

## INFLUENCE OF INCREASING SHARES OF MISCANTHUS ON PHYSICAL AND MECHANICAL PROPERTIES OF PELLETS PRODUCED IN AN INDUSTRIAL SOFTWOOD PELLETS PLANT

### Part 1: Material & Method

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### Abstract

It has been possible to pelletize Wood-Miscanthus mixtures (12.5 – 25 & 50%) without modifying production process settings of a softwood pellets plant. Pure Miscanthus material tested in the same conditions has led to unstable production, mainly explained by hammer-mill overfeeding. The unstable production has been identified as the main responsible factor of the low quality of pellets produced with pure Miscanthus for these trials. The pellets produced were tested in a 25 kW boiler and compared with agro-pellets of various origins: winter barley straw, rapeseed straw, reed, old hay, Miscanthus, & wood pellets. During the trials, O<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, SO<sub>2</sub> and NO<sub>x</sub> emissions were measured. The flue gas chlorine content was also determined and the data were linked to the specifications of the fuels used. Trials showed that pure agro-pellets do not reach the combustion quality of wood or wood-Miscanthus mixes.

**Keywords:** pellets, miscanthus, solid biofuels, wood, mixture, physical properties, combustion, emissions, straw, agro-pellets

### 1. Introduction

Wood-burning technology is well established, especially for pellets. The market for pellets is growing rapidly as well as industrial and non industrial scales; and the European demand may outstrip the raw material resources without importing as a consequence. It is an important issue to identify new biomass resources.

Another consideration is that farmers are looking for new outlets, notably by producing crops for energy. However, agricultural products are reputedly not ideal for fuel use, as several studies have shown [1, 2, 3, 4].

Trials have been setup already to study pure agro pellets production, especially Miscanthus. These studies have demonstrated this raw material does not lead to specific problems regarding pelletizing [5, 2]. Fuel pellets could then be considered as an interesting output for this agricultural diversification. But the effects on combustion of raw material from agricultural origin are known. On one hand these products lead to slagging and fouling due to ash content and composition. On the other hand the chlorine and sulfur content lead to toxic and corrosive emissions [3, 6, 7]. Consequently, it has been suggested the use of additives to mix with agricultural raw materials, as limestone for instance [8]. Indeed some additives are known to improve the combustion behavior, especially the ash melting. Another possible way to improve the use of these difficult raw materials is to mix it with wood, in order to dilute the impact on combustion.

The first objective is here to evaluate the influence of the *Miscanthus* proportion on the physical and mechanical properties of the produced pellets. The measured values are compared to the limit fixed by the European standard EN 14961 (parts 1 - 2 – 6) [9,10, 11], with following question to answer: "Up to what proportion may *Miscanthus* be added to wood without exceeding limits given by these documents and changes in the process?" Trials have been performed on five "wood – *Miscanthus* mixtures" in an industrial pellets production line. Additionally, this study has for objectives to describe the influence of *Miscanthus* proportion on variability of the assessed properties.

The second goal is then to characterize the produced pellets combustion behavior and to compare it with pure wood and pellets made of other agricultural raw materials. It therefore appears useful to characterize emissions (especially NO<sub>x</sub>, SO<sub>2</sub> and Cl) from specific resources and to link it to the physical and chemical properties of agro-fuels that affect their combustion emissions. The tests described here were conducted in a small scale boiler (25 kW) in order to be representative of domestic use or closed-circuit use by farmers (likely to be medium or low power).

## 2. Material & Methods

### 2.1. Pelletizing trials on wood-*Miscanthus* mixes

#### *a. Raw material mixtures preparation*

Five mixtures have been prepared in sufficient amount to insure at least one hour pellets production. About one ton raw material has been prepared for each mixture. *Miscanthus* proportions were (in mass) 0%, 12.5%, 25%, 50% and 100% - pure *Miscanthus*. Mixture were prepared prior to the experiments and stored in big bags. For the trials, the big bag was unloaded directly in the production chain, just upstream the hammermilling step.

#### *b. Pellets production chain*

Pelletizing trials have been performed on a common softwood pellets production chain. The process is characterized by following steps: raw material delivery, dryer, material mixing, hammermill, hopper, screw, pellet press (6 mm diameter, 45 mm length), cooler, fines sieving, packing.

Production parameters were kept unchanged for the five tested mixtures. Not any problem had to be noticed during processing, except for pure *Miscanthus* material. In this case, overfeeding of the hammermill has been observed, which induced unstable press feeding. The hammermill overfeeding may be explained by a higher coarseness of *Miscanthus* particle size distribution, compared to other mixtures.

