

**Impact of sward type, cutting date and conditioning  
temperature on mass and energy flows in the  
integrated generation of solid fuel and biogas from  
semi-natural grassland**

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Impact of sward type, cutting date and conditioning temperature on material and energy fluxes in the integrated generation of solid fuel and biogas from semi-natural grassland

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## Preface

This thesis is submitted to the Faculty of Organic Agricultural Sciences of the University of Kassel to fulfil the requirements for the degree Doktor der Agrarwissenschaften (Dr. agr.). This dissertation is based on four papers as first author and one paper as co-author, which are published by or submitted to international refereed journals. A list of the original papers including the chapter in which they appear in this dissertation will be given on the next page. A list of other publications (e.g. contributions to conference proceedings) is given in chapter 13.

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## List of papers

- Chapter 3: WACHENDORF M., RICHTER F., FRICKE T., GRAB R. and NEFF, R. (2009) Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. I. Effects of hydrothermal conditioning and mechanical dehydration on mass flows of organic and mineral plant compounds, and nutrient balances. *Grass and Forage Science*, **64**, 132-143.
- Chapter 4: RICHTER F., GRAB R., FRICKE T., ZERR W. and WACHENDORF M. (2009) Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. II. Effects of hydrothermal conditioning and mechanical dehydration on anaerobic digestion of press fluids. *Grass and Forage Science*, **64**, 354-363.
- Chapter 5: RICHTER F., FRICKE T. and WACHENDORF M. (2010) Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. III. Effects of hydrothermal conditioning and mechanical dehydration on solid fuel properties and on energy and green house gas balances. *Grass and Forage Science*, **65**, 185-199.
- Chapter 6: RICHTER F., FRICKE T. and WACHENDORF M. (2010) Influence of sward age and pre-conditioning temperature on the energy production from grass silage through the integrated generation of solid fuel and biogas from biomass (IFBB): 1. The fate of mineral compounds. *Bioresource Technology*, accepted for publication.
- Chapter 7: RICHTER F., FRICKE T. and WACHENDORF M. (2010) Influence of sward age and pre-conditioning temperature on the energy production from grass silage through the integrated generation of solid fuel and biogas from biomass (IFBB): 2. Properties of energy carriers and energy yield. *Bioresource Technology*, accepted for publication.

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## Abbreviations

ADF:	Acid detergent fibre
ADL:	Acid detergent lignin
AST:	Ash softening temperature
CF:	Crude fibre
CH:	Combustion of hay
CHP:	Combined heat and power plant
CP:	Crude protein
DM:	Dry matter
EE:	Ether extract
EU:	European Union
FIT:	Feed-in tariff
GHG:	Green house gas
GLM:	General linear model
+/-hc:	With/without hydrothermal conditioning
HHV:	Higher heating value
HRT:	Hydraulic retention time
IFBB:	Integrated generation of solid fuel and biogas from biomass
LIHD:	Low-input high-diversity
L <sub>N</sub> :	Normal liter
LRT:	Null model likelihood ratio test
m <sup>3</sup> <sub>N</sub> :	Normal cubic meter
NFC:	Non-fiber carbohydrates
NFE:	Nitrogen-free extracts
NDF:	Neutral detergent fibre
OLR:	Organic loading rate
OM:	Organic matter
PC:	Press cake
PF:	Press fluid
PM:	Parent material
PMC:	Parent material after hydrothermal conditioning
RES:	Renewable energy source
RMSE:	Root mean square error
VS:	Volatile solids
WCD:	Whole-crop digestion

## 1 General introduction

In view of the global climate change debate, the finite nature and instability of fossil fuel supply and the requirements of the European Union (EU) regarding environment protection, there are two important trends in agriculture, which increase the pressure on agricultural production.

On the one hand, as result of the EU's goal to increase energy efficiency and the share of renewable energy sources in total energy production to 20% by 2020 (Anonymous, 2006), the cultivation of biomass crops for energy production requires a continuously increasing proportion of agricultural land (Fischer et al., 2010). In this context, questions arise about the efficiency of energy production from agricultural crops, greenhouse gas (GHG) mitigation and the competition with food production on a finite land resource (Cannell, 2002; Hoogwijk et al., 2003; Faaij, 2006). In Germany, which is the largest producer of biomass energy in the EU 27 (23% of the total biomass energy in 2008; EUROSTAT, 2010), 80% of the arable land used for energy crops is covered with rapeseed and maize (FNR, 2010). Their cultivation requires an intensive consumption of fertilizers and pesticides, which may result in reduced energy conversion efficiencies and increased GHG emissions (Crutzen, 2007). Furthermore continuous maize or rapeseed cropping is associated with other environmental impacts, such as soil erosion, nutrient leaching and reduced soil fertility (Riffaldi et al., 1994; Graß and Scheffer, 2005; Frondel and Peters, 2007). The conflicts regarding competition with food production, low conversion efficiencies and negative environmental impacts could be reconciled by an intensified use of residual material for bioenergy production (Bergsma et al., 2007).

On the other hand, feeding patterns in modern livestock farming have changed from species-rich forage obtained from semi-natural grassland with low energy content to an increased use of arable forage crops and concentrates, leaving vast areas of grassland without management (WallisDeVries et al., 2002; Lindborg and Eriksson, 2004). In Germany, it is estimated that approximately 25% of the total grassland area (4.87 Mha in 2007; Anonymous, 2008) will be abandoned in the near future due to high costs of harvesting and low nutritive value of the forage (Isselstein et al., 2005; Prochnow et al., 2007). Under central European conditions this abandonment causes, in the course of natural succession, the formation of woods and shrubs and results in a dramatic loss of species (Poschlod et al., 2005). As the EU has set the goal to conserve biodiversity in these ecologically valuable habitats, subsidies have to be paid in the

form of agri-environment schemes to reach this goal (Kleijn and Sutherland, 2003). In order to ensure conservation in a long term, the traditional site-adapted management practices have to be continued, but new utilization concepts for the biomass are needed (Poschlod and WallisDeVries, 2002; Donath et al., 2004).

The production of energy from semi-natural grassland biomass could be a creative solution to utilize a renewable energy source that is not competing with food production, that is limiting negative environmental impacts and that is ensuring the regular management and thus the conservation of ecologically valuable habitats. However, common conversion techniques, i.e. anaerobic whole-crop digestion (WCD) of silage in a biogas plant and combustion of hay (CH), face important limitations with regard to semi-natural grassland. In the case of WCD, these are associated with high amounts of lignocellulose, which is resistant to anaerobic fermentation and inhibits the digestion of readily fermentable compounds through inclusion. The consequences are low methane yields (Shiralipour and Smith, 1984; Amon et al., 2007), low conversion efficiencies (Ress et al., 1998; Prochnow et al., 2005; Herrmann et al., 2007) and high retention times in the digester (Noike et al., 1985; Lemmer and Oechsner, 2001). As for combustion of hay, a major limitation are high concentrations of ash and elements, which cause ash melting, fouling, slagging (K, Mg) and corrosion (K, Cl) inside the combustion chamber or hazardous emissions (N, S, Cl) during combustion (Jenkins et al., 1998; Obernberger et al., 2006). Another important limitation lies in the increased dependency on weather conditions for achieving high dry matter (DM) levels to prevent microbial deterioration during storage.

With the aim of overcoming these limitations, the integrated generation of solid fuel and biogas from biomass (IFBB) was developed. Its basic principle is the separation of biomass, conserved as silage, into a liquid fraction (press fluid, PF) and a solid fraction (press cake, PC) after a hydrothermal conditioning (a mash of silage and heated water) in order to optimise energy conversion. The PF, which is more easily digestible than the silage due to the separation process, feeds the biogas plant with an adapted solid-state digester. Subsequently, the biogas is used in a combined heat and power plant (CHP) to produce electricity and heat. The PC is dried with the heat from the CHP and processed to a solid fuel with improved combustion characteristics in comparison to the untreated biomass. As co-product, the digestate from the biogas plant can be used as liquid fertilizer with high concentrations of readily available nutrients.



In experiments with arable crops (maize and wheat silage), the IFBB technique showed promising results regarding the reduction of elements in the PC and thus an improvement of combustion quality, and the methane yield of PF (Reulein et al., 2007; Graß et al., 2009). Based on these experiences, the performance of the IFBB process, when utilizing biomass from different semi-natural grassland swards, was to be investigated in the first experiment of this study. Different temperature treatments of hydrothermal conditioning were also tested, as a variation of this parameter influenced element mass flows in the study of Graß et al. (2009). In the second experiment, an investigation with different cutting dates within an undisturbed spring growth of a grassland sward should give more insight into the internal mass and energy flows and about crucial factors for the optimisation of the IFBB process.

## 2 Research objectives

The objectives of this study were to investigate mass and energy flows as well as quality aspects of energy carriers within the IFBB process and determine their dependency on biomass-related and technical parameters.

Two different experiments were conducted, using biomass from permanent semi-natural grassland swards and converting it according to the IFBB process. In the first experiment, biomass from five different grassland swards was harvested at one single date, in the second experiment biomass from one single grassland sward was harvested at eight consecutive dates of one growing season. In the IFBB process, all technical parameters were kept at a constant level in both experiments, except for the temperature of hydrothermal conditioning, which was varied in three levels in the first experiment and in five levels in the second experiment.

The specific objectives of the experiments were

- (i) to evaluate the influence of hydrothermal conditioning at different temperatures as well as different botanical composition and different maturity stages of grassland biomass on mass flows of dry matter (DM), organic compounds and elements into the PF,
- (ii) to evaluate nutrient balances on grasslands through the removal of herbage for the conversion to energy in the IFBB process and the application of the IFBB digestate,
- (iii) to determine the chemical composition of PF and PC under the influence of different temperatures of hydrothermal conditioning and different maturity stages of the grassland biomass,
- (iv) to determine the methane production and the degradation of organic matter in anaerobic digestion of PF in comparison to WCD of grassland silage,
- (v) to determine combustion related quality parameters of PC in comparison to untreated grassland biomass,
- (vi) to evaluate energy and GHG balances of the IFBB process in comparison to WCD and CH and
- (vii) to identify easily determinable biomass-related and technical parameters, based on which mass flows, concentrations of plant compounds in the PC and the PF as well as energy yield and conversion efficiency of the IFBB process can be estimated.

### **3 Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. I. Effects of hydrothermal conditioning and mechanical dehydration on mass flows of organic and mineral plant compounds, and nutrient balances.**

**Abstract** The use of semi-natural grasslands for the production of renewable energy through conventional conversion techniques faces major limitations due to chemical and physical properties of the biomass. A new conversion procedure was developed which separates the biomass, as silage, into a liquid phase for biogas production and into a solid fraction to be used as fuel. Separation (mechanical dehydration) is carried out with a screw press after mashing with water (hydrothermal conditioning). The effect of hydrothermal conditioning at different temperatures (5°C, 60°C and 80°C) and mechanical dehydration on mass flows of plant compounds into the press fluid was investigated for five grassland pastures typical of mountain areas of Germany. Results show that 0.18 of the crude fibre was transferred into the fluid, whereas more digestible organic compounds, such as crude protein and nitrogen-free extract, showed mass flows of 0.40 and 0.31, respectively. While 0.52 to 0.89 of potassium (K), magnesium (Mg) and chloride (Cl), which are detrimental for the combustion of the press cake, were transferred into the press fluid, more than 0.50 of calcium, which has positive combustion properties, remained in the press cake. Significantly ( $P < 0.05$ ) higher mass flows were detected at conditioning temperatures of 60°C (K and Mg) and 80°C (crude fibre and nitrogen-free extract) compared to the 5°C treatment. Due to the separation of solids and liquids, high proportions of P (0.61-0.74) and K (0.64-0.85) but only 0.32-0.45 of nitrogen exported from the grassland would be recycled with an application of the digestates from the anaerobic digestion of the press liquid.

#### **3.1 Introduction**

The rising consumption of energy globally, the growing shortage of fossil fuels and a world-wide increase in CO<sub>2</sub> emissions means that there is an urgent need to develop sustainable, eco-friendly energy supplies. The Commission of the European Union (EU) has set the goal of raising the proportion of electricity made from renewable energy in the EU to 0.21 by 2010 and the proportion of energy consumption from renewable energy to 0.20 by 2020

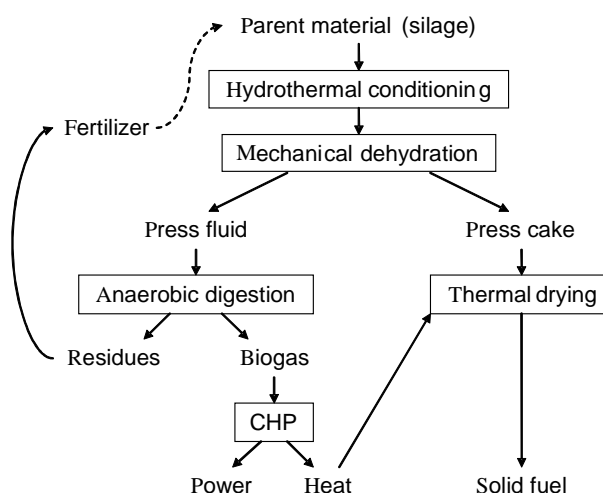
(Anonymous, 2006). Energy from biomass can make a significant contribution, yet its potential is far from being fully exploited. However, the cultivation of energy crops often competes with food production for limited agricultural land. In practice there are only few crops, such as maize, rapeseed and cereals, which are suitable for bioenergy production. Their cultivation is restricted to arable soils and their production requires the intensive consumption of resources which may result in reduced efficiencies and in increased greenhouse gas emissions (Crutzen, 2007), soil erosion, nutrient leaching and reduced soil fertility (Graß and Scheffer, 2005).

In order to achieve the goals established by the EU, the potential of all possible sources of biomass, such as municipal biological wastes and residual material from forest, agriculture and nature conservation areas, needs to be evaluated. In relation to semi-natural grassland, specific regulations at a national or regional level, according to the different situations and definitions of semi-natural grassland, have been established throughout Europe for the conservation of these grasslands. Some countries in the EU, however, are facing great difficulties in ensuring regular utilisation of herbage by ruminants from these grasslands in order to meet conservation objectives. For instance, it is estimated that approximately 0.25 of all German grassland (4.87 m ha in 2007; Anonymous, 2008) will be abandoned in the near future due to high costs of harvesting forage and the low nutritive value of the forage (Prochnow et al., 2007). Thus, for countries rich in semi-natural grasslands it is important to evaluate their utilisation as biomass for renewable energy.

Common conversion techniques, i.e. anaerobic fermentation of grass silage in a biogas plant and combustion of hay, face important limitations with regard to semi-natural grassland. In the case of biogas production, these are associated with the ligno-cellulose content of the biomass which is resistant to anaerobic fermentation and which inhibits the digestion of readily fermentable compounds through their inclusion. There are low yields of methane (Shiralipour and Smith, 1984; Amon et al., 2007a), low efficiencies of conversion (Ress et al., 1998; Prochnow et al., 2005; Herrmann et al., 2007) and high retention times in the digester (Noike et al., 1985; Lemmer and Oechsner, 2001). Furthermore, in order to achieve satisfactory conversion efficiencies the waste heat from combined heat and power plants (CHP), which accounts for more than 0.60 of the total energy contained in the biogas, must be used. This is difficult to realise in rural areas as there is only little demand for heat compared to industrialised urban areas.

As for combustion of hay, a major limitation lies in the increased dependency on weather conditions for achieving dry matter (DM) contents of  $850 \text{ g kg}^{-1}$ . Such DM contents are necessary to prevent microbial deterioration during storage. Field losses during haymaking are usually higher than for silage production which results in reduced yields of energy. Compared to wood as the most common solid biofuel, hay contains more nitrogen (N), potassium (K), magnesium (Mg) and chlorine (Cl). During combustion N is almost completely transformed into nitrogen oxides ( $\text{NO}_x$ ) which are major air pollutants (Greul, 1998). K and Cl are significantly involved in corrosion processes in the furnace and K, together with Mg, promotes the melting of the ash at high temperatures, while Ca works against it (Hartmann, 2001). While the ash from wood melts at temperatures above  $1200^\circ\text{C}$ , ash from hay melts at temperatures below  $1000^\circ\text{C}$  (Hartmann, 2001). The melting of the ash leads to slagging and fouling processes inside the combustion chamber, which reduces the availability of the processing plant and its life (Oberberger et al., 2006). Complex technologies are required to prevent the adherence of the ash to iron walls within the furnace and to reduce the emission of particles and chemical compounds harmful to the environment.

With the aim of overcoming the problems outlined above, a procedure is proposed which separates the easily fermentable constituents of biomass from grassland, as silage, into a liquid form to be converted into biogas from the more fibrous parts which are processed to form a solid fuel (Figure 3.1). The first step is a hydrothermal conditioning of the silage in which silage and water are mixed and heated under continuous stirring for a short time. This treatment aims to macerate cell walls and produces a mash which is then mechanically dehydrated by a screw press.



**Figure 3.1** Flow chart of the IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass) procedure. CHP refers to a combined heat and power plant.

As an effect of the conditioning and dehydration, several minerals (e.g. K, Mg, P and Cl) and organic compounds (e.g. carbohydrates, proteins and lipids) are transferred into the press fluid. Data from the dehydration of whole-crop silages of maize and wheat show that press fluids make an excellent substrate for anaerobic digestion with yields of methane of up to 500 L<sub>N</sub> CH<sub>4</sub> kg<sup>-1</sup> volatile solids and almost total degradation of the organic matter in less than 15 days (Reulein et al., 2007). The remaining press cake is rich in cellulose, hemicellulose and lignin, and contains relatively low proportions of the minerals, which increase the quality of the resulting biofuel (Hartmann, 2001). The DM content of the press cake is about 450-500 g kg<sup>-1</sup>. With the need to dry it to 850 g kg<sup>-1</sup> DM in order to make it suitable for pelleting and storage (Hartmann, 2001), the IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass) system provides a year-round demand for heat produced in the CHP (Wachendorf et al., 2007).

For arable crops Graß et al. (2009) calculated that approximately 0.50 to 0.60 of the gross energy contained in the crop is converted to electricity and heat (which is stored in the solid fuel). This is within the range found for modern conventional biogas plants where all the heat is utilized. The benefit of IFBB has not been determined so far but it is hypothesised that, notwithstanding the low fermentation rates of mature herbage from grasslands in whole crop digestion, such a source of biomass with the IFBB system could have a similar energy conversion efficiency as arable crops. The conversion of semi-natural grasslands to energy with IFBB results in three major products: (i) a solid fuel of acceptable quality, which is suitable for combustion, gasification or subsequent processing to, for example, synthetic fuels; (ii) electricity produced in the CHP from the combustion of biogas which may be utilised on-site or fed into the public electricity grid and (iii) digestates from the biogas plant which can be used as liquid fertilizer with high concentrations of readily available nutrients.

