



## Converting Organic Matter from European Urban and Natural Areas into Storable Bio-Energy



## Analysis of mass and energy fluxes



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## 1 Target of the study

This report describes substantial (parent) materials delivered by three partners of the COMBINE project. The basic mass and energy fluxes of the different biomass types during the conversion into biofuels are presented. The knowledge about the material flows during the biomass processing is essential for the subsequent plant conception and quantification of outputs, such as solid fuel and biogas. The further energetic evaluation of the solid fuel and measurement of methane yields are crucial for the environmental impact assessment. Thus, data presented in this report are the basis for subsequent studies. Additionally, they are basic figures about the potential of the IFBB process for bioenergy recovery as they allow to compare the IFBB product (press cake) with other solid fuels, e. g. wood and coal. We calculated the heating value for all materials comparing parent material with the press cake. Usually the briquettes produced from the press cake have a water content of 15 %. Thus, we are presenting the heating value referring to dry fuel and the heating value referring to fuel with 15 % water content.

Additionally, we present the ash content and mineral composition of harvested materials. The ash content of semi-natural grassland is high in general, which lowers its heating value and increases the maintenance effort of the combustion unit. Additionally, the ash forming minerals are causing problems in combustion as they regularly lead to corrosion and lower the ash melting point, whereby slagging is enhanced. Hartmann (2009) give the following influences of some minerals:

- K      ash melting behaviour, corrosion, particle emission
- Mg     ash melting behaviour, particle emission, bonding of contaminants
- Ca     ash melting behaviour, particle emission, bonding of contaminants
- S      SO<sub>x</sub>-emissions, corrosion, particle emission
- Cl     emissions of polyhalogenated organic compounds, corrosion, particle emission

Thus, it is favourable to reduce their concentration in the solid fuel, which is possible by applying the IFBB technique. In this study we investigated the mass flows during the process, as well as concentrations in parent materials from three partner regions and in the produced solid fuels.

## 2 Methodology

This chapter describes the processing of round bales from the COMBINE partner sites representing a variety of grassland type. It took place at the large scale plant of PP 3 in Baden-Baden. Three biomass types were harvested and ensiled in 2013 at the partner sites, stored as round bales and afterwards transported to Baden-Baden.

Furthermore, this part presents the methodology of scientific investigations of the biomass and fuel parameters.

### 2.1 Processing of grassland into biofuel

#### 2.1.1 Pre-treatment

First step of processing was the dissolving of the compacted biomass of the round bale (Fig. 1, 2, 3). After removal of the foil, the bale was put into a twin-shaft shredder by a wheel loader with the aim to dissolve the bale. From this shredder, the silage was directly transferred into the dosing feeder of the defibrator with rotating chains. By the defibrator the silage containing high fractions of long fibres was transferred into a more homogeneous material with shorter fibre length, which is essential for its handling in the subsequent process steps (Fig. 4).



Fig.1: Dissolving of the round bales



Fig. 2: Round bale



Fig. 3: Silage after dissolving



Fig. 4: Silage after defibrator

### 2.1.2 Hydrothermal conditioning

The crushed silage was overloaded into the dosing feeder of the hydrothermal conditioning unit (Fig. 5). It consists of a round 25 m<sup>3</sup> tank (Fig. 6). In this tank, silage was mashed with water in a ratio of 1:15 (dry matter: water) for 40 min with permanent stirring (Fig. 7). After this time, the mash was pumped through a sieve bottom into the mash storage tank (Fig. 8). Fibrous material that did not pass through the sieve was removed by an automatic rake and dewatered by a hydraulic press (Fig. 9, 10).



Fig. 5: Loading of the dosing feeder



Fig. 6: Mashing tank



Fig. 7: Silage during conditioning



Fig. 8: Mash storage tank



Fig. 9: Removal of remaining fibres



Fig. 10: Pressed fibres

### 2.1.3 Separation, drying of the press cake and briquetting

The mash was pumped into the separator and the resulting liquid (Fig. 11) was stored before anaerobic digestion. The press cake (Fig. 12) as well as the pressed fibres from the mashing tank were transferred into the dryer, where the biomass was dried up to a dry matter content of around 90% (Fig. 13). The dried press cake was loaded into the feed tank for the briquetting unit before it was continuously compacted by the mechanical briquette press (Fig. 14, 15, 16).