#### *c. Sampling*

Raw material samples were taken just upstream the pellet press. In order to reduce the impact of sampling schedule and to avoid periodicity effects, samples were made of six subsamples taken within two minutes. Ten raw material samples were taken for each mixture at intervals of 6 minutes.

Ten pellets samples were taken downstream the cooler, within one hour at fix time intervals. Pellets (and fines) samples were made of two successive 6 liters subsamples.

#### *d. Physical, mechanical and chemical properties*

All the used procedures and methods to determine the raw material and pellets properties are the ones listed in the European standard EN 14961 [9]. Samples were

divided using riffle and rotary dividers.

The raw material mixtures were characterized regarding the ash content, moisture content and particle size distribution after milling. Minor and major elements have been measured for pure wood and Miscanthus only. Chemical content of mixtures were calculated based on pure raw material content and composition.

Prior to properties measurements, pellets and fines are separated and the fine content is stated. Additionally the fines ash content is measured for each mixture.

Measurements on pellets were performed for: durability (DU in %), particle density (PD in g/cm<sup>3</sup>), length and diameter (in mm), gross calorific value (GCV in MJ/kg), moisture content (MC in %) and ash content in %.

## 2.2 Combustion trials

### a. Pellets raw material

Beside the mixed wood & Miscanthus pellets produced through the production experiments (2.1), 4 other agricultural crops pellets (produced by a prototype pellets press) have been used for combustion trials. Additional raw materials were: Reed, old Hay, winter Barley straw & rapeseed straw. Table 1 shows the various fuels tested and the name used below to denote each fuel

Table 1. Raw materials tested and replicates (n).

<i>Tested Fuel</i>	<i>Name</i>	<i>n</i>
0% Miscanthus / 100% wood	wood	2
12.5% Miscanthus/87.5% wood	misc 12.5	2
25% Miscanthus / 75% wood	misc 25	2
50% Miscanthus / 50% wood	misc 50	2
100% Miscanthus / 0% wood	Miscanthus	1
Reed	reed	1
Old hay	hay	1
Winter barley straw	winter barley	1
Rapeseed straw	rapeseed	1

### b. Pellets properties

The tested pellets properties are shown in Table 2. The pellets properties were compared to those specified in the EN 14961-2 and prEN 14961-6 (wood and non woody biomass pellets standards). It appears most of the components comply with standards specifications except Cl and S content. Durability varied from 90% to 98% according to the production method. Moisture content leveled around 10% except for Miscanthus, reed and hay, where it was higher. Agro-pellets ash content was higher than wood pellets. Lastly, fines accounted for no more than 1% of the material except for 100% Miscanthus.

All the produced and tested pellets complied with the EN 14961-1 standard; regarding the maximum length proportion (Less than 5% of the pellets were between 40 and 45 mm long).

The fines content (particles under 3.15 mm) ranged from 3.2 up to 9.8% for pellets made of agricultural raw materials. Pellets containing wood never exceed 2% fine content.

In addition, it has to be noticed the high share of small and broken pellets (length small than the 6 mm diameter). Pellets made of agricultural residues have a proportion of such particle ranging from 20 to 65%. Pellets containing wood are under 5% regarding this property.

No conclusion may be drawn regarding the influence of the raw material origin on the length distribution of pellets. Indeed most of the agricultural pellets were produced by a prototype press while Miscanthus pellets were produced by an industrial press having settings prepared for wood. Nevertheless the length distribution of the produced pellets has been considered has acceptable to be used in combustion trials.

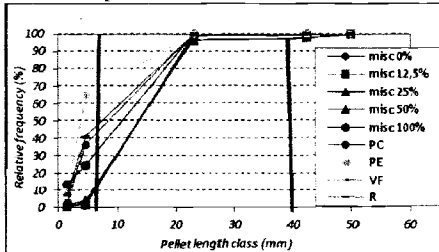


Figure 1. Length distribution of pellets used for combustion tests. Miscanthus shares in the wood Miscanthus pellets are stated in percentage after the misc abbreviation, PC: rapeseed straw, PE: winter barley straw, VF: old hay, R: reed c. Boiler

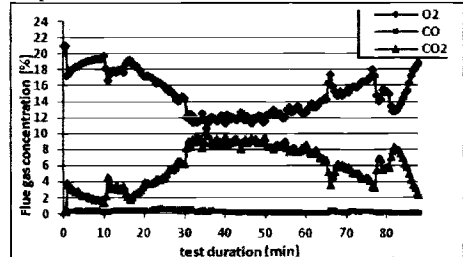


Figure 2. O<sub>2</sub>, CO and CO<sub>2</sub> concentration over time (min) in the Winter Barley straw test.

