale	CENER	Extremadura, Spain	2013	10,2%	10mm	100kg/m <sup>3</sup>
Sorghum	CENER	Spain	2013	10,2%	8mm	155kg/m <sup>3</sup>
Miscanthus	BP	Burgundy, France	2013	15%	10mm (CFN)	107kg/m <sup>3</sup>
Willow	CRL	Retford, Nottinghamshire, UK	2013	12%	10mm (CFN)	125kg/m <sup>3</sup>

### 2.1.2. Briquetting press principle



Figure 10 Briquetting press BP6500

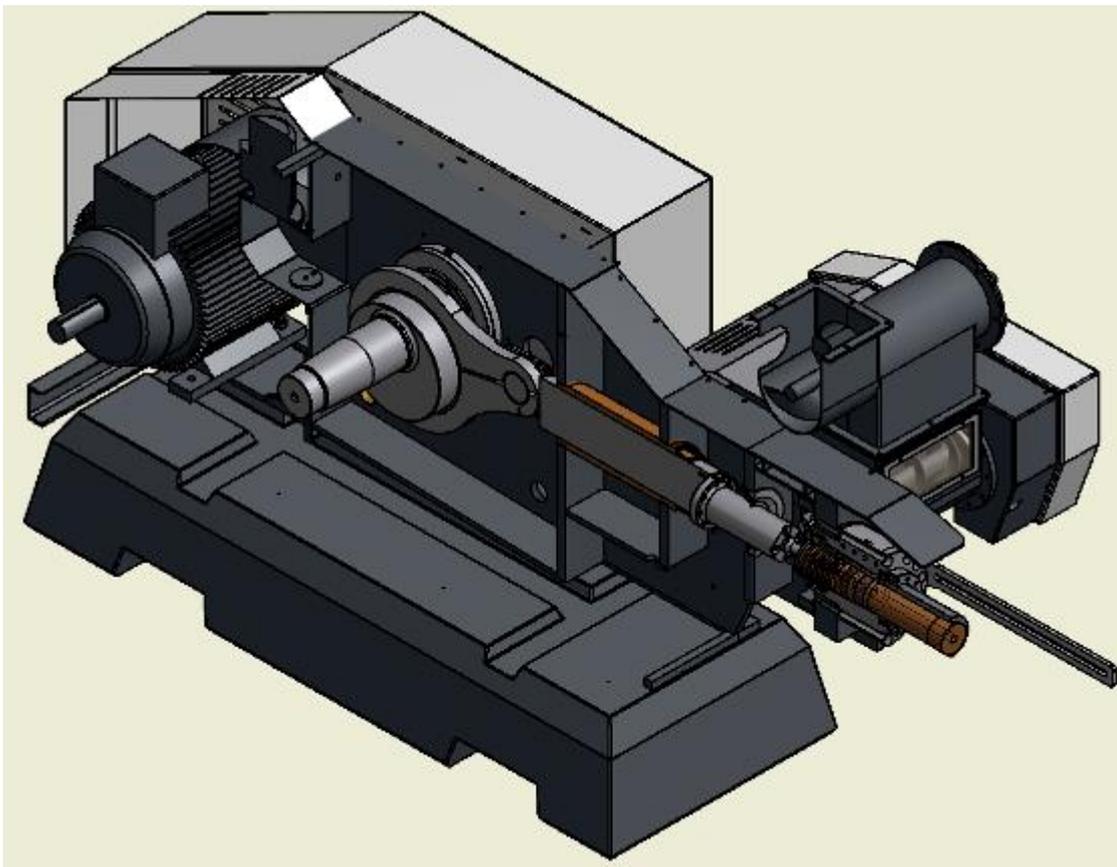


Figure 11 Cut-away of a briquette press

*Working principle of a briquetting press (see figure 11)*

- An eccentric mechanism, operated by a big electrical motor (15-75kW) drives a piston in a reciprocating motion. The eccentric shaft rotates at 270 revolutions per minute.
- Raw material is fed through a frequency controlled feeding screw (not illustrated) into an inlet chamber near the front of the machine.
- Here, a distribution screw separates the material, so that half of the volume goes left, and half goes right.
- Compression screws on both sides of the piston receive the material, and feed it into the compression chamber in front of the piston.
- As the piston moves forward, material is rammed into a conical pipe (main die). The conicity of the die is adapted to the specific raw material, by changing the outlet diameter. After the main die, a cylindrical extension die is placed, with the same diameter as the outlet of the main die.
- For each piston stroke, a new portion of material is compacted against the material already in the die, creating a string, consisting of “discs” of dense, cohesive material. This means the output of the briquette press in reality is a continuous, long briquette, which is gradually pushed into a cooling line by the strokes of the piston (cooling line not illustrated).
- The cooling line is a simple, open conveying pipe, allowing ambient air to cool the briquette string while under mechanical pressure, which makes the string harder and even more cohesive.
- When the string reaches the end of the cooling line, it breaks of by itself in smaller pieces. Length of the pieces can be controlled to be between 50 and 300mm.