Fig. 11: Press fluid



Fig. 12: Press cake



Fig. 13: Press cake after drying



Fig. 14: Loading of the briquetting unit



Fig. 15: Pressing cylinder



Fig. 16: Briquettes

After opening and chopping of the bales, samples of the silage were taken in triplicates. Samples of press cake were also taken in triplicates after mashing, pressing and drying (Fig. 17). Chemical analysis of silage and press cake samples was identical. Additionally, samples of press fluid were taken in triplicates (Fig. 18).

The following table (Tab. 1) gives an overview of all samples and parameters, which have been analysed:

Tab. 1: Samples of three biomass types as processed with IFBB technique.

| Biomass type | Number of biomass types | Replicates | Total number of investigated samples |
|--------------|-------------------------|------------|--------------------------------------|
| Silage       | 3                       | 3          | 9                                    |
| Press cake   | 3                       | 3          | 9                                    |
| Press fluid  | 3                       | 3          | 9                                    |
| Sum          |                         |            | 27                                   |

In order to determine the content of dry matter (DM), the samples were dried at 105°C for 48 h in a drying oven to lose water (Fig. 19). To determine volatile solids (VS) and ash content dried samples were incinerated in a muffle furnace on 550°C. The mass loss during incineration is proportional to organic constituents (VS) and the remaining material is ash. Another sample (also in triplicates) was taken and dried at 65°C for subsequent chemical analysis (carbon, hydrogen, nitrogen). Samples were grinded with a cutting mill (SM 1, Retsch) to 5 mm and subsequently with a sample mill (1093 Cyclotec, Foss) to pass a 1 mm sieve. C, H, N analysis was conducted using an elemental analyser (Vario MAX CHN Elementar Analysensysteme GmbH). Mineral constituents besides C, H, N have been measured by X-ray fluorescence spectroscopy by an accredited laboratory.

The liquid (press fluid) was stored in a freezer at -20°C and investigated in the biogas laboratory (Fig. 20) of Kassel University for the methane yield, and also for its dry matter and VS (all parameters analysed in triplicates). To measure the methane yield, 4 kg of press fluid were mixed with 12 kg inoculum in polyethylene containers for a batch experiment. Containers were kept at a steady temperature of 37°C. The mixture was stirred every hour for 15 min. by electrical stirrers. Biogas was collected in aluminium gas containers and the amount was measured for 14 days with a wet drum gas meter (TG1, Ritter Ltd.). Methane concentrations in biogas were measured with an infrared gas analyser (GS IRM 100). In the calculation of methane yields the biogas and methane production of the inoculum was monitored and subtracted from the methane yields of the samples. To give results in  $\text{L}_\text{N} \text{ kg}^{-1}$  VS values for VS in press fluid were gained by measuring the mass loss during incineration of material that was beforehand dried at 105°C.



Fig. 17: Sampling of press cake



Fig. 18: Sampling of press fluid



Fig. 19: Drying oven with samples



Fig. 20: Biogas laboratory

Mass flows have been calculated according to the formula:

$$MF = \frac{\text{percentage of } DM_{\text{press cake}} \text{ in mash} * DM_{\text{press cake}} * Content_x \text{ in press cake}}{DM_{\text{mash}} * Content_x \text{ in silage}}$$

where x is the measured element.

The higher heating value (HHV) was calculated from C, H and N according to the formula (Friedl et al., 2005):

$$HHV \left[ \frac{MJ}{kg \ DM} \right] = 3.55 \ C^2 - 232 \ C - 2230 \ H + 51.2 \ C * H + 131 \ N + 20,600$$

From the HHV the lower heating value (LHV) was calculated using the formula

$$LHV \left[ \frac{MJ}{kg \ DM} \right] = HHV - \left( 8.937 * \frac{H \% \ DM}{100} \right) * 2.2$$

where 8.937 is the mass share of hydrogen in water molecule and 2.2 is enthalpy of vaporisation.

Our briquettes (solid fuel) have a water content of about 15 %, which lowers the heating value. Thus, we calculated the LHV with a water content of 15 % ( $LHV_{15\%wt}$ ) according to the formula (Hartmann 2009):

$$LHV_{wt} = \frac{LHV_{water\ free} (100 - w) - 2.443 w}{100}$$

where w is water content in %.

Mean values for each partner and analysis were calculated from replicates to present a clear picture of results.

### 3 Results

#### 3.1 Mass flows of dry matter concentrations and organic constituents

The dry matter content of the silage, which had been harvested in three different regions, was varying between 27.32 and 66.01 % fresh matter (Tab. 2). This high range is known from grass cut from nature conservation areas due to different biotopes and cutting dates (Kutschmitt, 2009). For the IFBB approach late cut material is favourable and water content should not lie below 25% approximately, as material with lower water content clogs the machinery.