An HS Tarm Multi-Heat multi-fuel boiler was used for the tests. This model is suitable for burning (agro) pellets, cereals and wood chips. The nominal power of the boiler is 25 kW. The nominal efficiency is 86-87%. It is stoked automatically via an auger. Ash is removed manually. Between 7 and 10 kg of pellets were burned per trial (see table 3 for test sample size and burning conditions).

#### d. Measuring Equipment

The installation diagram of the equipment used for these trials is provided in Figure 6. Two measuring lines were set up for the purpose of this study.

The first line being dedicated to SO<sub>2</sub>, CO, CH<sub>4</sub>, NO, CO<sub>2</sub> and O<sub>2</sub> concentration measurements in the flue gas. The following equipment was used:

- ☞ Vacuum pump: to keep an isokinetic sampling rate throughout the trial.
  - ☞ Peltier cooler: to condensate water content prior to gas analysis.
  - ☞ Peristaltic pump: to remove condensates to prevent liquid entering the gas analyser.
  - ☞ Filter paper: back-up filter to keep dust out of the gas analyser.
  - ☞ Rotameter: to control the sampling rate. The water circulation rate in the boiler is thus known and averaged 368 l/h.
  - ☞ Gas analysers: to analyse the gas SO<sub>2</sub>, CO, CH<sub>4</sub>, NO, CO<sub>2</sub> and O<sub>2</sub> content. The analysers first have to be calibrated using a standard gas.
    - ✦ Magnos 6G oxygen analyser (Hartmann & Braun): for continuous measurement of the oxygen content of gas mixes in ranges from 0 to 100% O<sub>2</sub> by volume.
    - ✦ Uras 14 infrared absorption analyser (Hartmann & Braun): the Uras 14 continuously measures SO<sub>2</sub>, CO, CH<sub>4</sub> and NO in gas mixes.
    - ✦ Fuji infrared gas analyser: this device continuously measures the CO and CO<sub>2</sub> concentration in a gas in ranges from 0 to 10% by volume (CO) and 0 to 20% by volume (CO<sub>2</sub>).
  - ☞ Data Logger (Testo 350 454): used to record the data & transfer it to the computer
- The oxygen concentration during the trials varied from 11.3% to 14.5%. CO was the

most prevalent element, amounting to nearly 2,300 mg/m<sup>3</sup> at 13% O<sub>2</sub> in the case of winter barley straw whereas the Belgian standard (Decree published on 12/10/2010 [2]) is 5,000 mg/m<sup>3</sup> in 2011, 3000 mg/Nm<sup>3</sup> in 2012-2013 and 1,500 mg/Nm<sup>3</sup> from 2013 to 2016.

Figure 2 shows the O<sub>2</sub>, CO and CO<sub>2</sub> concentration curve during the winter barley straw pellet test.

The second measuring line was dedicated to chlorine concentration determination in the flue gas. The following equipment was used for this purpose:

↳ Vacuum pump: to maintain an isokinetic sampling rate throughout the trial.

↳ Flow meter: to control the sampling rate.

↳ Gas washing bottles (4): used to trap chloride ions in flue gas by passing the gas through water in order to measure the concentrations according to the ISO 10304 standard. Chloride concentration resulting from combustion was determined by relating the gas flow rate through the washing bottles to the flue gas flow rate.

Parallel to the washing bottle set a Pitot tube was installed to measure the flow rate of the sampled flue gas. The ΔP (dynamic pressure) measured by the Pitot tube averaged 0.0095 hPa.

Flue gas & water (in & out) temperature were measured by thermocouples and PT100 sensors. The IN: water temperature was between 21 and 39°C and the OUT: water temperature ranged from 35 to 57°C. The desired value is a boiler IN / OUT temperature difference (δT) of 20°C. A maximum variation of 12°C in relation to the desired value was observed during the tests. The flue gas temperature varied between 106°C and 122°C (Table 3).



Figure 3. Type 1



Figure 4. Type 2



Figure 5. Type 3

The slag was classified in three different types on a qualitative basis (visual). The first case, called Type 1 (Figure 3) contained almost no slag. In the second case, Type 2, (Figure 4) some slag formation was observed. Lastly, the third case, Type 3, (Figure 5) is in between type 1 & 2 and contains friable slag designated as 'pre-slag'. This was removed from the core of the boiler together with the ash and therefore caused no problems.

Table 2. Pellet composition and element content with regard to CEN/TS 14961 2 & 6 - B: wood.