This paper is the first of a series of papers which explores the IFBB procedure when applied to a range of typical semi-natural grasslands, addressing the following questions:

- (i) To what extent does the mechanical dehydration result in a separation among organic and mineral plant compounds?
- (ii) How does temperature in the hydrothermal conditioning treatment affect the mass flows into the press fluid?
- (iii) How does IFBB through the removal of herbage and the application of digestate affect nutrient balances on the grassland?

## 3.2 Material and Methods

### 3.2.1 Herbage

The herbage used in this study was collected from five semi-natural grasslands in mountain areas of the Black Forest and Rhön, Germany, representing typical European mountain areas with two different poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*). Information about the dominant species in the swards, site-related characteristics, harvest dates and yields are presented in Table 3.1 and Table 3.2.

**Table 3.1** Dominant species of semi-natural grasslands used to make silage for use as parent material.

Grassland types	Dominant species
<i>Arrhenaterion</i> I	<i>Agrostis tenuis</i> , <i>Galium pumilum</i> , <i>Arrhenaterum elatius</i> , <i>Hypericum maculatum</i> , <i>Achillea millefolium</i> , <i>Vicia cracca</i>
<i>Arrhenaterion</i> II	<i>Agrostis tenuis</i> , <i>Hypericum maculatum</i> , <i>Holcus mollis</i> , <i>Arrhenaterum elatius</i> , <i>Alchemilla vulgaris</i> , <i>Galium pumilum</i> , <i>Plantago lanceolata</i> , <i>Campanula patula</i>
<i>Caricion fuscae</i>	<i>Carex nigra</i> , <i>Carex canescens</i>
<i>Filipendulion ulmariae</i>	<i>Filipendula ulmaria</i> , <i>Scirpus sylvaticus</i>
<i>Polygono-Trisetion</i>	<i>Festuca rubra</i> , <i>Trisetum flavescens</i> , <i>Sanguisorba officinalis</i> , <i>Geranium sylvaticum</i> , <i>Alopecurus pratensis</i> , <i>Polygonum bistorta</i> , <i>Avena pubescens</i> , <i>Knautia arvensis</i> , <i>Anthoxanthum odoratum</i>

The grasslands were managed according to the directives of regional agri-environmental schemes with one late harvest per year. Due to the late harvest date, most species had finished flowering. No mineral or organic fertilizer had been applied for more than 10 years. Herbage was harvested with a finger-bar mower at a cutting height of 5 cm on three randomly located plots of 25 m<sup>2</sup> and fresh weight was assessed immediately. Sub-samples were taken for assessment of DM content. About 20 kg fresh matter (FM) from each plot were chopped in 5-cm long pieces, compacted and ensiled for three months in a 50 L polyethylene barrel with no additives being applied. After opening the barrels, fermentation quality was checked for colour, texture and smell (Wilkinson, 2005). All attributes indicated that silages had been well preserved.

**Table 3.2** Characteristics of semi-natural grasslands with silage made from the grasslands used as parent material in the study

Grassland types	Location	Altitude (m a.s.l)	Annual recipitation (mm)	Mean annual temperature (°C)	Harvest date	DM yield (t ha <sup>-1</sup> )
Arrhenaterion I	Black Forest	850-860	1520	6.0	31/08/2006	3.64
Arrhenaterion II	Black Forest	850-860	1520	6.0	31/08/2006	4.94
Caricion fuscae	Black Forest	850-860	1520	6.0	31/08/2006	5.75
Filipendulion ulmariae	Black Forest	850-860	1520	6.0	31/08/2006	10.27
Polygono-Trisetion	Rhön	760-820	1070	5.9	19/07/2006	3.39

### 3.2.2 Hydrothermal conditioning and mechanical dehydration

Prior to hydrothermal conditioning and mechanical dehydration, silage from each of the three barrels from each sward was pooled, thoroughly mixed and three sub-samples of 20 kg FM each were taken. Sub-samples were mixed with 80 kg water of different temperatures (5°C, 60°C and 80°C, respectively) in a modified concrete mixer with a volume of 200 L, resulting in a mash with a silage:water ratio of 1:4. The mash was kept at a constant temperature with gas burners and stirred for 15 min in order to thoroughly rinse the silage with water. Subsequent mechanical dehydration of the silage was conducted with a screw press (type AV, Anhydro Ltd., Kassel, Germany). The conical screw had a pitch of 1:7.5 and a rotational speed of 12 revolutions min<sup>-1</sup>. The cylindrical screen encapsulating the screw had a perforation of 1.5 mm. Sub-samples of the silage and the press fluid were stored at 4°C for anaerobic digestion experiments, sub-samples of the silage and the press cake were immediately dried at 60°C for 24 h for chemical analysis. Dry matter (DM) content of all sub-samples of the silage, the press cake and the press fluid was determined by oven-drying at 105°C for 48 h.

### 3.2.3 Chemical analyses and mass flow calculation

The silage (parent material, PM) and the press cake (PC) were analyzed for K, Mg, Ca, Cl, S and P by X-ray fluorescence analysis and for the Weende constituents, crude ash, crude protein (CP), ether extract (EE), crude fibre (CF) and nitrogen-free extract (NFE) according to standard methods (Bassler, 1976). The concentrations of ash, CP, EE, CF and NFE



(represented by Z; g kg<sup>-1</sup> DM) in the press fluid (PF) were calculated from the proportions of PC and PF in the silage after hydrothermal conditioning (PMC) according to:

$$Z_{PF} = \frac{DM_{PMC} * Z_{PM} - Y * DM_{PC} * Z_{PC}}{X * DM_{PF}}$$

where X and Y are the quantities of the PF and the PC as a proportion of the parent material after hydrothermal conditioning, respectively, which were calculated by:

$$X = \frac{DM_{PC} - DM_{PMC}}{DM_{PC} - DM_{PF}} \quad Y = 1 - X$$

The mass flow (MF) of DM and of any other compound (Z) from the PM into the press fluid (equations 1 and 3) and the PC (equations 2 and 4) were determined by:

$$(1) \quad MF_{DM_{PF}} = \frac{X * DM_{PF}}{DM_{PMC}} \quad (2) \quad MF_{DM_{PC}} = 1 - MF_{DM_{PF}}$$

$$(3) \quad MF_{Z_{PF}} = \frac{X * DM_{PF} * Z_{PF}}{DM_{PMC} * Z_{PMC}} \quad (4) \quad MF_{Z_{PC}} = 1 - MF_{Z_{PF}}$$

A full derivation of these equations is given in the Appendix.

### 3.2.4 Statistical analysis

As the main objective of this study was to explore the various processes involved in the IFBB procedure, priority was given to tests of effects of temperature during conditioning, as this treatment proved to have the greatest effect on mass and energy flows in preliminary studies (Reulein et al., 2007). Analysis of variance was performed on the mass flow of each plant compound with conditioning temperature as the only factorial effect using the procedure GLM in SAS (SAS Institute, 1996). Due to the fact that the three different temperature treatments were not replicated but equally applied to each of the five grasslands, grasslands were considered as replicates in the models. Tukey's test was used to test for significant differences among the temperatures. Pearson correlation analysis was performed with the GLM procedure in SAS to determine the relationship between the mass flow of DM and the mass flow of other chemical compounds. Mass flow of DM and the mass flow of each plant compound was compared using a one-factorial analysis of variance with the procedure GLM in SAS.

### 3.3 Results

#### 3.3.1 Chemical composition of the herbage

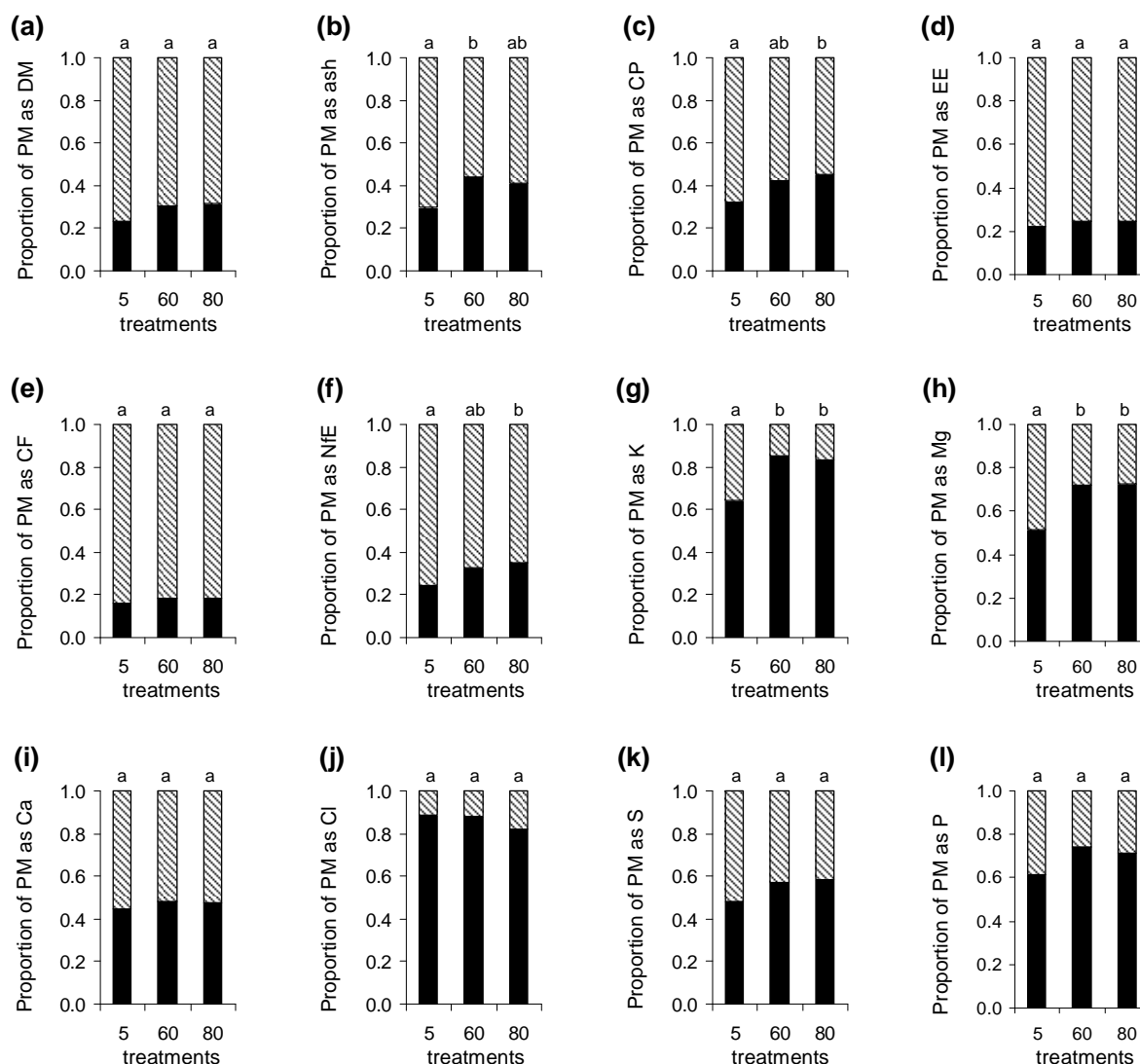
Although the botanical composition was very different between the grasslands, the chemical composition of the silages from the five semi-natural grasslands was quite similar with higher CF and lower CP and mineral concentrations compared to grasslands commonly harvested at the stage of ear emergence for feeding to ruminants (Table 3.3).

**Table 3.3** Dry matter content (DM, g kg<sup>-1</sup> FM) and chemical composition (g kg<sup>-1</sup> DM) of silages from semi-natural grasslands used as parent material.

	Grassland types				
	Arrhena- terion I	Arrhena- terion II	Caricion fuscae	Filipendulion ulmariae	Polygono- Trisetion
DM content (g kg <sup>-1</sup> FM)	405.5	354.2	244.8	382.2	382.1
Chemical composition (g kg <sup>-1</sup> DM)					
Ash	86.3	65.2	82.8	63.5	79.8
Crude protein	70.2	74.2	97.0	99.8	87.4
Ether extract	26.5	19.3	10.0	11.0	20.8
Crude fibre	264.7	286.8	306.7	329.2	246.0
Nitrogen-free extract	552.3	554.4	503.6	496.4	566.0
Potassium	13.1	13.9	9.8	10.1	13.0
Magnesium	2.3	2.3	3.6	3.7	3.8
Calcium	11.1	7.7	6.6	10.6	15.9
Chloride	3.1	5.8	3.9	4.7	6.7
Sulphur	1.3	1.4	2.0	1.6	1.6
Phosphorus	1.9	2.2	1.6	1.5	1.7

#### 3.3.2 Mass flows into the press fluid and the press cake

Proportionately 0.23 - 0.32 of the DM contained in the parent material was directed into the press fluid during mechanical dehydration, while 0.68 - 0.77 was left in the press cake (Figure 3.2). Mass flows of ash into the press fluid were somewhat higher (0.30 - 0.44).



**Figure 3.2** Mean values of proportions of parent material (PM), made from five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)), that contribute to the mass flow of (a) dry matter (DM), (b) ash, (c) crude protein, (d) ether extract (EE), (e) crude fibre (CF), (f) nitrogen-free extract (NFE), (g) K, (h) Mg, (i) Ca, (j) Cl, (k) S and (l) P into the press fluid (■) and the press cake (▨) for treatments of different temperatures during hydrothermal conditioning [5°C (5), 60°C (60), 80°C (80)]. Mean values with different letters indicate significant differences ( $P < 0.05$ ).

On average, the highest transfer of ash was obtained with a conditioning temperature of 60°C which was significantly ( $P < 0.05$ ) higher than that of 5°C (Table 3.4). Mass flows of organic compounds into the press fluid influence the availability of carbon for anaerobic microbes. Crude fibre includes ligno-cellulose structures which are very resistant to anaerobic digestion. Therefore, the low transfer of CF of 0.16 - 0.18 into the press fluid is considered desirable for subsequent biogas production. Crude protein was transferred at rates of 0.32 - 0.45 into the press fluid which was significantly ( $P < 0.05$ ) higher at a conditioning temperature of 80°C than that of 5 °C. Mass flows of EE into the press fluid were remarkably low, which most

likely had only a small effect on further processing as the concentrations in the parent material were low anyway. Mass flows of NFE were of the same magnitude as those of DM, with lowest values at 5°C and significantly ( $P < 0.05$ ) higher values at 80°C. Mass flows of K, Mg and P into the press fluid were proportionately between 0.52 and 0.85 and increased significantly ( $P < 0.05$ ) in the case of K and Mg as the temperature increased from 5°C to 60°C. Transfer of Ca into the press fluid was between 0.44 and 0.48 with no significant ( $P < 0.05$ ) effect of temperature. Thus, 0.52 – 0.56 of the Ca, which has positive effects on the combustion process, remained in the press cake. Mass flows of S ranged from 0.48 to 0.58 and increased slightly, though not significantly, with increasing temperature. Transfer rates of Cl into the press fluid were between 0.82 and 0.89 and were remarkably high even on the lowest temperature treatment.

**Table 3.4** Proportion of mass flow of plant components into the press fluid at three conditioning temperatures (5°C, 60°C and 80°C). *F* values and levels of significance derived from a general linear model are also presented.

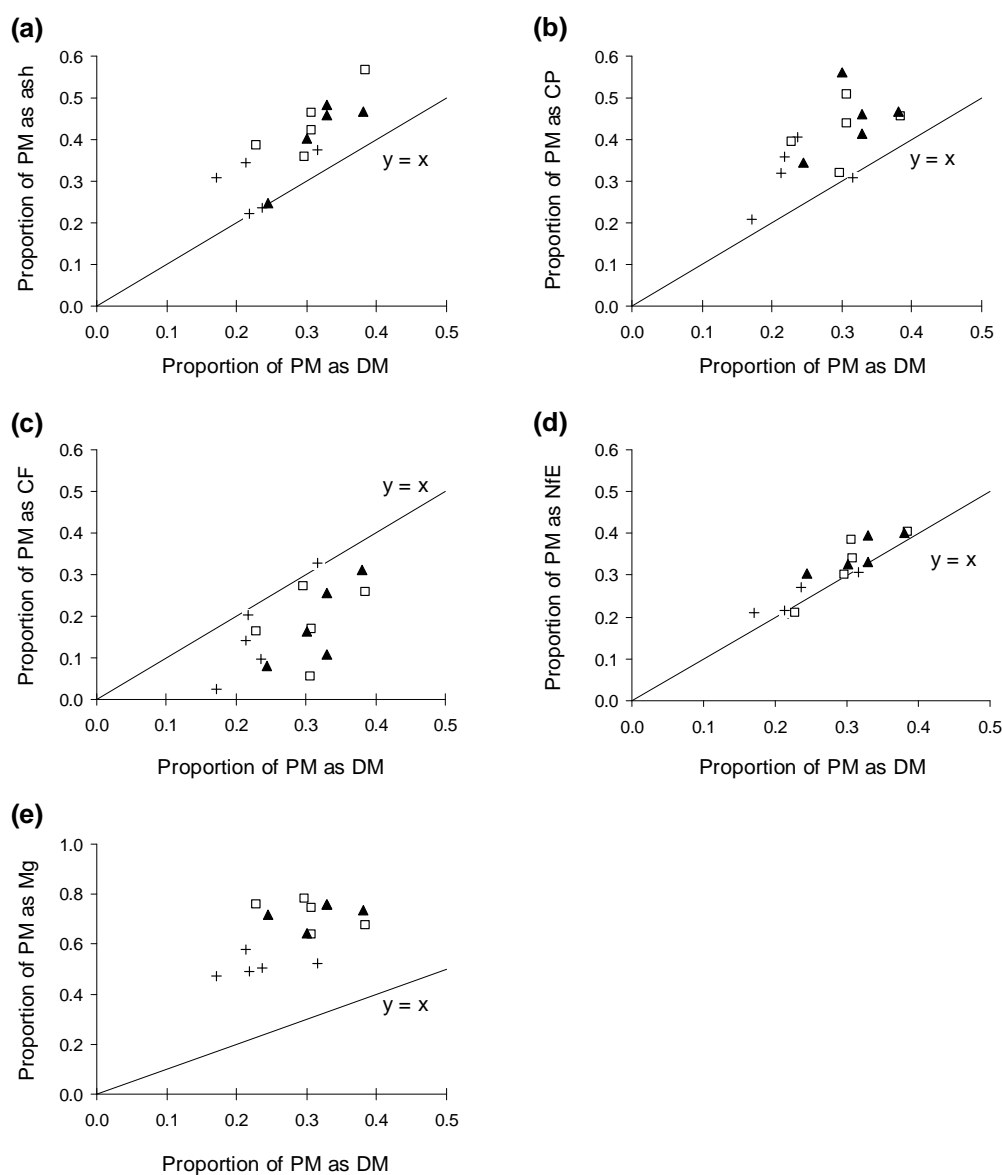
Plant component	Conditioning temperatures (°C)†			s.e. of mean	<i>F</i> -value	Level of significance
	5	60	80			
Dry matter	0.23	0.30	0.32	0.05	3.77	NS
Ash	0.30	0.44	0.41	0.08	4.38	*
Crude protein	0.32	0.42	0.45	0.07	4.44	*
Ether extract	0.22	0.24	0.24	0.14	0.04	NS
Crude fibre	0.16	0.18	0.18	0.10	0.11	NS
Nitrogen-free extract	0.24	0.33	0.35	0.06	4.90	*
K	0.64	0.85	0.83	0.08	10.25	**
Mg	0.52	0.72	0.72	0.05	28.83	***
Ca	0.44	0.48	0.47	0.11	0.19	NS
Cl	0.89	0.88	0.82	0.14	0.35	NS
S	0.48	0.57	0.58	0.06	3.24	NS
P	0.61	0.74	0.71	0.11	2.08	NS

† Means of five grasslands

NS, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

The IFBB system is supposed to produce a press cake with low concentrations of detrimental minerals (K, Mg, Cl) and CP in order to improve its quality as a solid fuel. Enrichment or depletion effects occur if mass flows of single components differ from those of the DM.