The press cake was produced by two different kinds of pressing technique: a screw press and a screen compactor. In most cases, the resulting press cake had similar DM contents. Only for biomass from Wales, the screw press led to significant higher DM contents. The DM content of the press cake after drying was in all samples but the Belgian ones above 90% FM. For the Belgian samples a DM content of 88.75 % FM was measured.

The volatile solids in the silage were constantly slightly above 85 % of DM and in the press cake about 95 % of DM. This is in accordance to results of former studies (Hensgen et al., 2012). In the press fluid the content of VS had a mean value of about 79 % DM.

The mean mass flow of VS into the PC was about 54 % DM with a range from 45.99 to 60.92 % DM. Bühl et al. (2012) expect a mass flow of VS about 82 % DM, thus the mass flow of volatile solids into the press fluid is lower in this study.

Tab. 2: Dry matter content (DM) in silage, press fluid and press cake produced by screw press, press cake produced by screen compactor and press cake after drying in % of fresh matter (FM). Concentrations of volatile solids (VS) in the different fractions and VS mass flow into press cake are given.

|  | Belgium | France | Wales | Mean  |
|--|---------|--------|-------|-------|
| DM silage (% FM)                           | 66.01   | 27.32  | 38.42 | 43.92 |
| DM press cake using screw press (% FM)     | 28.20   | 29.43  | 32.46 | 30.03 |
| DM press cake using screen compactor(% FM) | 27.69   | 30.08  | 21.11 | 26.29 |
| DM press fluid (% FM)                      | 1.17    | 2.19   | 1.91  | 1.754 |
| DM after drying (% FM)                     | 88.75   | 97.84  | 92.71 | 93.1  |
| VS silage (% DM)                           | 92.60   | 86.04  | 92.93 | 90.52 |
| VS press cake (% DM)                       | 96.12   | 95.60  | 96.46 | 96.06 |
| VS press fluid (%DM)                       | 80.19   | 77.96  | 78.41 | 78.85 |
| Mass flow of VS in press cake (%)          | 60.92   | 45.99  | 55.62 | 54.18 |

### 3.2 Mass flows of mineral compounds and ash

The ash content of silage varied between 7.07 and 13.96 % DM (Tab. 3). The lower values (Wales and Belgium) correspond with findings of Hensgen et al. (2012), who detected mean ash contents between 5 and 9.4 % DM in semi-natural grasslands. However, due to sampling technique (e.g. harvester for roadside verges) the ash content can easily increase due to soil adherence. With mass flows from 20.48 to 34.11 % into the press cake we observed an ash content in the press cake varying between 3.54 and 4.4 % DM. In comparison to the findings of Hensgen et al. (2012), both, mass flow and ash content in press cake, were lower causing a higher quality of the solid fuel. Differences might be due to the different mashing and pressing technique. The industrial technique used at the sewage water plant in Baden-Baden and for this study seems to lower the ash content more effectively than the demonstration unit used by Hensgen et al. (2012).

Ash consists of several elements including Al, Ca, Fe, K, Mg, Na, P, Si, Ti and As, Ba, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Tl, V, Zn (Baernthaler et al., 2006). Cl, N and S can be particularly detrimental in combustion, Ca, K, Mg and Na are mainly involved in slagging and P is a main ash-forming element. Therefore, in Tab. 3 the concentration of these elements and additionally some heavy metals are presented.

All elements had a lower concentration in the press cake than in the silage for all samples. The levels of, K, P, Na and S concentrations in silage and press cake were similar to former investigated semi-natural grassland samples (Hensgen et al., 2012). In contrast, concentrations of Ca, Cl and Mg were lower than in the mentioned study, in both, silage and press cake.

The difference in concentrations between silage and press cake is reflected by low mean mass flows into the press cake of 6.37 % of Cl and 7.66 % of K. For P, Mg and S mean mass flows of 14.60, 19.09 and 22.26 % were detected, respectively. The mass flow of N into the press cake was 30.61 %. These values are lower than mass flows determined in former studies on semi-natural grassland (Hensgen et al., 2012), which is probably due to the advanced technique used. The pattern, however, is similar, thus Cl and K are most leachable because of their high water solubility and their low chemical bonding in the plant cells, whereas N is less leachable. Nevertheless, we achieve N concentration levels in the press cake (0.78 % DM) that are close to the guiding concentration given by Obernberger et al. (2006). High N concentrations can lead to high NO<sub>x</sub> emissions. Though, the boiler technique is more crucial for NO<sub>x</sub> emissions than the N content in the fuel (Kasuya et al., 1995). The emissions caused by our product are presented in the combustion report. The mean mass flow of Ca was 46.01 %, which is similar to former results (Hensgen et al., 2012).