*M 12.5: misc 12.5; M 25: misc 25; M: misc; R: reed; VF: hay; PE: winter barley;*

*PC: oilseed rape - Type 1: Ashes; Type 2: slag; Type 3: pre-slag*

Material	B	M 12.5	M 25	M 50	M	R	VF	PE	PC
Moisture (%)	9.5	10.0	9.6	8.1	20.4	21.7	11.7	12.5	13.1
Ash content (%)	0.9	1	1.2	1.7	2.6	5.5	6.1	5.2	5.8
Durability (%)	98.0	98.0	97.9	98.0	90.5	91.3	95.2	93.9	89.8
Net density (g/cm <sup>3</sup> )	1.3	1.3	1.3	1.3	1.3	0.8	1.2	0.9	1.2
Fines (%)	0.7	0.7	1.2	1.3	14.8	0.9	0.3	1.0	0.9
GCV (MJ/kg)	20.23	20.12	20.01	19.79	19.36	18.84	18.70	18.68	18.81
N (%)	<u>0.08</u>	<u>0.08</u>	<u>0.09</u>	<u>0.11</u>	<u>0.14</u>	<u>1.02</u>	<u>1.21</u>	<u>0.57</u>	<u>0.69</u>
C (%)*	51	50.5	49	48	47	46	46	47	48

Material	B	M 12.5	M 25	M 50	M	R	VF	PE	PC
H (%)*	6.3	6.3	6.2	6.15	6.1	5.8	5.9	6.0	6.0
O (%)*	42	42	42	42	42	42	40	41	41
NCV (MJ/kg)	16.86	16.86	16.81	16.91	16.45	13.98	15.23	14.94	14.96
Cl (mg/kg)	< 100	< 100	< 100	< 100	< 100	2600	5700	3300	1500
S (mg/kg)	< 500	333	465.5	731	1262	1200	1400	970	2800
Na (mg/kg)	21	33.3	45.63	70.25	119.5	300	180	340	160
Si (mg/kg)	125	189	253.75	382.5	640	1100	7200	8200	2300
Ca (mg/kg)	1426	1408	1389.5	1353.5	1282	3600	4600	4800	1100
K (mg/kg)	387	459	531.88	676.75	966.5	1000	1700	1500	1100
Mg (mg/kg)	181.5	191	201	220.5	259.5	800	1300	600	790
Cd (mg/kg)	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.4	< 0.4	< 0.4	< 0.4
Cr (mg/kg)	< 8	< 8	< 8	< 8	< 8	6.3	2.8	5.0	3.1
Cu (mg/kg)	< 5	< 5	< 5	< 5	< 5	5.3	4.2	3.7	6.6
Zn (mg/kg)	< 100	< 100	< 100	< 100	< 100	32	24	8.1	12
As (mg/kg)	< 0.8	< 0.8	< 0.8	< 0.8	< 0.8	0.38	0.3	0.26	0.21
Mg (mg/kg)	< 10	< 10	< 10	< 10	< 10	1.3	1.0	0.7	0.6
Hg (mg/kg)	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Ni (mg/kg)	< 10	< 10	< 10	< 10	< 10	12	1.8	2.5	4.3
Slag type	1	3	3	3	2	3	3	3	3

\*: Characteristic value according to standard EN 14961 – 1, i.e. not measured in trial

- Val:** Value conforming to grade WP A1 according to CEN/TS 14961 – 2  
**Val:** Value not conforming to grade WP A1 according to CEN/TS 14961 – 2  
**Val:** Value not conforming to any of the CEN/TS 14961 – 6 quality classes  
**Val:** Value conforming to CEN/TS 14961 – 6  
**Val:** Value not conforming to CEN/TS 14961 – 6

Table 3. Test Characteristics.

Average flue gas temperature,  $t^\circ$  in and  $t^\circ$  out ( $t^\circ$  in: boiler inlet water temperature;  $t^\circ$  out: boiler outlet water temperature), Flue gas flow rate ( $Q$ ), Chlorinated water volumes in washing bottles, Quantity of fuel burned (kg)

Substrate	Flue gas $t^\circ$ (°C)	IN water $t^\circ$ (°C)	OUT water $t^\circ$ (°C)	$\delta T$ (°C)	$Q$ (Nm <sup>3</sup> /s)	Total volume (l)	Qty pellets burned (kg)
wood	110.23	20.74	53.58	32.84	0.0176	0.847	10.000
misc 12.5	110.23	35.79	54.93	19.13	0.0154	0.914	7.648
misc 25	108.90	39.06	57.48	18.42	0.0109	0.935	8.787
misc 50	119.84	34.09	56.70	22.61	0.0120	0.900	8.202
misc 100	129.46	27.49	53.65	26.17	0.0132	0.850	9.968
reed	113.28	19.09	45.81	26.72	0.0175	0.856	10.080
hay	99.56	16.58	38.75	22.17	0.0159	0.832	10.002
winter barley	113.88	17.99	44.40	26.41	0.0168	0.822	9.997
rapeseed	117.17	20.42	47.76	27.34	0.0162	0.826	10.080

#### *e. Data recording*

All the above mentioned data were recorded at a 30s time interval and recorded by the data acquisition computer. The data were averaged when the boiler was operating at full speed, i.e. for 30 minutes. The average test duration was 1 hour 41 minutes. For each fuel, ash content was determined according to the Belgian standard 'Solid Biofuels - NBN Ash Content Determination' CENT/TS 14775 (January 2005).