Significant correlations were found between mass flows of DM and mass flows of most organic compounds (except EE) with correlation coefficients ranging from 0.62 (CF) to 0.92 (NFE) (Figure 3.3).



**Figure 3.3** Linear relationships between proportions of parent material (PM) of silages, made from five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)), that contribute to the mass flow (Pearson's correlations in parentheses) of (a) ash (0.81\*\*\*), (b) crude protein (CP, 0.63\*), (c) crude fibre (CF, 0.62\*), (d) nitrogen-free extract (NFE, 0.92\*\*\*) and (e) Mg (0.55\*) into the press fluid, and proportion of mass flow as dry matter (DM) for treatments of different temperatures during hydrothermal conditioning [5°C (+), 60°C (□), 80°C (▲)]; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$ .

Mean CF mass flows of 0.16 - 0.18 into the press fluid at simultaneous mean DM mass flows of 0.23 - 0.32 imply a considerable CF depletion in the press fluid and thereby the enrichment of CF in the press cake. The differences between the mass flows were significant ( $P < 0.05$ )

for the 60°C and 80°C treatments. While mass flows of CF were lower than those of DM for all temperature treatments, mass flows of EE and NFE were in the same range as those of DM without any significant difference, so that no enrichment or depletion effects occurred (Table 3.5). Crude protein was transferred into the press fluid at significantly ( $P < 0.05$ ) higher rates than DM on the 60°C and the 80°C treatments, resulting in enrichment in the press fluid and depletion in the press cake. Correlations between mass flows of DM and mineral compounds were only significant for ash ( $r = 0.81$ ,  $P < 0.001$ ) and for Mg ( $r = 0.55$ ,  $P < 0.05$ ). Ash and DM mass flows were significantly different ( $P < 0.05$ ) only for the 60°C treatment.

**Table 3.5** Analysis of variance ( $F$  values and levels of significance) when comparing DM mass flows with mass flows of any other component or element into the press fluid at different conditioning temperatures (5°C, 60°C, 80°C) using a general linear model.

Plant component	Conditioning temperature (°C)			Element	Conditioning temperature (°C)		
	5	60	80		5	60	80
Ash	5	2.8	NS	K	5	94.0	***
	60	9.5	*		60	208.5	***
	80	3.91	NS		80	110.7	***
Crude protein	5	4.8	NS	Mg	5	81.0	***
	60	9.1	*		60	137.6	***
	80	10.3	*		80	162.9	***
Ether extract	5	0.0	NS	Ca	5	12.0	**
	60	0.8	NS		60	15.5	**
	80	1.3	NS		80	7.9	*
Crude fibre	5	1.7	NS	Cl	5	98.3	***
	60	7.0	*		60	105.7	***
	80	7.1	*		80	38.2	***
Nitrogen-free Extract	5	0.1	NS	S	5	34.5	***
	60	0.2	NS		60	42.1	***
	80	1.4	NS		80	63.2	***
				P	5	67.9	***
					60	66.7	***
					80	45.9	***

NS, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$ .

K and Cl were transferred into the press fluid at higher rates than DM with differences between mass flows of K and Cl, and DM, being significant ( $P < 0.001$ ) at all temperatures. Mass flows of Mg, P and S were significantly ( $P < 0.001$ ) higher than those of DM and that

of Ca was also significantly higher than that of DM for the 5°C and 60°C treatments ( $P < 0.01$ ) and for the 80°C treatment ( $P < 0.05$ ).

Contents of DM of the parent material increased proportionately by 0.21 to 1.10 after mechanical dehydration and were 464 to 543 g kg<sup>-1</sup> for the press cake. There was a significant negative linear relationship between DM content of the parent material (x) and increase in DM content (y) during mechanical dehydration (y) ( $y=480 - 0.93x$ ;  $R^2=0.86$ ;  $P < 0.001$ ).

### 3.4 Discussion

The most important step in the IFBB procedure is the mechanical dehydration combined with the hydrothermal pre-treatment of the silage. The several requirements that the conditioning and dehydration technique must comply with are:

- (i) a reduction in the water content of the press cake in order to facilitate the subsequent thermal drying to DM contents of more than 850 g kg<sup>-1</sup>;
- (ii) a reduction in the concentrations of minerals in the press cake which are detrimental to combustion;
- (iii) an enrichment of fibrous constituents in the press cake, as they are less digestible in anaerobic fermentation, but form important oxidizable, carbon-rich constituents in solid fuels; and
- (iv) a transfer of easily degradable organic substances into the press fluid which is subjected to anaerobic digestion to produce biogas.

A vast number of mechanical techniques for separating solids and liquids exist but, compared to thermal techniques, common standards for the choice of type and dimensioning of devices are not available (Wakeman and Tarleton, 1991). Considering the non-availability of information in the literature on the mechanical dehydration of biomass from grassland, the present study follows the approach of Reulein et al. (2007) who used a screw press for removing water from various arable crops. In relation to the dehydration of moist herbage, plant cells which contain the liquid phase have to be macerated before the separation of solid and liquid can take place. Screw presses, which provide a high degree of maceration of cell walls due to axial movement and abrasion of the tissue under high pressure, are frequently used in the sugar beet and rapeseed processing industries (Raß, 2001), in the production of leaf-protein concentrates from leguminous crops (Telek and Graham, 1983) and in bio-refineries, where lactic acids and amino-acids are extracted from herbage (Mandl et al., 2006).

Energy consumption to achieve a target DM content in the press cake is less for the mechanical dehydration of silages than it is for thermal techniques, with the greatest superiority at low DM contents in the parent material (Graß et al., 2009). It is well known from the removal of water from sugar-beet pulp, brewers' spent grains and other applications that the press fluid contains solid particles in a diluted or suspended form, whereas the remaining press cake comprises the more coarse-structured fractions of the raw material (Raß, 2001).

The main application of hydrothermal treatments in conversion to biofuel is the solubilisation of hemicellulose, cellulose and lignin from woody biomass through hydrolysis reactions, e.g. as a pre-treatment in ethanol production (Avellar and Glasser, 1998). Temperatures in this process are usually between 150°C and 230°C, since the kinetics of hydrolysis are low at temperatures below 100°C (Garotte et al., 1999). However, the results of the present study show that water at temperatures in the range of 60-80°C modifies cell structures such that the transfer of certain organic compounds (CP and NFE) into the press fluid is enhanced compared to the 5°C treatment, while the transfer rate of CF remains low. Considering the relationship between mass flows of DM and the organic compounds, enrichment and depletion effects in the press cake are to be expected for CF and CP, respectively, but only for the 60°C and 80°C treatments.

Large differences occurred among the mineral elements in the extent of mobilisation. Minerals which are usually located in the vacuole of plant cells (e.g. K, P and Cl) are easily transferred into the liquid phase once the cells are macerated. Others, such as Ca, are frequently located in cell-wall structures, such as membranes and middle lamellas. Obviously, they are less mobile even with an intensive conditioning procedure prior to pressing. Since there were significant differences in mass flows between DM and any mineral element, enrichment in the press fluid and depletion in the press cake occurred across all temperature treatments. As the concentration of nitrate-nitrogen (N) is low in mature herbage (Weissbach et al., 1993), most of the N is bound in proteins. Presumably, it is primarily cell-wall protein, which is interwoven with structural carbohydrates and lignin, that contributes to the relatively low transfer rates of N in the liquid phase. This is in agreement with the low digestibility of cell-wall proteins, measured as acid-detergent fibre-N in mature forages for ruminants (Goering and Van Soest, 1975).

Hydrothermal conditioning of whole-crop silages from maize resulted in a lower DM content at temperatures above 60°C and in higher mineral concentrations in the press cake compared



to lower temperatures (Graß et al., 2009). A similar result was found for ethanol production from cereals (Senn, 2001). This result may have been due to high concentrations of starch in the maize crop which starts to paste at high temperatures and prevents the water and the minerals from being discharged from the mash. No such effects were observed for silages from semi-natural grassland, so that temperatures of 80°C exhibited maximum mass flows of DM and minerals in the fluid phase.

If grasslands are to remain productive in the long run, nutrients exported with the harvest should be replaced. The conversion of carbon from the press fluids into biogas and solid fuels does not allow an appreciable return of carbon. However, pasture plants transfer 0.30 to 0.50 of assimilates below ground (Kuzyakov and Domanski, 2000), accomplishing a substantial replenishment. Unlike arable land where intensive carbon mineralisation occurs due to cultivation, mineralisation processes on permanent grassland occur at very low rates, resulting in a net accumulation of carbon in the soil.

In relation to the proportions of N, P and K in the press fluid, there are no gaseous emissions during anaerobic digestion (Karpenstein-Machan, 2005) which allows their full return with the digestate to the grassland. However, due to the previous separation of solid and liquid only 0.32 to 0.45 of the N exported from the grassland will be replaced by the return of digestates. In order to offset the balance, additional N would be required from N-fixing legumes or fertilizers. As mass flows into the press fluid are higher for P and K (0.61- 0.74 and 0.64- 0.85, respectively), their return with the digestate is more complete and there is less need for additional fertilizer. However, as a neutral nutrient balance is an essential aspect of a sustainable sward management, great care should be taken to replace nutrients in the long run when applying the IFBB concept. With the exception of carbon and N, nutrient balances could largely be adjusted through the application of ash from the combustion of biomass fuels to the grassland. However, regulations in many European countries make such practices difficult or even prevent them.

### **3.5 Conclusions**

Based on data from typical semi-natural grasslands in German mountain areas the following conclusions can be drawn from the results:

(i) Low transfer rates into the press fluid occurred for CF (0.18 of CF in parent material, on average) which contains less fermentable substances such as lignin, whereas NFE (0.31 on average) and CP (0.40 on average) with a higher potential for fermentation were transferred at

higher rates. Mineral elements, such as K and Cl which are detrimental during combustion, were transferred into the fluid at rates of 0.77 and 0.86 on average, respectively, whereas over 0.50 of Ca in the parent material, which has beneficial effects during combustion, remained in the solid.

(ii) Mass flows into the press fluid of some organic and mineral plant compounds increased with increasing conditioning temperature from 5 to 60°C (K and Mg) or from 5 to 80°C, respectively. The heating to 80°C did not result in a further increase in the case of K and Mg. Although benefits could be expected from this both for fermentation of the fluid and combustion of the solid, a comprehensive evaluation requires consideration of energy demands for heating the mash.

(iii) Due to a low mass flow of N in the liquid phase only 0.32 to 0.45 of the N exported from the grassland would be replaced by the return of digestates. In order to offset the balance, additional N would be required from N-fixing legumes or fertilizers. Return of P and K with the digestate (0.61- 0.74 and 0.64 - 0.85 on average, respectively) is more complete, reducing the need for additional fertilizer. With the exception of carbon and N, application of ash from the combustion of the biomass fuels to the grassland would largely adjust nutrient balances.

## **4 Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. II. Effects of hydrothermal conditioning and mechanical dehydration on anaerobic digestion of press fluids**

**Abstract** A procedure (Integrated Generation of Solid Fuel and Biogas from Biomass, IFBB) was developed which uses a screw press to separate the readily digestible constituents of mature grassland biomass into a press fluid for conversion into biogas and a fibrous press cake for processing into a solid fuel. Effects of mechanical dehydration and prior hydrothermal conditioning at different temperatures (5°C, 60°C and 80°C) on concentrations of organic compounds in the press fluid and on methane production in batch experiments were evaluated for five semi-natural grasslands typical of mountain areas of Germany. Results show that the crude protein concentration of the press fluids was higher and crude fibre concentration was lower than that of the parent material (herbage conserved as silage). Digestion tests in batch fermenters showed that the methane yield of the press fluids was double (397-426 normal litre (L<sub>N</sub>) kg<sup>-1</sup> volatile solids (VS) after 13 d) that of the whole-crop grassland silage (218 L<sub>N</sub> kg<sup>-1</sup> VS after 27 d) but no consistent effect of higher temperature during conditioning was observed. Within 13 d of fermentation decomposition of the organic matter (OM) occurred in the press fluids was 0.90, whereas after 27 d of fermentation more than 0.40 of the OM remained undigested in the whole-crop silage, pointing at a marked reduction in retention time for anaerobic digestion of press fluids in continuous systems. Press fluids produced 0.90 of the maximum methane yield after 4 to 7 d compared with 19 days for the whole-crop silage.

### **4.1 Introduction**

The generation of renewable, 'green' energy, the substitution of fossil fuels and the reduction of CO<sub>2</sub> emissions are major worldwide issues that impact on agriculture. The European Union has the goals of increasing energy efficiency and the proportion of renewable energy sources (RES) in total energy production to 0.20 by 2020. Many of its member states have implemented national regulations to guarantee feed-in tariffs (FIT) for electricity from RES. In most European countries the level of the FIT depends on the size of plant as well as the type of biomass used and in some cases on the region, the time of the day or the utilisation of

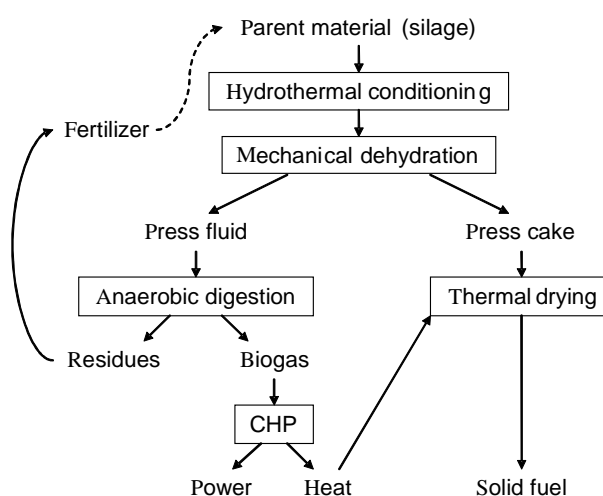
waste heat (Thrän et al., 2006). In 2006 the FIT was on average 5-7 cents kWh<sup>-1</sup> while the value was considerably higher in Germany (up to 21.5 cents kWh<sup>-1</sup>) and Austria (up to 16.5 cents kWh<sup>-1</sup>). In these two countries the biogas production from agricultural biomass has increased rapidly and plays an important role among the RES today.

In Germany, which is the largest biogas producer in Europe, 3711 agricultural biogas plants accounted for, proportionately, 0.012 of the gross electricity consumption in 2007 (BMU, 2008). However, in 0.80 of these plants maize silage is used as the fermentation substrate and most of them have no comprehensive provision for the use of the waste heat from the biogas combustion (FNR, 2005). Continuous maize cropping has degrading effects on the soil organic C and N pools as well as on the exchange capacity and fertility of the soil (Riffaldi et al., 1994). Furthermore, its cultivation for energy production competes with food production on limited agricultural land (Hoogwijk et al., 2003). A comprehensive utilisation of the waste heat from biogas combustion in a combined heat and power plant (CHP) would double the fuel efficiency, reduce emissions and improve the economics of the plant (Korhonen, 2002). Several studies have shown that the highest energy efficiency and the lowest CO<sub>2</sub> mitigation costs could be realized by combined heat and power production from biogas and the provision of heat from solid biofuels (Jungk, 2000; Leible et al., 2008).

Semi-natural grasslands in Europe produce vast amounts of biomass but which can be difficult to exploit both as forage for ruminants and for energy production. A new technology for the conversion of biomass to energy was described (Wachendorf et al., 2009) that takes into account the specific chemical composition of biomass from semi-natural grasslands: both in regard to the negative impact of high fibre concentrations on anaerobic digestion (Prochnow et al., 2005) and the detrimental influence of high mineral and nitrogen (N) concentrations on combustion (Hartmann, 2001). In the IFBB procedure (Integrated Generation of Solid Fuel and Biogas from Biomass) the biomass from grassland is subjected to hydrothermal conditioning and subsequently processed using a screw press, which results in a press fluid for biogas production and a press cake for direct combustion as solid fuel (Figure 4.1).

Drying of the cake with the waste heat from the biogas combustion is a key aspect of the procedure as a dry matter (DM) content of more than 850 g kg<sup>-1</sup> is necessary for storage and pelleting (Reulein et al., 2007). For this reason an adequate supply of biogas is needed. To serve as a suitable substrate, the press fluid, which contains only a minor proportion of the parent material's organic compounds, is required to be highly digestible and to produce high

methane yields. There is ample evidence that anaerobic digestion of biomass from extensive or landscape-management grasslands show rather low methane yields ( $100\text{-}300\text{ L}_N\text{ kg}^{-1}\text{ VS}$ ) in comparison to those of maize or other cereals ( $300\text{-}450\text{ L}_N\text{ kg}^{-1}\text{ VS}$ ) which is mainly due to a low digestibility of the organic matter (Baserga, 1998; Lemmer and Oechsner, 2001; Gronauer and Aschmann, 2003; Prochnow et al., 2005). Not much is known, however, about the fermentation properties of press fluids from such grasslands. One study with press fluids from grassland, in the context of a green bio-refinery project, found a methane yield of  $573\text{ L}_N\text{ kg}^{-1}\text{ VS}$  in a batch experiment (Kromus et al., 2000).