Tab. 3: Concentrations of several elements and ash content in % DM in the silage and the press cake for each sampling region. Mass flow of elements/ash into the press cake is given.

|                             | Belgium |               |                              | France |               |                              | Wales   |               |                              | Mean   |               |                              |
|-----------------------------|---------|---------------|------------------------------|--------|---------------|------------------------------|---------|---------------|------------------------------|--------|---------------|------------------------------|
|                             | Silage  | Press<br>cake | Mass flow into<br>press cake | Silage | Press<br>cake | Mass flow into<br>press cake | Silage  | Press<br>cake | Mass flow into<br>press cake | Silage | Press<br>cake | Mass flow into<br>press cake |
| Ca (% DM)                   | 0.54    | 0.43          | 51.57                        | 0.69   | 0.33          | 30.65                        | 0.41    | 0.35          | 55.83                        | 0.55   | 0.37          | 46.01                        |
| Cl (% DM)                   | 0.45    | 0.04          | 5.35                         | 1.24   | 0.11          | 5.79                         | 0.86    | 0.11          | 7.97                         | 0.85   | 0.09          | 6.37                         |
| Cu (mg kg <sup>-1</sup> DM) | 6.07    | 4.60          | 49.29                        | 11.17  | 5.80          | 33.76                        | 8.37    | 5.53          | 42.99                        | 8.54   | 5.31          | 42.01                        |
| K (% DM)                    | 0.95    | 0.08          | 5.61                         | 1.96   | 0.22          | 7.44                         | 1.02    | 0.16          | 9.93                         | 1.31   | 0.15          | 7.66                         |
| Mg (% DM)                   | 0.21    | 0.06          | 19.30                        | 0.18   | 0.04          | 16.01                        | 0.23    | 0.08          | 21.95                        | 0.21   | 0.06          | 19.09                        |
| Mn (mg kg <sup>-1</sup> DM) | 137.33  | 53.50         | 25.32                        | 185.00 | 46.77         | 16.43                        | 1151.33 | 392.67        | 22.17                        | 491.22 | 164.31        | 21.31                        |
| N (% DM)                    | 1.58    | 0.89          | 36.55                        | 1.79   | 0.66          | 24.09                        | 1.64    | 0.79          | 31.19                        | 1.67   | 0.78          | 30.61                        |
| Na (% DM)                   | 0.37    | 0.07          | 11.58                        | 0.29   | 0.07          | 15.16                        | 0.42    | 0.10          | 16.16                        | 0.36   | 0.08          | 14.30                        |
| P (% DM)                    | 0.27    | 0.05          | 13.02                        | 0.26   | 0.06          | 14.18                        | 0.21    | 0.05          | 16.62                        | 0.25   | 0.05          | 14.60                        |
| Zn (mg kg <sup>-1</sup> DM) | 39.70   | 23.80         | 42.15                        | 68.37  | 42.27         | 40.19                        | 75.33   | 41.73         | 36.01                        | 61.13  | 35.93         | 39.45                        |
| S (%DM)                     | 0.18    | 0.07          | 24.11                        | 0.20   | 0.06          | 20.22                        | 0.23    | 0.08          | 22.46                        | 0.20   | 0.07          | 22.26                        |
| Ash (% DM)                  | 7.4     | 3.88          | 34.11                        | 13.96  | 4.40          | 20.48                        | 7.07    | 3.54          | 32.55                        | 9.48   | 3.94          | 29.05                        |

Concentrations of heavy metals were lower in the press cake than in the silage in general. Mean mass flows between 21.31 % (Mn) and 42.01 % (Cu) were detected indicating a higher flow of metals into the press fluid than in the press cake. While mean concentrations of Cu and Zn in the silage were in the range of levels found in agricultural grass, mean concentrations of Mn were rather high, mainly due to elevated concentrations in the Welsh material (range found for grass in GB: 79-160 mg kg<sup>-1</sup> DM; Kabata-Pendias, 2011).