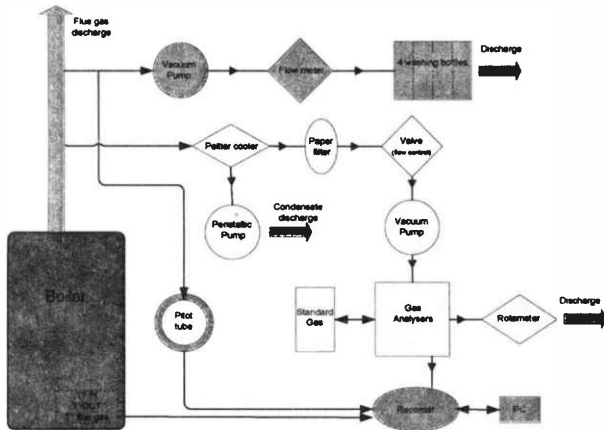


Figure 4. Combustion test equipment layout.

f. Reference Concentration

Following data acquisition the element concentration (NO, CH<sub>4</sub>, CO, etc.) was expressed in ppm or mg/m<sup>3</sup>. The results were reduced to a 13% oxygen content in mg/m<sup>3</sup> (Table 4).

The conversion formula used is shown below.

$$[\text{mg/m}^3] = [\text{ppm}] * \alpha$$

where  $\alpha = 0.717$  for CH<sub>4</sub>  
 $\alpha = 1.25$  for CO  
 $\alpha = 1.44$  for NO

$$[\text{mg/m}^3]_{\text{ref to 13\%O}_2} = [\text{mg/m}^3] * \frac{(21 - 13)}{(21 - [\text{O}_2])}$$

The SO<sub>2</sub> and CH<sub>4</sub> concentrations were the lowest between 0 and 64 mg/m<sup>3</sup>. NO concentrations varied from 50 to 290 mg/m<sup>3</sup>. Lastly, the CO concentrations ranged from 30 to 2,300 mg/m<sup>3</sup>.

The quantities of SO<sub>2</sub>, CH<sub>4</sub>, CO and NO emitted per kg of pellets burned were calculated (g/kg) from the mg/m<sup>3</sup> concentrations, flue gas volume (Nm<sup>3</sup>) and quantity of pellets burned (kg).

Table 4: SO<sub>2</sub>, CH<sub>4</sub>, CO and NO concentration at 13% O<sub>2</sub>

Substrate	SO <sub>2</sub>	CH <sub>4</sub>	CO	NO
	Ref. conc. 13% O <sub>2</sub> (mg/m <sup>3</sup> )			
wood	14.06	0.00	189.98	144.09
misc 12.5	12.81	29.09	1193.24	51.18
misc 25	10.16	13.18	799.95	74.56
misc 50	13.99	1.94	143.16	107.99
Miscanthus	47.44	0.10	30.38	155.52
reed	57.05	2.32	1140.80	311.68
hay	20.15	5.05	1439.24	318.88
winter barley	36.88	16.42	2271.60	176.95
oilseed rape	37.33	3.31	417.47	253.41

*g. Combustion Efficiency*

The combustion efficiency was 90% overall (Table 5). It was calculated by a method derived from European standard EN 15378. The formula is shown below. Factors A and B in the formula refer to the fuels. For the considered pellets, the values of 0.765 and 0.000 have been considered for A and B, respectively.

$$\text{Eff} = 100 - \text{losses}$$

$$\text{With losses} = (t^{\circ}_{\text{flue gas}} - t^{\circ}_{\text{oxidizer}}) * ($$

A, B = fuel-specific factors

**Table 5. Efficiency of different agro-pellets.**

<i>Substrate</i>	<i>Efficiency</i> %	<i>Substrate</i>	<i>Efficiency</i> %
wood	92.9	reed	91.7
misc 12.5	89.1	hay	91.3
misc 25	91.2	winter barley	89.1
misc 50	84.6	oilseed rape	91.4
Miscanthus	89.5		

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