**Figure 4.1** Flow chart of the IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass) procedure. CHP refers to a combined heat and power plant.

This is the second of a series of papers focusing on the use of the IFBB procedure on herbage (in the form of silage) from various types of semi-natural grasslands. The first paper (Wachendorf et al., 2009) gave an introduction to the IFBB procedure and the semi-natural grasslands used for this study and presented mass flows of plant compounds during mechanical dehydration. The present paper addresses the following questions:

- (i) how does mechanical dehydration and hydrothermal conditioning at different temperatures affect the chemical composition of the press fluid and the resulting biogas production,
- (ii) what methane yields can be achieved from press fluids compared to whole-crop grassland silage and what is the range of other relevant digestion parameters, i.e. degradation of organic matter (OM) and daily methane production rate, and
- (iii) do chemical constituents provide appropriate information to predict methane yields from press fluids?

## 4.2 Material and Methods

### 4.2.1 Herbage and press fluids

The herbage used in this study was collected on five semi-natural grassland sites in mountain areas of the Black Forest and Rhön, Germany, representing typical European mountain areas with two different poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*). These grassland sites were managed according to the directives of regional agri-environment schemes with one harvest annually. The chemical composition of the herbage (parent material as silage), the method of preparing the silage, the method of producing the press fluids used as fermentation substrates with the two steps of hydrothermal conditioning and mechanical dehydration, as well as the calculation of mass flows and concentrations of plant compounds, were described in detail elsewhere (Wachendorf et al., 2009).

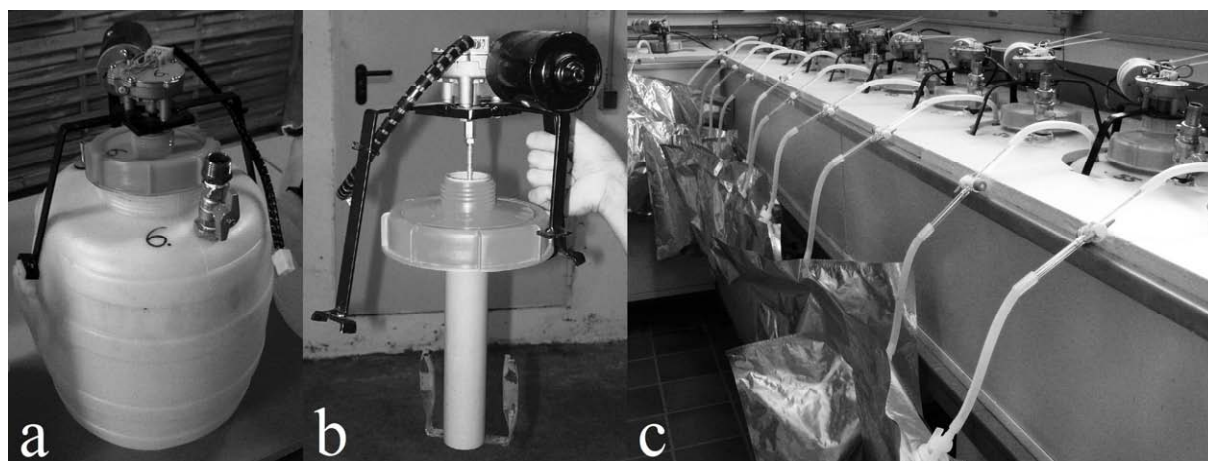
Whole-crop silages from the five grassland sites, as well as their press fluids after hydrothermal conditioning at temperatures of 5°C, 60°C and 80°C, respectively, were used as fermentation substrates in this study. Graß et al. (2009) showed in IFBB experiments with maize silage that a conditioning temperature of 60°C achieved the best results regarding the quality of press cake as the main product so that this temperature was also chosen for the present study. At temperatures above 60°C the starch began to agglutinate. As biomass of grassland herbage contains low starch concentrations, a conditioning treatment of 80°C was included in this experiment. The 5°C treatment was included as the conditioning procedure without additional heat energy input because the cold tap water, used as conditioning liquid, was of this temperature.

### 4.2.2 Anaerobic digestion experiments

Determination of biogas production was performed in batch experiments, in accordance with the German Standard (VDI 4630, 2004) and based on a method described by Zerr (2006), with two replicates (Figure 4.2). Fermentation of the substrates took place in gas-proof 20-L polyethylene containers. Mixing of the fermenter content was carried out for 15 min every 3 h with a U-shaped stirrer attached to a central shaft that was run through a dip tube. The shaft was driven by a 12 v direct current motor fixed to a tripod above the fermenter cap. The experiments were performed in a mesophile temperature range of 37°C with a fluctuation of

$\pm 1^\circ\text{C}$  continuously adjusted by temperature sensors in water basins. Gas discharge from the fermenter passed through a gas-proof nozzle on the top of the container before channelling into aluminium gas containers. At the start of the test the fermenter was filled with an inoculum of 15 kg fresh matter (FM) of digested slurry from a biogas plant with and either 800 g FM of press fluid or 400 g FM of whole-crop grassland silage.

The relationship between the organic matter (OM) in the inoculum (600 g OM) and the OM in the substrate was on average 0.02 for the press fluid (12 g OM) and 0.2 for the grassland silage (120 g OM), which is below the value of 0.5 as required by VDI 4630 (2004). Two fermenters filled with pure biogas slurry served as a control. The fermentation time was 27 d for the whole-crop grassland silage and 13 d for the press fluids. The end of the fermentation tests was determined according to VDI 4630 (2004) as the time when the daily biogas production had decreased to, proportionately, about 0.01 of the total biogas volume produced up to this time. This was in accordance with experiences with these substrates in preliminary studies (Graß et al., 2009).



**Figure 4.2** Experimental setup of anaerobic digestion tests showing (a) a 20 L polyethylene batch fermenter, (b) a stirring unit and (c) fermenters with aluminium gas bags in a water basin.

### 4.2.3 Biogas analysis, methane yield and degree of degradation

Measurement of gas fluxes started 24 h after incubation and was repeated once daily. The total daily biogas volume was determined with a wet drum gas meter (TG1, Ritter Ltd., Bochum, Germany). The biogas composition ( $\text{CH}_4$ ,  $\text{CO}_2$  and  $\text{O}_2$ ) was measured by infrared detection with a landfill gas analyser (LFG 20, Bernt Ltd., Düsseldorf, Germany). Methane volumes (L) that were measured under laboratory room conditions were converted to standard conditions (273.15 K, 101.325 kPa) and expressed as normal litre ( $\text{L}_\text{N}$ ) or normal cubic metre ( $\text{m}^3_\text{N}$ ). These methane volumes were referred to as methane yields when they were related

both to the amount of VS in the substrate ( $L_N \text{ kg}^{-1} \text{ VS}$ ) and to the grassland area harvested ( $\text{m}^3_N \text{ ha}^{-1}$ ). VS, a term of environmental chemistry, are those solids in a liquid that are lost on ignition of the dry matter at  $550^\circ\text{C}$  and equate to the OM concentration. Methane yields of the inoculum were determined and proportionately subtracted from the overall methane quantity of the incubated fermentation substrate.

The degree of degradation ( $\eta_{gas}$ ), which indicates the decomposition of OM in the substrate during anaerobic digestion and its transformation into biogas, was determined by relating the mass ( $m$ ) of  $\text{CH}_4$  and  $\text{CO}_2$  to the mass of VS in the substrate according to VDI 4630 (2004):

$$\eta_{gas} = \frac{m_{CH_4} + m_{CO_2}}{0.93 * m_{subst.} * VS}$$

where VS was set at 0.93, as it is assumed that about 0.07 would be decomposed but used for the production of bacterial biomass.

#### 4.2.4 Calculation of methane yield based on chemical constituents

According to standard guidelines (VDI 4630, 2004; FNR, 2006), biogas volume and composition were calculated based on the concentrations of the Weende constituents, crude fibre (CF), nitrogen-free extract (NFE), ether extract (EE) and crude protein (CP). Maximum methane yield was calculated with guiding values for the methane yield of the respective constituents (VDI 4630, 2004), which were derived from the stoichiometric equations of Buswell and Mueller (1952) and Boyle (1977) as follows:

$$\text{Methane yield}_{\max} = \frac{373 * (CF + NFE) + 1000.8 * EE + 560 * CP}{1000 * \frac{VS}{DM}} \quad [L_N \text{ kg}^{-1} \text{ VS}]$$

This represents the maximum potential of biogas production at complete digestion of all organic compounds. Thus, for a realistic estimation of methane yields, digestibility values (D) of the organic compounds have to be considered (VDI 4630, 2004). In this study they were based on feeding experiments with ruminants (University of Hohenheim, 1997), as this data is well-proven and provides a good indication for a system that is comparable to some extent, even if there are differences in the conditions inside the rumen and the digester. The methane yield was calculated according to:

$$\text{Methane yield} = \frac{373 * (CF * D_{CF} + NFE * D_{NFE}) + 1000.8 * EE * D_{EE} + 560 * CP * D_{CP}}{1000 * \frac{VS}{DM}} \quad [L_N \text{ kg}^{-1} \text{ VS}]$$



As digestibility data for press fluids were not available, values for corn steep liquor ( $D_{CF}$ , 0.61;  $D_{NFE}$ , 0.95;  $D_{EE}$ , 0.88; and  $D_{CP}$ , 0.87; University of Hohenheim, 1997) were used since the chemical composition for both liquids is similar.

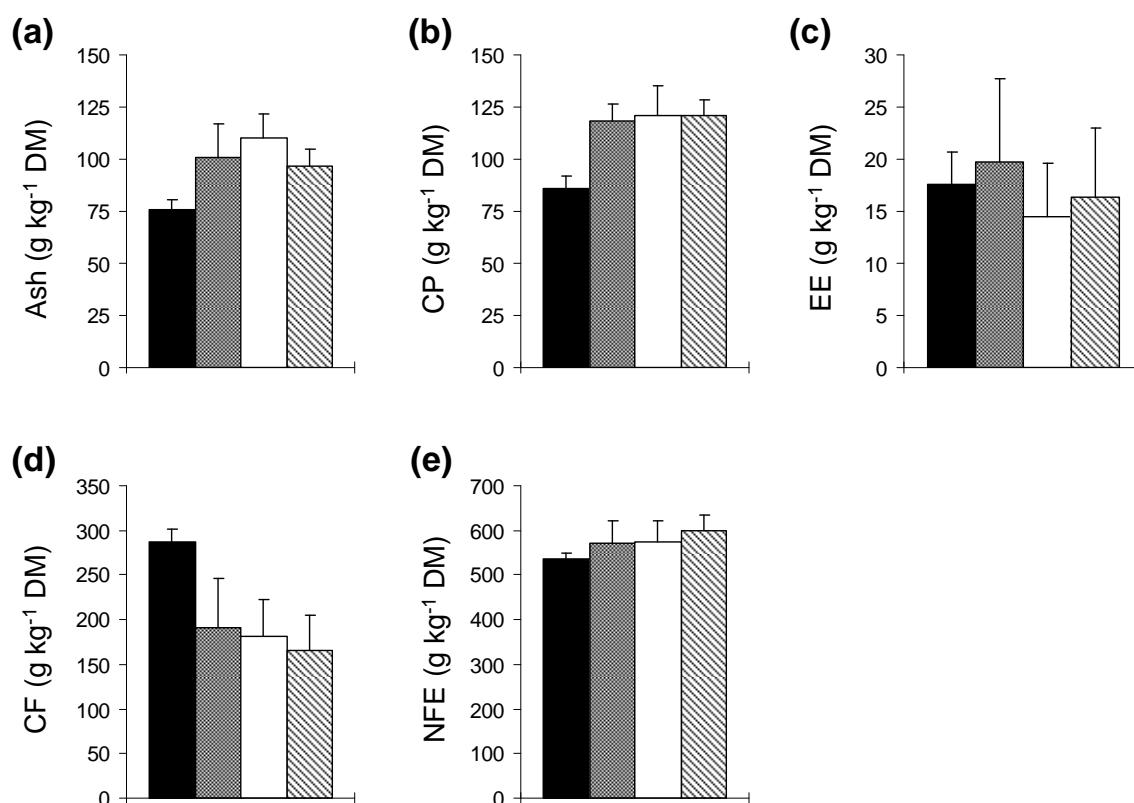
#### 4.2.5 Statistical analysis

As the main objective of this study was to quantify and to gain insight into the various processes involved in the IFBB procedure, priority was given to investigating temperature effects during conditioning, as this treatment had the most effect on mass and energy flows in preliminary studies (Reulein et al., 2007; Graß et al., 2009). Analysis of variance was performed for each Weende constituent and methane production variable, with conditioning temperature as the only factorial effect, using the procedure GLM in SAS (SAS Institute, 1996). As the three different temperature treatments were not replicated but equally applied to each of the five sward types, grassland types were considered as replicates in the models. Tukey's test was used to test for significant differences among the temperatures. Regression analyses were performed using the procedure GLM in SAS.

### 4.3 Results

#### 4.3.1 Chemical composition of the herbage (parent material as whole-crop silage) and the press fluids

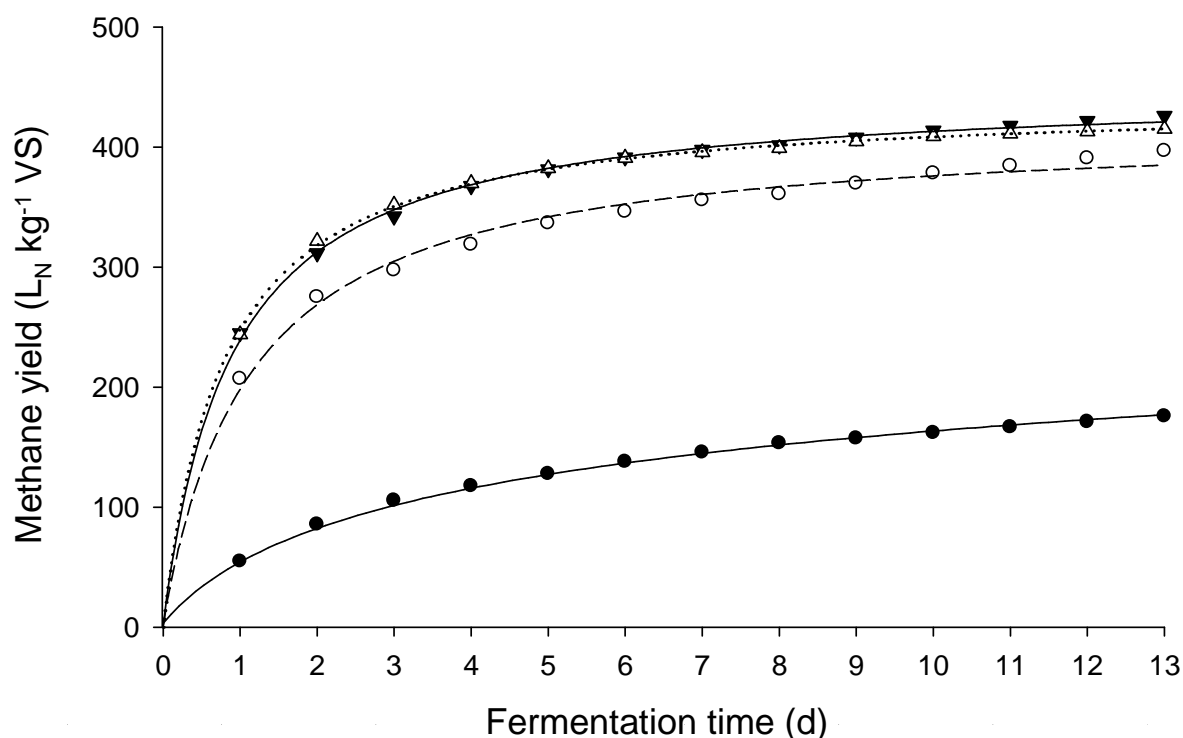
Crude protein concentrations in the press fluids were significantly ( $P < 0.05$ ) higher than in the parent material with a maximum increase when the conditioning temperature was 60°C (proportionately an increase of 0.41) prior to the mechanical dehydration (Figure 4.3). The increase in CP concentration was lowest for *Filipendulion ulmariae* and highest for *Arrhenaterion II*. Tukey's tests did not show any significant differences between the mean concentrations of ash, CF, EE and NFE in the parent material and those in the press fluids at the different conditioning temperatures. The standard errors of mean indicate a large variation among the different grassland communities. However, mean CF concentrations of the press fluids were proportionately 0.34 - 0.42 lower than those of the parent material with the largest decrease for the treatments with a conditioning temperature of 80°C. The lowest CF concentrations were found in the press fluids of *Filipendulion ulmariae* and the highest in the press fluids of *Polygono-Trisetion*.



**Figure 4.3** Mean values (with s.e. of mean) of concentrations of (a) ash, (b) crude protein (CP), (c) ether extract (EE), (d) crude fibre (CF) and (e) nitrogen free extracts (NFE) in the herbage (parent material as whole-crop silage, ■) of five semi-natural grasslands [poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)], and in the corresponding press fluids at three conditioning temperatures [5°C (■), 60°C (□), 80°C (▨)].

### 4.3.2 Methane yields and degrees of degradation

The cumulative methane production of all press fluids, irrespective of conditioning temperature, was characterised by high yields in the first 4 d of the experiment and a decreasing production in the following days, whereas the total yield of the 5°C-treatment was on average 20-30 L<sub>N</sub> kg<sup>-1</sup> VS lower than those of the 60°C or 80°C treatments (Figure 4.4). The regression curves fitted to the data of the type  $y = a - bx^{-1} + cx^{-2}$  had R<sup>2</sup> values of 0.99<sup>\*\*\*</sup>. According to these regressions 0.90 of the total methane yield had been generated by day 7 (5°C treatment), day 5 (60°C treatment) and day 4 (80°C treatment), respectively. Proportion of methane in the biogas was between 0.53 and 0.56 with a low variability among conditioning temperatures and grassland communities. Cumulative methane production in the whole-crop silage proceeded much slower, with slightly higher yields in the first 4 d of the experiment and an almost constant production until day 27 (Figure 4.4 only shows the first 13 d).