### 3.3 Energy yields of press cake and press fluid

The energy yield of the press cakes is defined by the higher (HHV) and the lower heating value (LHV). For every region the HHV and the LHV of both, silage and press cake, are presented in Tab. 4. The mean HHV of silage was 18.37 MJ/kg DM with lowest value in silage from France with an HHV of 17.65 MJ/kg DM and highest values in silage from Wales with an HHV of 18.86 MJ/kg DM. In the press cake the HHV increased to a mean value of 18.78 MJ/kg DM. For silage and press cake from semi-natural grassland similar values could be found with sampling in June (18.27-18.83 MJ/kg, Richter et al., 2011). In general, wood has a HHV close to 20 MJ/kg, whereas grass species have an HHV about 18 MJ/kg. The LHV of grass is for *Festuca rubra* 16.4 MJ/kg DM and for hay from nature conservation areas 17.4 MJ/kg DM (Kutschmitt, 2009). This is in accordance with the findings in this study: the LHV in silage from all regions was about 17 MJ/kg DM with a range from 16.67 to 17.81 MJ/kg DM. The value increases in the press cake to a mean LHV of 17.70 MJ/kg DM. This is an increase of 2% only, which might be due to an already high fibre content of the silage. Hensgen et al. (2011) found an increased LHV in the press cake caused by the IFBB technique of 25 % of LHV calculated from silage, which was potentially less fibrous. However, the LHV of the press cake from the material tested in this study is close to the LHV presented by Büchle et al. (2012) for semi-natural grassland. The LHV<sub>15%wt</sub>, which refers to a water content of 15 % in the fuel, ranged between 12.85 and 13.09 MJ/kg fuel<sub>15%wt</sub>. Dried woodchips from beech have a LHV<sub>15%wt</sub> of 15.3 MJ/kg (Hartmann H, 2009). The LHV<sub>15%wt</sub> of the press cake is about 13% less than of beech woodchips and this fact has to be considered in the economic evaluation.

Tab. 4 Lower heating value (LHV) in MJ/kg DM in silage and press cake, as well as LHV<sub>15%wt</sub> in MJ/ kg fuel with 15 % water content in the fuel of different sampling regions is given. For comparison with other solid fuels the LHV will be relevant though the LHV<sub>15%wt</sub> gives the actual performance of the fuel.

|                      | Belgium |            | France |            | Wales  |            | Mean   |            |
|----------------------|---------|------------|--------|------------|--------|------------|--------|------------|
|                      | Silage  | Press cake | Silage | Press cake | Silage | Press cake | Silage | Press cake |
| HHV                  | 18.61   | 18.78      | 17.65  | 18.65      | 18.86  | 18.92      | 18.37  | 18.78      |
| LHV                  | 17.55   | 17.69      | 16.67  | 17.57      | 17.81  | 17.85      | 17.34  | 17.70      |
| LHV <sub>15%wt</sub> | 12.84   | 12.96      | 12.09  | 12.85      | 13.06  | 13.09      | 12.66  | 12.97      |

Methane content of obtained biogas varied between 57.75 (France) and 60.96 % (Belgium) with mean values of  $58.97 \pm 1.42$  % of biogas. These are high proportions in comparison to methane contents found in former studies. Richter et al. (2009) found proportions of 53 to 56 % of biogas.

Highest methane yield could be obtained with a value of  $206.09 \text{ l}_N \text{ CH}_4/\text{kg VS}$  from press fluid of silage harvested in Belgium (Fig. 21). Lowest methane yields were found for France with  $179.07 \text{ l}_N \text{ CH}_4/\text{kg VS}$ . Mean methane yields were  $221.06 \pm 33.14 \text{ l}_N \text{ CH}_4/\text{kg VS}$ . These values were below methane yields known from former studies on semi-natural grassland. Richter et al. (2009) measured  $426 \text{ l}_N \text{ CH}_4/\text{kg VS}$  in fermentation of press fluid obtained by IFBB treatment at  $60^\circ\text{C}$ . However, they did not take the amount of highly volatile acids into account, whereby the biogas yields are overestimated. Average methane yields of whole-crop silage from grassland vegetation, methane production from grass without IFBB procedure, were in the range of 158 to  $268 \text{ l}_N \text{ CH}_4/\text{kg VS}$ . In general, the press fluid is well digestible because easy soluble carbohydrates are transferred from the silage into the press fluid by IFBB treatment. However, silage of this study might have had a very small content of easy soluble carbohydrates due to its mature stage.

The methane development according to fermentation day is presented in Fig. 21. A regular methane production could be observed for every region. In general, the main methane yield is obtained with in the first 5 to 8 days of fermentation causing kind of a saturation curve.