The briquette retains its shape because of the combination of several factors :

- High pressure (up to 200 Mpa) is reached each time the piston strikes.
- Heat development occurs (between 70-150°C) because of the friction between the material and die walls, and because the piston delivers kinetic energy as it strikes.

These influences cause the lignin in the material to reach a liquid state, and act as a glue in the briquette. Furthermore, the mechanical actions on the material makes the fibers become entangled, adding to the cohesion.

These factors mean, that by most materials, addition of a binder is not necessary.

## 2.2. Test setup and scope for test

### 2.2.1. Test setup

As mentioned above, the limited volumes of test materials available from the suppliers in this project has forced us to develop and use a smaller machine, than what we normally use for testing. The setup of this machine can be seen in figure 12. The specifications for the machine are :

Main motor :	22kW
Capacity :	200kg/hour
Die size :	40 - 50mm in various conicities
Contents of batch/buffer storage :	400L
Physical dimensions, LxHxW :	2.500x2.500x700mm, weight 2.000 kg.



Figure 12 Lab scale press



**Figure 13 Controls of lab-scale press**

The controls of the lab-scale press are through a touch screen interface, as seen in figure 13. Here, different parameters can be monitored and to a certain degree controlled, such as die temperature, power consumption for main motor, set-point for the output of the press, etc.

### 2.2.2. Scope for test

The main goal of the test is to see if the different materials are suitable for briquetting as a densification technique, and to produce briquette samples for further testing by the other participants within this project. Furthermore, the aim is to determine optimum production parameters, such as die conicity, die temperature, and corresponding power consumption. Changing conicity means altering the ratio between inlet- and outlet cross sectional area of the die (by mounting another die with a different conical shape).

The procedure for each material will be to first find a suitable die size and capacity set point, through a short trial run. Then, these two parameters will be kept, while adjusting the die temperature to two different settings, and record the actual output and power consumption with these two different die temperatures. The two different temperature settings are chosen on each side of the slope change in figure 6. This will give an indication if the sudden slope of the curve for relationship between necessary force and die temperature found during pelletizing, also applies for briquetting.

It is important to note, that it may not be possible to achieve very low temperatures in the setup, where this test takes place. Even though we are actively cooling the die,

experience shows that friction and kinetic energy force the temperature up, often to around 130°C. Cooling consumes energy as well, typically around 1 kWh.

## 2.3. Results and evaluation

### 2.3.1. Results

The spreadsheet below holds the recordings from the test runs. Figure 14 shows the briquette quality.

Species	Die temp	Die size	Capacity set point	Capacity measured	Power consumption, main motor	Briquette density (kg/m <sup>3</sup> )
Triticale	95°C	Main 50 B	150kg/h	120kg/h	26A = 15kW	1,085
	105°C	Ext 50B400		120kg/h	23A = 13kW	1,018
Sorghum	100°C	Main 50 A Ext 50A400	200kg/h	120kg/h	38A = 21kW	1,097
Miscanthus	80°C	Main 50 B Ext 50B250	150kg/h	102kg/h	23A = 13kW	0,969
	100°C			102kg/h	21A = 12kW	0,931
Willow	80°C	Main 50 B Ext 50B250	150kg/h	126kg/h	35A = 19kW	1,152
	100°C			126kg/h	31A = 17kW	1,133



Figure 14 Briquette samples from the trial runs

### **2.3.2. Evaluation**

In general, the temperatures chosen, were on a basis of what is practically possible, with our machine. Typically, low temperatures are not reachable, due to friction generating a lot of heat in the die.

Triticale makes a good briquette with a reasonable power consumption. Power consumption decreases with a rise in die temperature. This is clearly due to a loss of friction in the die, which can be seen, since the density decreases.

As seen in the spreadsheet, Sorghum is a difficult species to briquette. In spite of having the highest bulk density (smallest particle size), a die with a higher conicity was needed, and even though power consumption was very high, it did not produce the highest density or capacity. With the amount of raw material available, we were not able to produce with more than one temperature setting, since some material was lost, attempting to achieve steady production.

Miscanthus generates a rather low friction, meaning power consumption and density is low.

Willow generates a good friction and thus high density in the die.

### **2.4. Conclusion**

From the trials, it was seen that densities between 931 and 1152 kg/m<sup>3</sup> were achieved for the biomass used (triticale, miscanthus, sorghum and willow) at different die temperatures. The highest density achieved in briquettes for each kind of biomass was 1085 kg/m<sup>3</sup> for triticale, 969 kg/m<sup>3</sup> for miscanthus, 1097 kg/m<sup>3</sup> for sorghum and 1152 kg/m<sup>3</sup> for willow. All these densities were obtained at the lowest of the considered die temperatures. So, lower temperatures increase the friction and backpressure that result in a higher density briquette. However higher die temperatures require lower power consumption. Depending on the location of the briquetting plant, in relation to the place where the briquettes are consumed, transport costs may be of a larger importance than power consumption for production. A calculation for each specific project, comparing extra production costs to saved logistic costs (due to a higher density) and finding the optimum, would be necessary.

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