**Figure 4.4** Mean values of specific cumulative methane yield of whole-crop silages (WS, —●—), made from five semi-natural grasslands [poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)], and of the corresponding press fluids at three conditioning temperatures [5°C (PF5, --○--), 60°C (PF60, —▼—), 80°C (PF80, .....△.....)] in batch experiments with a fermentation time (FT) of 13 days. The equations of the fitted curves are: WS =  $3.375 + 76.197 \ln(\text{FT}) - 3.933 \ln(\text{FT})^2$  ( $R^2 = 0.99^{***}$ ); PF5 =  $417.671 - 462.991 \text{FT}^{-1} + 47.184 \text{FT}^{-2}$  ( $R^2 = 0.99^{***}$ ); PF60 =  $449.087 - 391.372 \text{FT}^{-1} - 56.773 \text{FT}^{-2}$  ( $R^2 = 0.99^{***}$ ); PF80 =  $438.788 - 324.498 \text{FT}^{-1} - 114.780 \text{FT}^{-2}$  ( $R^2 = 0.99^{***}$ ).

A regression model of the type  $y = a + b (\ln x) + c (\ln x)^2$  was fitted to the data with an  $R^2$  value of  $0.99^{***}$ . With the whole-crop silage, 0.90 of the total methane yield was generated by day 19. Proportion of methane in the biogas was similar to that of the press fluids. The average total methane yield was  $218 \text{ L}_N \text{ kg}^{-1} \text{ VS}$  for the whole-crop silage, with *Polygono-Trisetion* as the most productive grassland vegetation ( $268 \text{ L}_N \text{ kg}^{-1} \text{ VS}$ ) and *Filipendulion ulmariae* as the least productive one ( $158 \text{ L}_N \text{ kg}^{-1} \text{ VS}$ ) (Table 4.1). Across all grassland communities there was a close relationship for the whole-crop silage between the observed methane yields ( $y$ ) and those calculated ( $x$ ) from the chemical constituents ( $y = 0.82 x + 43.51$ ;  $R^2 = 0.93^{**}$ , RMSE = 14.59). Methane yields of the press fluids were significantly ( $P < 0.001$ ) higher than those of the whole-crop silage, with an average of  $397 \text{ L}_N \text{ kg}^{-1} \text{ VS}$  at a conditioning temperature of 5°C,  $426 \text{ L}_N \text{ kg}^{-1} \text{ VS}$  at 60°C and  $415 \text{ L}_N \text{ kg}^{-1} \text{ VS}$  at 80°C.

**Table 4.1** Observed and calculated specific methane yield ( $L_N \text{ kg}^{-1} \text{ VS}$ ) as well as degree of degradation and area-specific methane yield ( $\text{m}^3_N \text{ ha}^{-1}$ ) of whole-crop silage (WS), made from five semi-natural grasslands [poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb vegetation (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)] and of the corresponding press fluids at three conditioning temperatures (5°C, 60°C, 80°C). F values and levels of significance derived from a general linear model are also presented.

Variable	Press fluids† (conditioning temperature, °C)				RMSE‡	MSD§	F -value	Level of significance
	WS†	5	60	80				
Observed specific methane yield	217.6	396.8	425.8	415.0	63.32	114.57	12.0	***
Calculated specific methane yield	211.6	356.7	355.4	358.4	32.54	58.89	24.9	***
Degree of degradation	0.551	0.902	0.879	0.883	0.1194	0.2161	10.0	***
Area-specific methane yield	918.5	387.0	543.6	581.2	316.59	572.86	2.5	NS

†Means of five swards

‡Root mean square error

§Minimum significant distance (Tukey)

NS, not significant; \*\*\*,  $P < 0.001$

Methane yields ranged from  $304 L_N \text{ kg}^{-1} \text{ VS}$  (*Arrhenaterion* I, 5°C) to  $522 L_N \text{ kg}^{-1} \text{ VS}$  (*Arrhenaterion* II, 60°C). Methane yields calculated from the chemical constituents of the biomass were on average  $56 L_N \text{ kg}^{-1} \text{ VS}$  lower than the yields obtained by the batch tests. There was a significant relationship between the observed (y) and the calculated (x) methane yields for the combined data set of whole-crop silages and press fluids ( $y = 1.18x - 13.49$ ;  $R^2 = 0.64^{***}$ ,  $\text{RMSE} = 64.91$ ). Weak but significant ( $P < 0.05$ ) relationships existed between observed methane yield (y) and some of the organic compounds, the strongest relationship was for CP and NFE in a multiple regression analysis ( $y = 2.82\text{CP} + 7.42\text{NFE} - 0.006(\text{NFE})^2 - 2202.8$ ;  $R^2 = 0.41^*$ ,  $\text{RMSE} = 88.10$ ). In the press fluids on average 0.89 of the OM was transformed into biogas and no clear effect of temperature could be detected (Table 4.1). In the whole-crop silage the degree of degradation was significantly ( $P < 0.001$ ) lower (0.55 on average), ranging from 0.36 (*Filipendulion ulmariae*) to 0.73 (*Polygono-Trisetion*). A significant ( $P < 0.001$ ) logarithmic correlation between degree of degradation (y) and specific methane yield (x) was found for the whole data set ( $y = 52.14\ln(x) - 224.58$ ,  $R^2 = 0.88$ ,  $\text{RMSE} = 6.60$ ).

Average methane output  $\text{ha}^{-1}$  of whole-crop grassland silage was higher ( $918.54 \text{ m}^3_{\text{N}} \text{ ha}^{-1}$ ) but not significantly than for the press fluids ( $5^\circ\text{C}$ ,  $387.02 \text{ m}^3_{\text{N}} \text{ ha}^{-1}$ ;  $60^\circ\text{C}$ ,  $543.61 \text{ m}^3_{\text{N}} \text{ ha}^{-1}$ ;  $80^\circ\text{C}$ ,  $581.24 \text{ Nm}^3 \text{ ha}^{-1}$ ) (Table 4.1). On average, the press fluids generated 0.42 ( $5^\circ\text{C}$ ), 0.59 ( $60^\circ\text{C}$ ) and 0.63 ( $80^\circ\text{C}$ ) of the methane yield  $\text{ha}^{-1}$  produced by the whole-crop silages, with only 0.19 ( $5^\circ\text{C}$ ), 0.28 ( $60^\circ\text{C}$ ) and 0.29 ( $80^\circ\text{C}$ ) of the total OM of the whole-crop silages that was extracted into the press fluid through hydrothermal conditioning and mechanical dehydration. The total area-specific methane yield of the whole-crop silage ( $y$ ) varied among the different grassland communities and depended significantly ( $P < 0.01$ ) on their DM yield ( $x$ ): ( $y = 98.37x - 431.82$ ;  $R^2=0.96$ ,  $\text{RMSE}=57.74$ ).

## 4.4 Discussion

In addition to the reduction of detrimental minerals in the press cake, the IFBB procedure aims at an appropriate transfer of readily digestible organic compounds from the parent material (whole-crop silage) into the press fluid, which is used as biogas substrate, while retaining the persistent, lignified material in the press cake (Wachendorf et al., 2009).

### 4.4.1 Chemical constituents and methane production

In the present study, total methane yields of the whole-crop silage ( $158\text{-}268 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$ ) were in the same magnitude as reported by Baserga (1998) who obtained  $280 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$  from extensive grassland, as well as by Lemmer and Oechsner (2001) who generated  $240 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$  from a silage originating from extensive grassland and  $100 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$  from biomass harvested from nature conservation areas. Prochnow et al. (2005) observed a decrease in methane yields with an increasing delay in the cutting date of the grassland from  $298 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$  in June to  $155 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$  in February. The corresponding proportions of methane in the biogas of 0.58 (Baserga, 1998), 0.54 – 0.57 (Lemmer and Oechsner, 2001) and 0.52 (Prochnow et al., 2005) are consistent with those found in the present study (0.53 to 0.56) and do not differ much from those of biogas in practical digestion plants (Baserga, 1998). However, the large difference in methane yield between silages of extensive grasslands and maize (e.g.  $312\text{-}365 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$ ; Amon et al., 2007a) is a major reason for the limited use of mature grassland biomass in the biogas industry (Prochnow et al., 2005).

Mean specific methane yields of the press fluids at different conditioning temperatures ( $397\text{-}426 \text{ L}_\text{N} \text{ kg}^{-1} \text{ VS}$ ) were 0.82 ( $5^\circ\text{C}$ ) to 0.96 ( $60^\circ\text{C}$ ) higher than those of the corresponding whole-crop silages. Studies on the fermentation of press fluids are rare and the only data

known to the authors give methane yields of 573 L<sub>N</sub> kg<sup>-1</sup> VS for a liquid from a grassland silage with a fermentation time of 40 d (Kromus et al., 2000) and 506-390 L<sub>N</sub> kg<sup>-1</sup> VS for maize silage with an increasing maturity (EC79 to EC87) which remained in the fermenter for 13 d (Reulein et al., 2007) respectively. These results indicate that the specific methane yields from maize and extensive grassland are in the same magnitude when only their press fluids are digested while maize is superior to extensive grassland when a whole-crop silage is digested (Amon et al., 2007b).

A possible reason may be the comparable composition of the OM of the two press fluids and its availability to bacteria involved in biogas production. This is expressed in the high degree of degradation of the OM in the present study (0.89 on average), which is comparable to that of maize silage press fluids (0.90 on average) observed by Bühle (2007). High degrees of degradation indicate an almost entire digestion of the OM of the press fluids while the average degree of degradation of the whole-crop silage was much lower (0.55 of OM) and showed a higher variation among the grassland communities.

The increase in CP concentration in the press fluids compared to the whole-crop silage suggests an increased methane potential since proteins produce higher biogas yields with a higher proportion of methane in the biogas compared to carbohydrates (VDI 4630, 2004; FNR, 2006). Similarly, a reduction in CF concentration would have a positive effect on biogas production, since CF contains ligno-cellulose, which accumulates in plants with increasing maturity and is detrimental to anaerobic digestion. This is due to its recalcitrance to decomposition and because it encrusts proteins, fats and carbohydrates (Kerr et al., 1984), reducing the availability of those compounds to microbes and, therefore, reducing the production of biogas (Akin, 2008). Although no significant decrease in CF concentrations could be ascribed to the conditioning and dehydration, differences in methane production among the press fluids nevertheless may be related to changes in the composition concentrations of CF and NFE, as they account for 0.66 of the OM both in the whole-crop silage and in the press fluids, whereas EE concentrations were lower in both cases. Since NFE is calculated from the other Weende constituents (ash, CP, EE and CF), it comprises the errors of every single analysis and the heterogeneity within this fraction can be very high (Ely et al., 1953). Furthermore, it may contain variable amounts of lignin depending on the exact CF method used (Van Soest, 1967).

Specific methane yields of the press fluids from both *Arrhenaterion* grasslands at the higher conditioning temperatures (60°C and 80°C) were considerably higher than those at the

5°C level, suggesting changes in the chemical composition brought about by increased temperatures during conditioning. Beside an enhanced maceration of cell wall material, the degradation of long-chain fatty acids, which may inhibit growth and activity of microorganisms in the fermenter (Shin et al., 2003), into volatile fatty acids, which augment the biogas production, may have occurred through the conditioning with hot water (Neyens and Baeyens, 2003). However, this did not occur across all grassland communities and even an inverse relationship occurred for *Caricion fuscae*, where the highest specific methane yield was obtained with the 5°C treatment. Amon et al. (2007b) reported that calculations using the methane energy value model (MEVM) produced good results for maize and cereals, but further refinement was necessary for grassland biomass. Thus, in order to obtain more insight into the rather complex relationship between the chemical composition and the methane production from press fluids, as well as into the effects of different conditioning and dehydration procedures, further investigation based on a diverse body of data from different grassland communities is required.

#### **4.4.2 Rates of digestion and consequences for the digestion technique**

Methane production of the press fluids was characterised by high rates in the first days of digestion (e.g. on average 109 and 47 L<sub>N</sub> kg<sup>-1</sup> VS d<sup>-1</sup> on days 1 and 2, respectively) and a rapid decline thereafter. While an increase in temperature from 5°C to 60°C during conditioning enhanced the overall methane production, although not significantly, it did not modify the general pattern of the curves. Comparable curve progressions were reported for press fluids from maize silage at different stages of maturity (Reulein et al., 2007). At the time when 0.90 of the total methane yield was produced, the production rate was on average 10.7 L<sub>N</sub> kg<sup>-1</sup> VS d<sup>-1</sup>, which was close to the average production rate of 10.6 L<sub>N</sub> kg<sup>-1</sup> VS d<sup>-1</sup> on day 5 in this study. In contrast, daily methane production rates in the whole-crop silage were lower with values of 35 and 23 L<sub>N</sub> kg<sup>-1</sup> VS d<sup>-1</sup> on days 1 and 2, respectively, and a more gentle decrease of the curve, which agrees with other studies that have used whole-crop silages from grassland and cereals (Amon et al., 2004; Schumacher et al., 2007).

With a screen perforation of 1.5 mm in the screw press, particles of that size or smaller were transferred into the press fluid. Vetter and Reinhold (2005) have shown that the digestibility of a substrate, and thus its methane potential, is related to the particle size in the material. They obtained a doubling of methane yields from maize silage with a particle size of 3 mm compared to common whole-crop maize silage and increased methane production rates in the

first days of the experiment. The physical and chemical characteristics of the press fluids in this study resemble waste-water and sewage sludge rather than common agricultural biomass used for biogas production. Particularly, the low volatile solids contents (8-24 g L<sup>-1</sup>), which are in the same range as in waste activated sludge (5-50 g L<sup>-1</sup>; Sayed and Fergala, 1995; Roberts et al., 1999), suggest the application of fermenter techniques similar to those in waste-water treatment plants. Unlike conventional fermenters, where stirring results in the discharge of a large proportion of the microbial biomass with the digestate after a hydraulic retention time (HRT) of at least 20 days, the technique for the treatment of low-solid liquids and sludges must guarantee the retention of active bacteria, as the time needed for reproduction is longer than the retention time of the substrate in the fermenter (Edelmann, 2001). This can be achieved either by the formation of highly settleable sludge aggregates combined with gas separation and sludge settling (e.g. upflow anaerobic sludge blanket (UASB) reactors) or by the entrapment of sludge aggregates between filter material supplied to the fermenter (e.g. fixed film reactors) (Rajeshwari et al., 2000).

The methane yield and the fermenter volume of continuous digesters are influenced by the organic loading rate (OLR) and the HRT of the substrate. The recommended OLR for whole-crop silage is around 3 kg VS m<sup>-3</sup> d<sup>-1</sup>, and somewhat lower for readily digestible substrates, in order to avoid instability of the fermentation process as a result of acidification (FNR, 2005). A press fluid of a continuous IFBB plant in practice would have proportionately higher VS contents of about 0.15 due to the circulation of the conditioning liquid. As an example, such a press fluid with an OLR of 1.5 kg VS m<sup>-3</sup> d<sup>-1</sup> and a HRT of 15 d would need a fermenter volume of 150 L compared to grassland whole-crop silage with a proportionately higher VS content of 0.25, an OLR of 3 kg VS m<sup>-3</sup> d<sup>-1</sup> and a HRT of 62.5 days (30-90 d is common in practice; FNR, 2005) that would need a fermenter volume of 1000 L. Thus, the IFBB procedure would imply the construction of much smaller fermenters at considerably lower costs.

#### **4.4.3 Area-specific methane yields**

Methane yields ha<sup>-1</sup> of the whole-crop grassland silages were higher than those of the press fluids, which is due to the fact that only ca. 0.29 of the DM of the silages were transferred into the liquid phase during dehydration (Wachendorf et al., 2009). Prochnow et al. (2005) obtained methane yields of 894 and 1587 Nm<sup>3</sup> ha<sup>-1</sup> with grass cut from landscape management in July and August, respectively, which is comparable to the average methane



yield of whole-crop silage used in this study ( $1040 \text{ m}^3_{\text{N}} \text{ ha}^{-1}$ ). However, methane yields  $\text{ha}^{-1}$  of maize were up to  $4400\text{-}10000 \text{ Nm}^3 \text{ ha}^{-1}$ , as observed by Oechsner et al. (2003) and  $3743\text{-}8529 \text{ Nm}^3 \text{ ha}^{-1}$  as reported by Amon et al. (2004). It is remarkable that press fluids from maize silage achieved 0.33 of the methane yield of maize whole-crop silage (Graß et al., 2009) whereas press fluids from semi-natural grassland had an average value of 0.56 of the methane yield of the whole-crop silage. This suggests that the IFBB process achieves relatively higher methane yields  $\text{ha}^{-1}$  with biomass of a low digestibility than with more highly digestible crops, which also produce high yields in a whole-crop digestion.

In case of the five semi-natural grasslands used in this study, a conversion through anaerobic digestion of whole-crop silage and a subsequent combustion of the biogas in a combined heat and power plant (CHP) with an electrical efficiency of 0.37 and a thermal efficiency of 0.48 would generate  $3.39 \text{ MWh ha}^{-1}$ , as electricity, and  $4.40 \text{ MWh ha}^{-1}$ , as heat. With a degree of utilization of the waste heat of 0.30, which is a guiding value from German commercial biogas plants (FNR, 2005), the total energy output would be  $5.59 \text{ MWh ha}^{-1}$ , of which 0.61 would be as electricity. With a conversion through the IFBB procedure at a conditioning temperature of  $60^\circ\text{C}$  and a complete utilization of the waste heat for the drying of the press cake, the total energy output would be  $4.61 \text{ MWh ha}^{-1}$ , of which 0.44 would be as electricity. However, it has to be considered, that, in the case of IFBB, a major proportion of the crop energy (0.81, 0.72 and 0.71 of the total OM in the  $5^\circ\text{C}$ ,  $60^\circ\text{C}$  and  $80^\circ\text{C}$  treatments, respectively) is bound in the press cake.

Although the energy input of the IFBB procedure (i.e. electricity for the screw press and heat for the hydrothermal conditioning), which accounts for about 0.06 of the total energy output according to Graß et al. (2009), is higher compared to a conventional whole-crop digestion, the total energy output is superior. Thus, to identify the optimum IFBB procedure and compare it to conventional conversion techniques, the total energy yield both from press fluid and press cake, as well as the internal energy demands for hydrothermal conditioning, mechanical dehydration, drying and pelleting of the press cakes have to be determined in detail, which will be reported in the third paper of the series.

## 4.5 Conclusions

Based on anaerobic digestion experiments in batch fermenters with press fluids and whole-crop silages from five typical semi-natural grasslands in German mountain areas, the following conclusions can be drawn:

(i) Compared to the corresponding grassland silages, press fluids generated through hydrothermal conditioning and subsequent mechanical dehydration showed higher concentrations of readily digestible CP (increase of 0.40 on average) and lower concentrations of persistent CF (non-significant decrease of 0.37 on average), corresponding to the superior methane production measured in anaerobic digestion experiments.