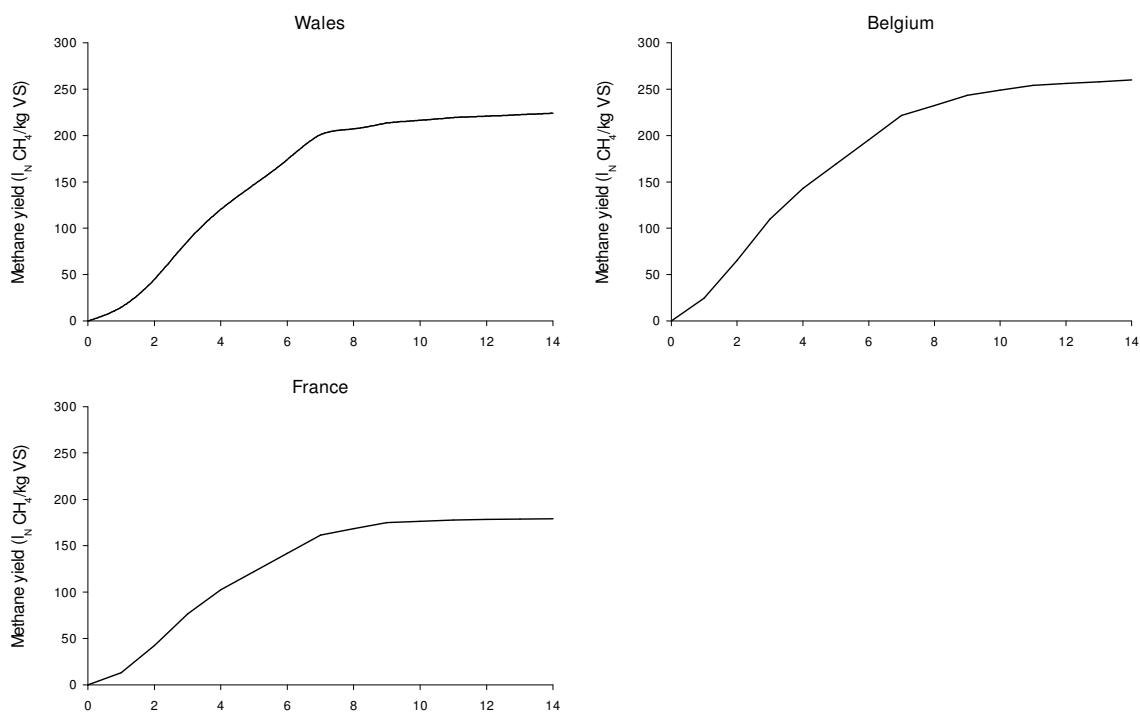


Fig. 21 Methane yields from press fluids gained from IFBB treatment of three silages from different regions.

The energy obtained by the IFBB process was mostly derived from the press cake (Tab. 5). In material from all three partner regions, the energy share of the press cake was higher than 80 % of overall energy output. In France, the press cake even reached an energy share of 87 %.

Tab. 5 Energy share of press cake and press fluid in relation to overall energy output.

|              | Belgium       |                | France        |                | Wales         |                | Mean          |                |
|--------------|---------------|----------------|---------------|----------------|---------------|----------------|---------------|----------------|
|              | Press<br>cake | Press<br>fluid | Press<br>cake | Press<br>fluid | Press<br>cake | Press<br>fluid | Press<br>cake | Press<br>fluid |
| Energy ratio | 81            | 19             | 87            | 13             | 84            | 16             | 84            | 16             |

## 4 Summary

In this study materials harvested by three partners were investigated for basic parameters in energy recovery. The material was processed with the IFBB technique and samples of silage, as well as of press cake and press fluid were taken. The silages and press cakes were analysed for heating values and the press fluids for methane production potential.

The IFBB procedure was conductible with all delivered biomasses. The concentration of VS in the silage was as expected and differences in VS concentration in the press cake between regions did barely occur. The HHVs and the LHV<sub>s</sub> of the press cakes from different regions were close and constantly higher in the press cake than in the silage. The value level was as expected from studies concerning semi-natural grassland. Methane yields on the other hand were rather low. Although the press fluid is generally well digestible, the methane production was in some cases either very small, which is probably due to the maturity of the material with a low content of soluble carbohydrates and a high content of fibres. However, the high content of fibres enhances the suitability as solid fuel.

In conclusion it was possible to convert the material of three partner regions into a solid fuel, whose heating value is higher than the heating value of hay. The higher heating value of the fuel produced is only about 10 % less than the higher heating value of wood.

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