(ii) In anaerobic digestion experiments with a fermentation time of 13 d, methane generation in press fluids was extremely high in the first few days, reaching 0.90 of the total methane yield (397, 426 and 415 L<sub>N</sub> kg<sup>-1</sup> VS at 5°C, 60°C and 80°C, respectively) in 7, 5, and 4 ds at 5°C, 60°C and 80°C, respectively, and levelling off in the remaining time of the experiment. By then on average 0.89 of the OM in the press fluids was transformed into biogas. In comparison, the corresponding whole-crop grassland silages had methane yields of 218 L<sub>N</sub> kg<sup>-1</sup> VS with a mean degree of degradation of 0.55 of OM. On average press fluids achieved 0.56 of the methane yields ha<sup>-1</sup> of their corresponding whole-crop silages (661, 803 and 897 m<sup>3</sup><sub>N</sub> ha<sup>-1</sup> at 5, 60 and 80°C, respectively) while only 0.25 of the OM of the whole-crop silages was transferred into the press fluids.

(iii) Chemical constituents provided good information to predict methane production for the whole-crop grassland silage but underestimated the methane potential of the press fluids by 56 L<sub>N</sub> kg<sup>-1</sup> VS, on average.

## **5 Utilization of semi-natural grassland through integrated generation of solid fuel and biogas from biomass. III. Effects of hydrothermal conditioning and mechanical dehydration on solid fuel properties and on energy and greenhouse gas balances**

**Abstract** The IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass) procedure separates biomass into a readily digestible press fluid, from which biogas is produced and a fibrous press cake that is used as solid fuel. The effects of mechanical dehydration and prior hydrothermal conditioning (5, 60 and 80°C) on biomass from five species-rich, semi-natural grasslands, typical of mountain areas of Germany were investigated. Proportional reduction of ash constituents in the press cake compared to the parent material was up to 0.80, 0.61, 0.81 for potassium, magnesium and chloride, respectively, at 60°C, resulting in potassium, magnesium and chloride concentrations in the press cake of 2.43, 1.22 and 0.93 g kg<sup>-1</sup> dry matter (DM). Emission-relevant constituents were reduced by up to 0.19 (nitrogen) and 0.39 (sulphur), yielding in nitrogen and sulphur concentrations of 11.13 and 0.97 g kg<sup>-1</sup> DM, respectively. Ash softening temperatures were significantly increased up to 1250°C, falling within the range of wood fuels. Thus, quality of IFBB fuels is superior compared to conventional hay and comparable to hay of delayed harvest in winter or next spring. Calculated energy conversion efficiency for IFBB was up to 0.51, compared to a maximum of 0.22 for anaerobic whole crop digestion (WCD) and 0.74 for combustion of hay (CH). High energy demands in IFBB resulted in a GHG mitigation potential of up to -4.40 t CO<sub>2</sub>eq ha<sup>-1</sup> which is lower than for CH (up to -6.17 t CO<sub>2</sub>eq ha<sup>-1</sup>), but higher than for WCD which mitigated up to -2.24 t CO<sub>2</sub>eq ha<sup>-1</sup>.

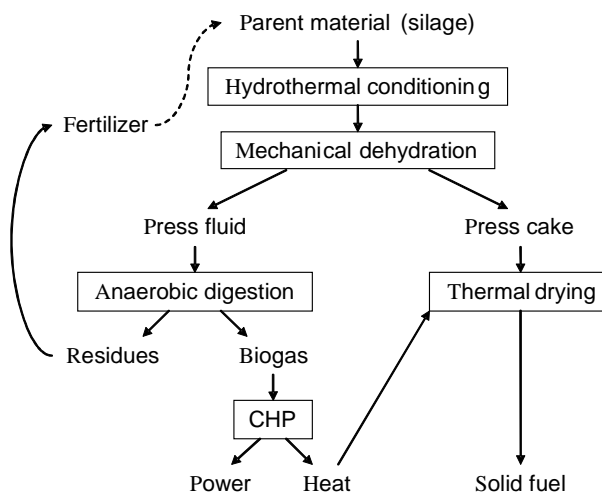
### **5.1 Introduction**

In view of the global climate change debate and the European Union's requirements regarding environment protection, a number of issues evolved that require solutions from an agricultural perspective. There is the question of efficient energy production from agricultural land, greenhouse gas (GHG) mitigation and the competition with food production on a finite land resource. A characteristic for many common bioenergy techniques is a relatively low conversion efficiency of the available energy contained within the biomass into a higher grade

form with greater utility, e.g. anaerobic digestion (conversion efficiency into electricity, 0.10-0.16; McKendry, 2002) or combustion (conversion efficiency, 0.2-0.4; McKendry, 2002). High GHG savings can only be obtained in a bioenergy system if the energy output is significantly higher than the energy input. The increasing production of bioenergy on arable land might lead to a competition with food production. This potential conflict could be reconciled by reduced meat consumption or by an intensified use of residual material for bioenergy production (Bergsma et al., 2007).

Feeding patterns in modern livestock farming have changed from species-rich forage obtained from semi-natural grassland with low energy content to an increased use of arable forage crops and concentrates, leaving vast areas of grassland without management. Under central European conditions this abandonment causes, in the course of natural succession, the formation of woods and shrubs and results in a dramatic loss of species (Poschlod et al., 2005). In order to conserve these ecologically valuable habitats, new utilization concepts for the biomass are needed. The production of energy could be a creative solution, providing the opportunity to utilize a renewable energy source without competing with food production, with the accompanying regular management of the grassland swards.

The development of the integrated generation of solid fuel and biogas from biomass (IFBB, Wachendorf et al., 2009) is aimed at increasing the efficiency of converting biomass into energy. Biomass, e.g. from semi-natural grasslands, which is difficult to exploit in conventional bioenergy converting systems, as the chemical composition is detrimental both for conventional anaerobic digestion due to high fibre concentrations (Prochnow et al., 2005) and for direct combustion due to high concentrations of elements that cause corrosion inside the combustion chamber, ash softening and hazardous emissions (Oberberger et al., 2006). In the IFBB procedure (Figure 5.1) the grassland biomass is subjected to hydrothermal conditioning and a subsequent separation using a screw press into a press fluid for biogas production and a press cake for direct combustion as solid fuel (Richter et al., 2009b; Wachendorf et al., 2009). With the need to dry the press cake to DM concentrations of 850 g kg<sup>-1</sup> FM in order to make it suitable for pelleting and storage, the IFBB system provides a year-round demand for heat produced in the CHP, where the biogas is combusted.



**Figure 5.1** Flow chart of the IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass) procedure. CHP refers to a combined heat and power plant.

By using biomass from semi-natural grasslands as a substrate for IFBB instead of arable crops, competition with food production will be limited. In addition, the management of biodiversity within semi-natural grasslands could eventually become economically viable since many European countries are struggling to ensure grassland conservation due to the high specific costs for harvesting and the low quality of the forage for ruminants (Isselstein et al., 2005).

This paper is the third of a series of three papers investigating the processing of biomass from various types of semi-natural grassland in the IFBB procedure, addressing the following questions:

- (i) how does mechanical dehydration and hydrothermal conditioning at three different temperatures affect the chemical composition of the press cake with regard to its quality as solid fuel,
- (ii) how high is the net energy yield and the conversion efficiency of the IFBB procedure when compared to conventional anaerobic digestion of whole-crop silage and to combustion of hay from the parent grassland material, and
- (iii) how effective is the IFBB procedure at mitigating greenhouse gases when compared to conventional anaerobic digestion of whole-crop silage and combustion of hay?

## 5.2 Material and Methods

### 5.2.1 Herbage and press cakes

The herbage used in this study was collected on five semi-natural grassland sites in mountain areas of the Black Forest and Rhön, Germany, representing typical European mountain areas with two different poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*). These grassland sites were managed according to the directives of regional agri-environment schemes with one late harvest annually. In 2006, when the experiment was conducted, the two *Arrhenaterion*, the *Caricion fuscae* and the *Filipendulion ulmariae* grasslands were harvested on 31 August, while the *Polygono-Trisetion* grassland was harvested on 19 July. The chemical composition of the herbage (parent material as silage), the method of preparing the silage, the method of producing the press cakes including hydrothermal conditioning and mechanical dehydration, as well as the methods of determining the concentrations of plant compounds were described in detail elsewhere (Wachendorf et al., 2009).

### 5.2.2 Calculation of fuel quality parameters

The ash softening temperature (AST) as an important indicator to describe the ash melting behaviour during combustion was calculated based on the concentrations of K, Ca and Mg (g kg<sup>-1</sup> dry matter, DM) for the parent material and the press cakes according to Hartmann (2001):

$$AST = 1172 - 5.39 * K + 25.27 * Ca - 78.84 * Mg \quad [^{\circ}C]$$

The higher heating value (HHV) of the parent material and the press cakes was calculated based on the concentrations of C, H and N (g kg<sup>-1</sup> DM) using the following empirical equation developed by Friedl et al. (2005) for biofuels:

$$HHV = 0.0355 * C^2 - 23.2 * C - 223 * H + 0.512 * C * H + 13.1 * N + 20600 \quad [kJ \text{ kg}^{-1} \text{ DM}]$$

C, H, N and O of the press cake and the parent material were analysed using an elemental analyzer (EA 1106, Carlo Erba Ltd., Rodano, Italy).

### 5.2.3 Energy balance

In order to assess the IFBB procedure with regard to its energy balance, it was compared to conventional anaerobic digestion of the untreated whole-crop silage (whole-crop digestion, WCD) and combustion of hay (CH). As the energy balance of a conventional biogas plant is heavily dependent on the degree of utilization of the waste heat from the combined heat and power plant (CHP), four different WCD scenarios were considered. The scenarios cover the utilization of the entire waste heat (1) and of no waste heat (0), as well as two more common situations with utilization of 0.2 (guiding value of practical biogas plants; FNR, 2005) and 0.5 of the waste heat. With respect to CH, an important limitation is the extent of biomass losses during field-curing. This is mainly weather dependent, resulting in a wide range of reported values in the literature for DM losses in the field (McGechan, 1989). Therefore three different scenarios were chosen for a conventional hay harvest, ranging from a total loss of the DM of 0.12 (good weather conditions), 0.18 (medium weather conditions) to a DM loss of 0.3 (bad weather conditions; KTBL, 2005). A fourth CH scenario was included which is based on the data of an experiment with delayed harvest (in February) of three different semi-natural grassland swards with a mean DM loss of 0.54 (Tonn et al., 2007). The DM losses in the WCD and IFBB systems due to microbial activities during the ensiling process were estimated to be 0.12 (KTBL, 2006).

The system boundaries for the energy balance included the cultivation, harvest and transport of the grassland biomass, the conversion of the biomass into useful energy and the return of the residual material (digestate, coarse ash) on agricultural land. It also included the indirect energy input for the provision of the technical infrastructure (agricultural machinery, energy conversion plants, buildings). The cultivation and harvest of the biomass in the IFBB and WCD scenarios comprised grass harrowing, mowing, turning, swathng, recovering the biomass with a field-chopper, transporting and ensiling the biomass in a bunker silo once per year. In the CH scenarios the operations were once per year grass harrowing, mowing, recovering the biomass with a round baler and transporting the bales, two times tedding and swathng and three times turning. No ploughing or re-seeding was assumed as this is prohibited on these grasslands due to legal directives. The distance from the farm to the field was assumed to be 4 km and from field to field 2 km.

Net energy yield per hectare grassland was calculated as the difference between energy input and energy output for every single grassland site and a mean value was calculated afterwards. Energy input parameters used for the calculations are shown in Table 5.1. As the design

layout of the conditioning unit, press, digesters and other technical components and infrastructures in a commercial IFBB plant is yet not known, implementation of exact figures for the indirect energy input is not possible at this stage. Several studies have shown that the indirect energy input for bioenergy plants is less than 0.1 of the direct energy input (GEMIS, 2009; Lootsma, 2006; Bachmaier et al., 2008). Therefore 0.1 of the direct energy input, divided evenly on electrical and heat energy, was assumed in this study. Because of the higher direct energy input of IFBB, this estimate takes into account the probably higher indirect energy input for IFBB compared to the other conversion systems.

**Table 5.1** Energy input parameters for an area-specific energy balance calculation of the anaerobic digestion of whole-crop silage (WCD), the combustion of hay (CH) and the IFBB procedure.

Energy input parameters	WCD	CH	IFBB	Source
Diesel:				
cultivation, harvest, transport	49.02 kWh t <sup>-1</sup> FM	40.63 kWh t <sup>-1</sup> FM	46.79 kWh t <sup>-1</sup> FM	KTBL (2006)
transport of pellets (30 km)	---	6.45 kWh t <sup>-1</sup> FM	6.45 kWh t <sup>-1</sup> FM	Bühle (2008)
Electricity:				
screw Press	---	---	14 kWh t <sup>-1</sup> FM silage	Bühle (2008)
biogas plant operation	15.16 kWh t <sup>-1</sup> FM silage	---	0.45 kWh t <sup>-1</sup> FM press fluid	Bühle (2008)
drying blower for press cakes	---	---	6.94 kWh t <sup>-1</sup> FM	Bühle (2008)
pelleting of hay/press cakes	---	113 kWh t <sup>-1</sup> FM	113 kWh t <sup>-1</sup> FM	Sokhansanj and Fenton (2006)
Heat:				
hydrothermal conditioning, 60°C	---	---	58 kWh t <sup>-1</sup> FM silage	Own Calculations
hydrothermal conditioning, 80°C	---	---	80 kWh t <sup>-1</sup> FM silage	Own Calculations
heating of fermenter	27 kWh t <sup>-1</sup> FM silage	---	35 kWh t <sup>-1</sup> FM press fluid	Own Calculations
drying of press cakes†	---	---	398 - 500 kWh t <sup>-1</sup> FM	Lootsma and Raussen (2008)

†DM concentration of fresh press cake after mechanical dehydration: 464-543 g kg<sup>-1</sup> FM, target DM concentration of dry press cake: 850 g kg<sup>-1</sup> FM



The heat energy demand ( $Q$ ) for hydrothermal conditioning of a certain quantity of silage ( $m = 1 \text{ g}$ ) was calculated based on the specific heat capacity of the silage ( $c = 3.6 \text{ J } ^\circ\text{C}^{-1} \text{ g}^{-1}$ ) and the change in temperature ( $\Delta T$ ) between the silage in the bunker silo (mean temperature of  $10^\circ\text{C}$  throughout the year) and during hydrothermal conditioning ( $60$  or  $80^\circ\text{C}$ ) according to:

$$\frac{Q}{m} = c * \Delta T$$

In a commercial IFBB plant the water used as conditioning liquid will be captured after conditioning in an insulated sedimentation tank, recycled and used again as conditioning liquid under the assumption that the loss in temperature is no more than  $2^\circ\text{C}$ , so that an additional energy demand for heating the water ( $c = 4.1826 \text{ J } ^\circ\text{C}^{-1} \text{ g}^{-1}$ ) might be necessary.

Additional losses as heat transmission of 0.1 of the calculated heat demand were assumed and the heat required by the fermenter was calculated accordingly. The fermentation temperature in the WCD and IFBB scenarios was  $37^\circ\text{C}$ . In the IFBB process the fermenter for the digestion of press fluids needs only to be heated for the  $5^\circ\text{C}$  treatment, as the continuous inflow of warm press fluid in the  $60^\circ\text{C}$  and  $80^\circ\text{C}$  treatments provides enough heat for the fermentation in the mesophilic temperature range. If necessary, the press fluids can be cooled down in a practical IFBB plant without additional energy input by heat exchangers. Calculation of the energy output implies the combustion of the biogas from the press fluid in a CHP with the generation of heat and electricity. Methane yields used for the calculation were based on batch experiments described by Richter et al. (2009b) and reduced by 0.1 to correct for up-scaling effects from lab-scale to plant scale (Table 5.2).

Electrical efficiency of the CHP was estimated to be 0.37, while thermal efficiency was estimated to be 0.48, based on manufacturer information for biogas CHPs (Scholwin et al., 2006). Thermal energy from the combustion of biogas in the IFBB procedure is completely used as heat source for drying the press cake. However, the heat demand for drying tend to be higher than the generated waste heat from the CHP (Bühle, 2008), so that a commercial IFBB plant would probably comprise a biomass combustion furnace using press cake as solid fuel. The third output parameter is the amount of energy produced as heat from the combustion of the press cakes, which was calculated based on the HHV with a thermal efficiency during combustion of 0.9. The energy conversion efficiency of each conversion technique (IFBB, WCD, CH) was determined as the ratio of the net energy yield and the gross energy yield of the parent grassland biomass, with gross energy yield as the product of DM yield ( $\text{kg ha}^{-1}$ ) and HHV ( $\text{MJ kg}^{-1}$ ).

**Table 5.2** Energy output parameters for an area-specific energy balance calculation of the anaerobic digestion of whole-crop silage (WCD), the combustion of hay (CH) and the IFBB procedure.

Conversion technique	Electricity from Biogas* (MWh ha <sup>-1</sup> )	Heat from Biogas* (MWh ha <sup>-1</sup> )	Heat from solid fuel* (MWh ha <sup>-1</sup> )
WCD_1	2.46 - 4.46	3.19 - 5.79	---
WCD_0.5	2.46 - 4.46	1.59 - 2.89	---
WCD_0.2	2.46 - 4.46	0.64 - 1.16	---
WCD_0	2.46 - 4.46	---	---
CH_0.12	---	---	13.03 - 41.54
CH_0.18	---	---	12.14 - 38.71
CH_0.30	---	---	10.37 - 33.04
CH_0.54	---	---	6.81 - 21.71
IFBB_5°C	0.48 - 2.92	0.62 - 3.79	10.86 - 28.81
IFBB_60°C	1.04 - 3.38	1.35 - 4.39	9.17 - 30.03
IFBB_80°C	0.89 - 4.21	1.15 - 5.46	9.10 - 26.35

WCD, anaerobic digestion of whole crop silage with proportion of utilisation of the waste heat; CH, combustion of hay with proportion of dry matter loss in the field; IFBB, IFBB process with temperature of hydrothermal conditioning

\*Values cover the whole range of the five semi-natural grasslands of this study, biogas yields are based on Richter et al. (2009b)

#### 5.2.4 Greenhouse gas balance

Potential GHG mitigation (t CO<sub>2</sub>eq ha<sup>-1</sup>) of IFBB, WCD and CH was calculated as the difference between the GHG emissions (resulting from energy input parameters and methane losses during the biogas process) and the GHG savings by the substitution of fossil energy sources with electricity and heat generated from the respective biomass conversion. The system boundaries were the same as for the energy balance. Nitrous oxide (N<sub>2</sub>O) emissions from returning the digestate to the field, as well as soil borne emissions, were not considered as a considerable uncertainty exists in the literature data (Berenz, 2008). This may lead to a slight over-estimation of GHG mitigation for WCD and IFBB. According to GEMIS (2009), GHG savings were based on emission factors for Diesel of 309 g CO<sub>2</sub>eq kWh<sup>-1</sup>, the German electricity mix in 2005 for the substitution of electricity (589 g CO<sub>2</sub>eq kWh<sup>-1</sup>) and on a mix of 0.42 fuel oil and 0.58 natural gas for the substitution of heat energy (326 g CO<sub>2</sub>eq kWh<sup>-1</sup>).

Methane emissions during the fermentation process and during biogas combustion in the CHP were assumed to be 2 % of the total methane production (Bachmaier et al., 2008). N<sub>2</sub>O emissions during combustion of press cake pellets in the IFBB scenarios and of hay pellets in the CH scenarios were based on combustion experiments of various herbaceous fuels with an average value of 0.0576 g N<sub>2</sub>O kWh<sup>-1</sup> (Heinz et al., 1999). The equivalence factors for the contribution to the global warming potential were 1 for CO<sub>2</sub>, 25 for CH<sub>4</sub> and 298 for N<sub>2</sub>O according to IPCC (2007).

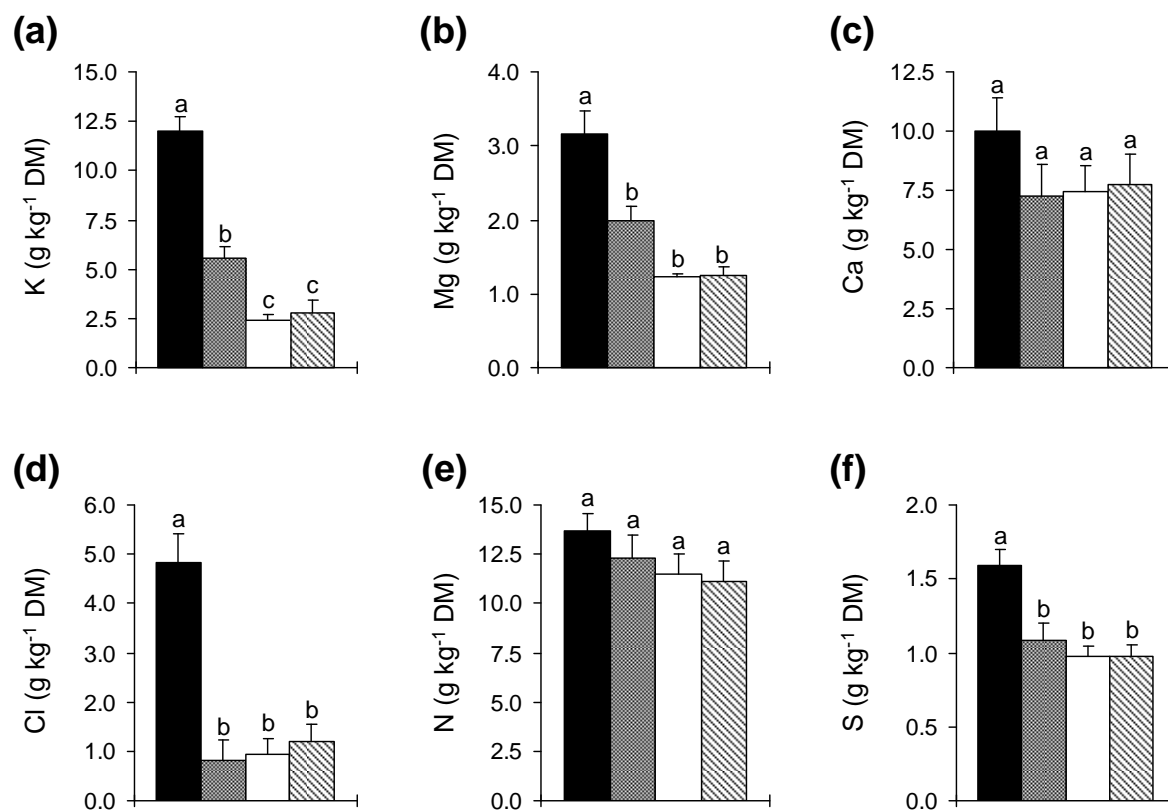
### 5.2.5 Statistical analysis

As the main objective of this study was to quantify and to gain insight into the various steps involved in the IFBB process, priority was given to investigating temperature effects during conditioning, as this treatment proved most effective on mass and energy flows in preliminary studies (Reulein et al., 2007; Graß et al., 2009). Analysis of variance was performed for each element, the AST and the HHV with conditioning temperature as the only factorial effect using the procedure GLM in SAS (SAS Institute, 1996). As the three different temperature treatments were not replicated but equally applied to each of the five sward types, grassland types were considered as replicates in the models. Tukey's test was used to test for significant differences among the temperatures. Analysis of variance was also performed for net energy yield, conversion efficiency and GHG mitigation with the conversion system as factorial effect, the five grassland swards as replicates and Tukey's test to test for significant differences.

## 5.3 Results

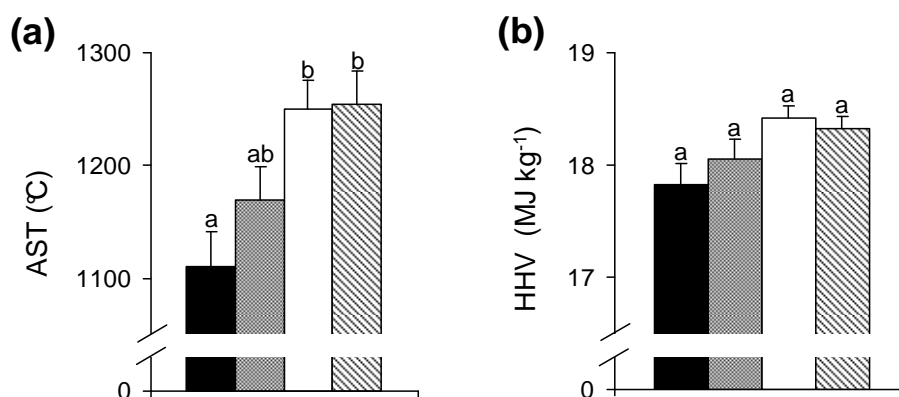
### 5.3.1 Chemical composition and fuel quality of press cakes

K, Mg and Cl concentrations in the press cakes were significantly ( $P < 0.001$ ) lower than in the parent material with a maximum proportional reduction of 0.80 (60°C), 0.61 (60°C) and 0.83 (5°C), respectively (Figure 5.2). A significant ( $P < 0.01$ ) reduction in S concentrations in the press cakes was also detected, with a maximum of 0.39 at 60°C. With regard to Ca and N concentrations, ANOVA did not reveal any significant differences between the mean values of the parent material and the different conditioning temperatures. However, a slight proportional decrease in the press cake of up to 0.19 for N and 0.27 for Ca was observed.



**Figure 5.2** Mean values (with s.e. of mean) of concentrations of (a) K, (b) Mg, (c) Ca, (d) Cl, (e) N and (f) S in the herbage (parent material, ■) of five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)), and in the corresponding press cakes after treatments of different temperatures during hydrothermal conditioning [5°C (■), 60°C (□), 80°C (▨)]. Mean values with different letters indicate significant differences ( $P < 0.05$ ).

The higher temperature treatments (60 and 80°C) resulted in a greater reduction in the concentration of K and Mg when compared to the 5°C treatment, however this was, only significant ( $P < 0.05$ ) for K. Only minor differences were observed between the remaining element concentrations at the different treatment temperatures. As a result of the significantly reduced K and Mg concentrations in addition to the slightly reduced Ca concentration, the AST was significantly ( $P < 0.05$ ) higher in the 60°C and 80°C press cakes (up to 1254°C) than in the parent material (1111°C) (Figure 5.3a). The mean increase was proportionally 0.05 for the 5°C treatment and 0.13 for the 60°C and 80°C treatments, respectively. No significant difference between the HHV of the parent material and the press cakes at different conditioning temperatures was determined in Tukey's test (Figure 5.3b). HHV ranged between 17.82 (parent material) and 18.41 (60°C) MJ kg<sup>-1</sup>. However, the treatments at 60°C and 80°C conditioning temperature show a slight increase of 0.04 and 0.03, respectively.



**Figure 5.3** Mean values (with s.e. of mean) of (a) ash softening temperature (AST) and (b) higher heating value (HHV) in the herbage (parent material, ■) of five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)), and in the corresponding press cakes after treatments of different temperatures during hydrothermal conditioning [5°C (■), 60°C (□), 80°C (▨)]. Mean values with different letters indicate significant differences ( $P < 0.05$ ).

### 5.3.2 Energy and greenhouse gas balance

The gross energy yield of the different grassland vegetation types was between 16.5 (*Polygono-Trisetion*) and 52.5 MWh ha<sup>-1</sup> (*Filipendulion ulmariae*) with a mean value of 28.0 MWh ha<sup>-1</sup>. The highest net energy yield was obtained from CH with 20.67 MWh ha<sup>-1</sup> at a DM loss of 0.12, while the best-case scenario for WCD yielded 5.51 MWh ha<sup>-1</sup> with the accompanying utilization of all the waste heat (Table 5.3). The lowest net energy yield for CH was obtained at a DM loss of 0.54 (10.80 MWh ha<sup>-1</sup>) and for WCD without any utilization of waste heat (1.56 MWh ha<sup>-1</sup>). The IFBB conversion achieved the highest net energy yield of 14.10 MWh ha<sup>-1</sup> for the 60°C treatment, followed by 13.48 MWh ha<sup>-1</sup> for the 80°C treatment and 11.93 MWh ha<sup>-1</sup> for the 5°C treatment. Although the IFBB procedure requires a high input of electricity, the net energy yield in the form of electric energy was 0.93, 0.78 and 0.15 MWh ha<sup>-1</sup> at 80°C, 60°C and 5°C, respectively, which can be fed directly into the national grid. The net electric energy yield of WCD was 2.76 MWh ha<sup>-1</sup> for all four scenarios. CH had a negative net electric energy yield of -0.38 to -0.72 MWh ha<sup>-1</sup> due to a demand for electric energy input and an absence of electric energy output. The mean energy conversion efficiency ranged from 0.07 (no utilization of the waste heat) to 0.22 (entire utilization of the waste heat) for WCD and from 0.39 (0.54 DM loss) to 0.74 (0.12 DM loss) for CH. The IFBB procedure was intermediate with values of 0.43, 0.51 and 0.48 for the 5°C, 60°C and 80°C treatment, respectively.

**Table 5.3** Net energy yield ( $\text{MWh ha}^{-1}$ ), conversion efficiency of energy and green house gas (GHG) mitigation ( $\text{t CO}_2\text{eq ha}^{-1}$ ) of anaerobic digestion of whole-crop silage (WCD) at four levels of waste heat utilization, combustion of hay (CH) at four levels of DM losses and IFBB conversion at three conditioning temperatures ( $5^\circ\text{C}$ ,  $60^\circ\text{C}$ ,  $80^\circ\text{C}$ ). Different letters indicate significant differences ( $P < 0.05$ ). RMSE, F values and levels of significance derived from a general linear model are also presented.

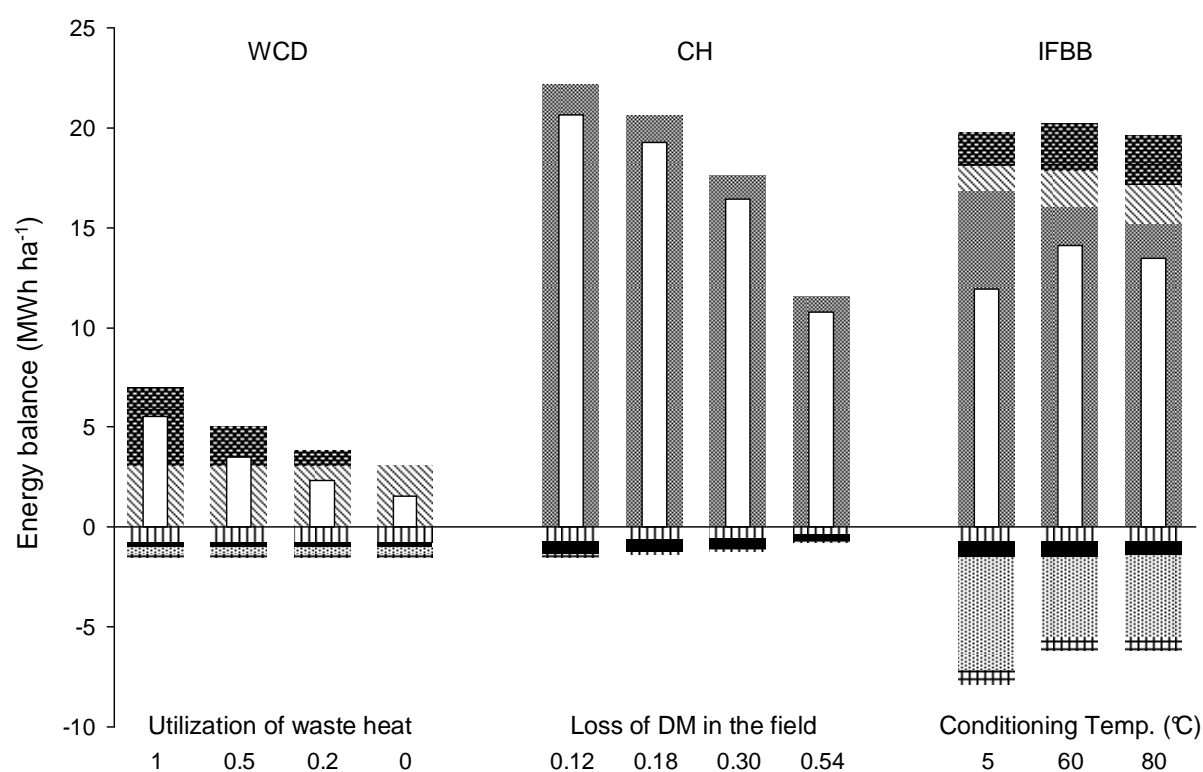
	WCD† (proportion of waste heat utilization)				CH† (proportion of DM loss in the field)				IFBB† (temperature of hydrothermal conditioning)			RMSE‡	F- value	Level of significance
	1	0.5	0.2	0	0.12	0.18	0.30	0.54	$5^\circ\text{C}$	$60^\circ\text{C}$	$80^\circ\text{C}$			
Net electric energy yield	2.7 a	2.76 a	2.76 a	2.76 a	-0.72 d	-0.67 c	-0.57 d	-0.38 cd	0.15 bcd	0.78 bc	0.93 b	0.60	32.58	***
Net thermal energy yield	2.75 bc	0.77 bc	-0.41 bc	-1.20 c	21.39 a	19.93 a	17.01 a	11.18 abc	11.78 abc	13.31 ab	12.55 abc	6.49	7.97	***
Total net energy yield	5.51 bcd	3.54 cd	2.35 d	1.56 d	20.67 a	19.26 ab	16.44 abc	10.80 abcd	11.93 abcd	14.10 abcd	13.48 abcd	6.51	5.39	***
Conversion efficiency	0.22 e	0.14 ef	0.10 f	0.07 f	0.74 a	0.69 a	0.59 b	0.39 d	0.43 cd	0.51 bc	0.48 c	0.04	187.80	***
GHG mitigation	-2.24 abc	-1.60 ab	-1.21 a	-0.95 a	-6.17 c	-5.75 bc	-4.91 abc	-3.23 abc	-3.55 abc	-4.40 abc	-4.25 abc	2.00	4.09	***

†Means of five swards

‡Root mean square error

\*\*\*,  $P < 0.001$

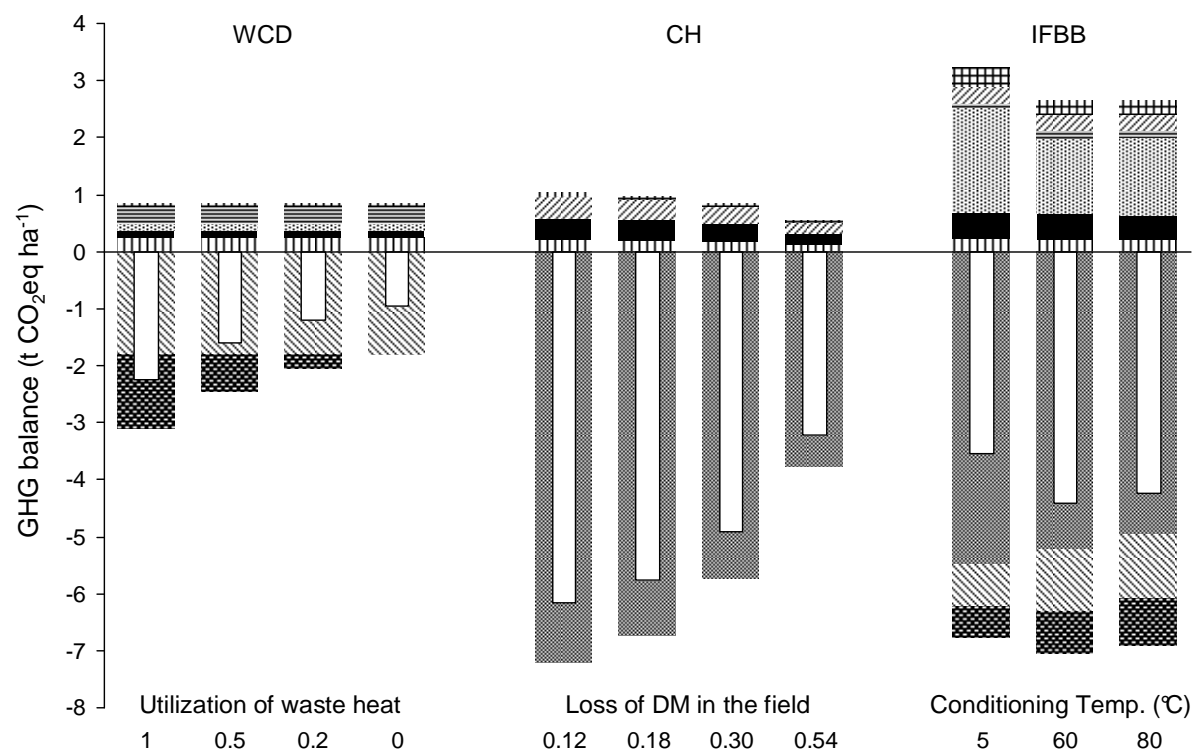
The energy input into the conversion system was relatively low for the WCD and CH scenarios when compared to the IFBB procedure, where the principal component for the WCD and CH was diesel for the cultivation, harvest and transport of the biomass (0.50 and 0.47 of the energy input for WCD and CH, respectively) (Figure 5.4). Proportionally, the largest energy cost in the IFBB procedure resulted from the heat energy used for hydrothermal conditioning (only 60 and 80°C treatments), for drying the press cake and for heating the fermenter (only 5°C treatment), which accounted for 0.72, 0.67 and 0.67 of the energy input in the 5°C, 60°C and 80°C treatment, respectively. The heat demand for drying the press cake was covered by the waste heat of the CHP to an average extent of 0.79, the remaining demand was covered by heat energy from the press cake. The indirect energy input for machinery and buildings was considerably higher in the IFBB procedure (0.56-0.72 MWh ha<sup>-1</sup>) compared to WCD (0.14 MWh ha<sup>-1</sup>) and CH (0.07-0.13 MWh ha<sup>-1</sup>).



**Figure 5.4** Net energy yield (□) of five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)) through three different conversion techniques (anaerobic digestion of whole-crop silage (WCD), combustion of hay (CH) and IFBB) as balance of direct energy input as diesel (cultivation, harvest, transport, ■), electricity (screw press, biogas plant operation, pelleting of solid fuel, ▨), heat (hydrothermal conditioning, heating of fermenter, drying of press cakes, ▩), indirect energy input (buildings, machinery, ▤) and energy output as electricity (▧) and heat (▦) from biogas and heat from solid fuel (▣).

Regarding WCD with a complete utilization of waste heat, the energy output was distributed according to the electrical efficiency of the CHP, with the ratio of 0.44 for electricity and 0.56 for heat. As the utilization of waste heat decreased (0.5, 0.2, 0), the proportion of heat in the total energy output gradually decreased (0.39, 0.21, 0). The energy output of CH consisted completely of thermal energy. The largest proportion of energy production in the IFBB procedure was made up of heat energy from pelleted press cake, which accounted for 0.85, 0.79 and 0.77 of the total energy output in the 5°C, 60°C and 80°C treatment, respectively.

The highest GHG mitigation (the most negative GHG balance) was achieved from CH at a DM field loss of 0.12 with  $-6.17 \text{ t CO}_2\text{eq ha}^{-1}$  and decreased with increasing DM field loss (up to 0.54) to  $-3.23 \text{ t CO}_2\text{eq ha}^{-1}$  (Table 5.3). A GHG mitigation of  $-4.40$ ,  $-4.25$  and  $-3.55 \text{ t CO}_2\text{eq ha}^{-1}$  for the 60°C, 80°C and 5°C treatment, respectively, was observed for IFBB conversion. The GHG balance from WCD was  $-2.24 \text{ t CO}_2\text{eq ha}^{-1}$  when the entire waste heat was used and  $-0.95 \text{ t CO}_2\text{eq ha}^{-1}$  when no heat was used. In the IFBB procedure, the input of heat (0.51-0.57) made up the largest proportion of the GHG emissions (Figure 5.5).



**Figure 5.5** Mean values of GHG mitigation ( $\square$ ) of five semi-natural grasslands (poor oat-grass meadows (*Arrhenaterion*), a small-sedge poor-fen (*Caricion fuscae*) meadow, a tall herb (*Filipendulion ulmariae*) meadow and a montane hay meadow (*Polygono-Trisetion*)) through three different conversion techniques (anaerobic digestion of whole-crop silage (WCD), combustion of hay (CH) and IFBB) as balance of GHG emissions from direct input of diesel ( $\square$ ), electricity ( $\blacksquare$ ), heat ( $\boxplus$ ), losses of methane ( $\boxminus$ ), nitrous oxide during combustion ( $\boxtimes$ ) and indirect energy input ( $\boxdot$ ) and GHG savings by the production of electricity ( $\boxtimes$ ) and heat ( $\boxplus$ ) from biogas and heat from solid fuel ( $\boxtimes$ ).



The proportional majority of the GHG emissions from the WCD system was due to methane losses (0.35) and the input of diesel (0.27). Electricity (0.20-0.39) and N<sub>2</sub>O during combustion (0.20-0.38) accounted for the largest proportion of GHG emissions in CH. For the IFBB procedure the main GHG savings were achieved by generating heat from solid fuel (0.72-0.81 of all GHG savings) and for WCD 0.58-1.0 of all GHG savings came from the generation of electricity. The GHG savings from CH were entirely due to the generation of heat from solid fuel as this was the only energy output from this process.

## 5.4 Discussion

The IFBB procedure is aimed at the conservation of semi-natural grasslands through an ecologically and economically viable utilization of the biomass for energy production. Towards this goal it will be essential to maximise the net energy yield, the energy conversion efficiency and the GHG mitigation. As the parent material lacks quality for a direct combustion as hay, it is furthermore of utmost importance to facilitate a maximum degree of release of detrimental compounds, such as K, Mg, Cl, N and S, which are responsible for corrosion inside the combustion chamber, ash melting and/or harmful emissions. Thus, quality requirements for the solid fuel (press cake) in this procedure are a reduction of these elements as well as an increase of dry matter content to at least 850 g kg<sup>-1</sup> FM in order to make it suitable for storage and pelleting (Wachendorf et al., 2009).

### 5.4.1 Chemical constituents and fuel quality of press cakes

The elemental concentrations in the parent material were in the range of hay from landscape management grassland as listed in the biofuel database by Hartmann et al. (2000), apart from the concentrations of Mg and Ca, which were higher. The mean concentrations were 12.0 g kg<sup>-1</sup> DM (5.4-23.4 g kg<sup>-1</sup> DM in Hartmann et al., 2000) for K, 3.2 g kg<sup>-1</sup> DM (1.2-2.2 g kg<sup>-1</sup>) for Mg, 10.0 g kg<sup>-1</sup> DM (3.7-7.2 g kg<sup>-1</sup>) for Ca, 4.8 g kg<sup>-1</sup> DM (1.1-10.5 g kg<sup>-1</sup>) for Cl, 13.7 g kg<sup>-1</sup> DM (8.7-16.1 g kg<sup>-1</sup>) for N and 1.6 g kg<sup>-1</sup> DM (1.1-3.6 g kg<sup>-1</sup> DM) for S. In five semi-natural grasslands harvested in August by Tonn et al. (2007) the concentrations of K, N and S (10.0-15.0, 12.8-18.9 and 1.3-1.7 kg<sup>-1</sup> DM, respectively) were comparable to this study, while the concentration of Cl was somewhat lower (2.5-3.7 kg<sup>-1</sup> DM). Kasper (1997) found in meadow foxtail and reed canary grass communities harvested in August and September lower concentrations for K (2.1-9.0 g kg<sup>-1</sup> DM), Mg (1.0-2.7 kg<sup>-1</sup> DM) and Ca (2.9-5.4 kg<sup>-1</sup> DM), comparable concentrations for N (10.0-18.1 kg<sup>-1</sup> DM) and S (0.9-1.7 kg<sup>-1</sup> DM) and higher concentrations for Cl (6.7-8.3 kg<sup>-1</sup> DM).

Compared to the parent material K and Mg concentrations in the press cakes were significantly reduced, while only a slight reduction of Ca occurred. These variations in chemical composition had a positive effect on the fuel quality. During combustion K reduces the ash melting point by forming potassium silicates that melt at low temperatures, causing slag formation in the combustion chamber (Jenkins et al., 1998). Furthermore the reaction of K with S and Cl forms chlorides and sulphates which play a major role in corrosion mechanisms inside the combustion chamber and may cause particulate emission (aerosols) as well as hard deposits on cooled furnace walls or heat exchangers. This may lead to serious technical problems and reduce the life span of the boiler. In contrast Ca increases the ash melting point by forming sulphates with less mobility and more favourable properties than potassium sulphates (Jenkins et al., 1998).

According to Obernberger et al. (2006) problems with ash melting occur at K concentrations in the ash above  $70 \text{ g kg}^{-1}$  ash and Ca concentrations in the ash below  $150 \text{ g kg}^{-1}$  ash. In this regard K in the parent material ( $159 \text{ g kg}^{-1}$  ash) and the  $5^\circ\text{C}$  press cake ( $81 \text{ g kg}^{-1}$  ash) exceeded the upper limit concentration, whereas press cakes after the  $60^\circ\text{C}$  and  $80^\circ\text{C}$  treatments ( $40$  and  $43 \text{ g kg}^{-1}$  ash, respectively) were below the upper limit. Regarding Ca, the concentrations in the ash of both the parent material and the press cakes were in a problematic range ( $106$ - $132 \text{ g kg}^{-1}$  ash). Woodchips of spruce, a common biofuel, have K concentrations of  $0.9$  to  $1.5 \text{ g kg}^{-1}$  DM, Mg concentrations of  $0.3$  to  $0.8 \text{ g kg}^{-1}$  DM and Ca concentrations of  $2.9$  to  $7 \text{ g kg}^{-1}$  DM (Van Loo and Koppejan, 2008). In relation to these elements, the press cakes derived from conditioning at high temperatures exhibited an almost 'wood-like' quality with K concentrations of  $2.43$  ( $60^\circ\text{C}$ ) and  $2.81 \text{ g kg}^{-1}$  DM ( $80^\circ\text{C}$ ), Mg concentrations of  $1.22$  ( $60^\circ\text{C}$ ) and  $1.26 \text{ g kg}^{-1}$  DM ( $80^\circ\text{C}$ ) as well as Ca concentrations of  $7.41$  ( $60^\circ\text{C}$ ) and  $7.76 \text{ g kg}^{-1}$  DM ( $80^\circ\text{C}$ ). Differences between the vegetation types were marginal for K, whereas the initial Mg concentrations were much lower in the two *Arrhenaterion* silages than in the other grassland silages. By far the highest initial Ca concentrations in both the parent material and the press cake were observed in the *Polygono-Trisetion* community which were well above the critical minimum concentration of  $150 \text{ g kg}^{-1}$  ash.

The significant reduction of the Cl concentration in the press cakes compared to the parent material has several positive effects on the fuel quality. During combustion Cl mainly forms gaseous HCl and alkali chlorides that are responsible for corrosive effects on the surface of the furnace and of the heat exchangers. Moreover, the emission of HCl and its influence on the formation of polychlorinated dibenzodioxins and dibenzofurans (PCDD/F) is critical,

especially for herbaceous biomass where levels increase drastically beyond a Cl concentration of ca.  $1.5 \text{ g kg}^{-1} \text{ DM}$  (Launhardt and Thoma, 2000). While the mean Cl concentration in the parent material ( $4.83 \text{ g kg}^{-1} \text{ DM}$ ) was about three times higher than the critical value mentioned above, mean Cl concentrations in the press cakes were well below this value ( $5^\circ\text{C}$ :  $0.81 \text{ g kg}^{-1} \text{ DM}$ ,  $60^\circ\text{C}$ :  $0.93 \text{ g kg}^{-1} \text{ DM}$ ,  $80^\circ\text{C}$ :  $1.19 \text{ g kg}^{-1} \text{ DM}$ ). Hustad et al. (1995) proposed a classification system for biofuels with three classes for each of the following parameters: thermal ash behaviour, N concentration and Cl concentration. For the elements the classes are: 1 (woodlike), 2 (strawlike), 3 (N- or Cl-rich); for the thermal ash behaviour: A (high softening), B (medium softening), C (low softening). This system characterises the press cakes in terms of Cl as class 1 (woodlike), while the parent material would be assigned to class 3 (Cl-rich).

No significant reduction of N could be obtained in the press cakes, even if a slight trend to lower N concentrations at conditioning temperatures of 60 and  $80^\circ\text{C}$  was observed. During combustion N is converted into gaseous  $\text{N}_2$  and nitric oxides ( $\text{NO}_x$ ), leading to emissions with great environmental impact. The N concentration limit for emission related problems is ca.  $6 \text{ g kg}^{-1} \text{ DM}$  (Oberberger et al., 2006). Mean concentrations of both the parent material ( $13.72 \text{ g kg}^{-1} \text{ DM}$ ) and the press cakes ( $5^\circ\text{C}$ :  $12.29 \text{ g kg}^{-1} \text{ DM}$ ,  $60^\circ\text{C}$ :  $11.46 \text{ g kg}^{-1} \text{ DM}$ ,  $80^\circ\text{C}$ :  $11.13 \text{ g kg}^{-1} \text{ DM}$ ) exceeded this limit and would be classified according to Hustad et al. (1995) as class 3 (N-rich).

$\text{NO}_x$  emissions measured in combustion tests with samples from IFBB press cakes and triticale whole crop in a reciprocating grate furnace ranged between 202 and 253 mg per normal cubic meter ( $\text{m}_\text{N}^3$ ) at N concentrations in the biomass of 9.7 -  $13.0 \text{ g kg}^{-1}$  (Heinz et al., 1999), which is comparable to the N concentrations in this study. These emission values are still below the emission limit value of  $\text{NO}_x$  in Germany ( $500 \text{ mg m}_\text{N}^{-3}$  for plants  $<1 \text{ MW}$ ; BMU, 2002) and can further be reduced by technological means, such as adapted air supply, geometry of the furnace, combustion temperature and type of combustion technology (Oberberger et al., 2006).

The significant reduction of S in the press cakes compared to the parent material improved the fuel quality. During combustion S forms gaseous  $\text{SO}_2$  and  $\text{SO}_3$  which are emitted to the environment or condense on fly-ash particles or the boiler surface. In addition S plays an important role in corrosion processes by sulphation of alkali chlorides which leads to the release of Cl that causes corrosion by the formation of  $\text{FeCl}_2$  or  $\text{ZnCl}_2$ . Problems during the combustion occur for S concentrations above  $1 \text{ g kg}^{-1} \text{ DM}$  (corrosion) and  $2 \text{ g kg}^{-1} \text{ DM}$

(emission), respectively (Oberberger et al., 2006). While the 60°C and 80°C press cakes (0.98 and 0.97 g kg<sup>-1</sup> DM, respectively) were well below both values, the parent material (1.59 g kg<sup>-1</sup> DM) and the 5°C press cake (1.08 g kg<sup>-1</sup> DM) were only below the critical emission value.

The AST mainly depends on the K, Mg and Ca concentrations. Reference values according to Van Loo and Koppejan (2008) are 1260°C for beech wood, 1410-1640°C for spruce wood and 950-1230°C for grass. The mean AST of the parent material (1111°C) was typical for grass and would be assigned to class B (medium softening) in the system of Hustad et al. (1995). While the 5°C press cakes obtained only slightly higher AST (1169°C), the 60°C and 80°C treatments produced press cakes with estimated AST values similar to those of beech wood with 1250 and 1254°C, respectively, which would be classified as class A (high softening).

At present, little information is available about the removal of mineral constituents of biomass through mechanical dehydration. Turn et al. (1997) subjected banagrass (*Pennisetum purpureum*) to different treatments of fine comminution, rinsing and multistep-dewatering in a plunger press. The optimum treatment showed high proportional reductions of K (0.90), Mg (0.70), Cl (0.98) and S (0.54) and an AST of 1250°C was predicted, which is similar to this study. N was reduced by 0.48 which is considerably higher than in this study. This might be due to a younger physiological age of the biomass, where higher proportions of non-protein N occur which may be more easily mobilised by physical pre-treatments, as it is less interwoven with persistent structural carbohydrates in cell walls of plants.

In press fluids from fresh biomass and silage of physiologically young bastard ryegrass (*Lolium hybridum*) and alfalfa (*Medicago sativa*) produced with a similar screw press as in the present study, the recovery rate of N (tantamount to the reduction of N in the press cake) was between 0.2 and 0.5 (Kromus et al., 2004). This rate was further increased by pressing the press cake for a second time, using clover grass silage (total N recovery rate of 0.5-0.6) and alfalfa silage (total N recovery rate of 0.6-0.7) as parent material (Mandl et al., 2006). The delay of the grassland harvest until winter or spring of the following year was shown to be a quite effective means to reduce detrimental elements in biomass (Table 5.4). Particularly wilting processes over winter brought about a considerable reduction of minerals through leaching or reallocation to the rhizome system (Hadders and Olsson, 1997; Christian et al., 2002). Experiments on semi-natural grasslands showed relative reductions in elemental concentrations that were comparable to those obtained by hydrothermal conditioning and

mechanical dehydration (Tonn et al., 2007). There is further evidence for the effects of delayed harvest on elemental concentrations in lowland grassland monocultures, where reductions in the same magnitude as in the IFBB procedure or even higher (in the case of N) were obtained (Landström et al., 1996; Burvall, 1997; Christian et al., 2002; Xiong et al., 2008).

**Table 5.4** Mean element concentrations in various delayed harvest experiments with grassland vegetation and relative reduction of element concentrations between conventional and winter/spring harvest.

Vegetation, region	Reference	Harvest Period	Mean concentration (g kg <sup>-1</sup> DM)						Relative reduction of concentration						
			K	Mg	Ca	Cl	N	S	K	Mg	Ca	Cl	N	S	
Three semi-natural grassland communities, Germany	Tonn et al. (2007)	September	10.6	–	12.5	1.9	13.7	1.6							
		February	3.4	–	8.8	0.7	11.4	1.4	0.68	–	0.30	0.63	0.17	0.13	
<i>Phalaris arundinacea</i> , China	Xiong et al. (2008)	September	13.5	–	5.5	14.0	12.0	–							
		April	2.1	–	3.2	2.5	7.4	–	0.84	–	0.42	0.82	0.38	–	
<i>Phalaris arundinacea</i> and <i>Panicum virgatum</i> , England	Christian et al. (2002)	September	6.1	–	–	3.0	12.0	–							
		March	2.0	–	–	1.0	8.0	–	0.67	–	–	0.67	0.33	–	
<i>Phalaris arundinacea</i> , Sweden	Burvall (1997)	July to October	12.3	1.3	3.5	5.6	13.3	1.7							
		March to May	2.7	0.5	2.0	0.9	8.8	0.9	0.78	0.62	0.43	0.84	0.34	0.47	
<i>Phalaris arundinacea</i> , Sweden	Landström et al. (1996)	August	11.2	1.2	–	3.6	11.8	–							
		April to May	2.5	0.5	–	0.8	9.3	–	0.78	0.58	–	0.78	0.21	–	

In comparison to IFBB with a harvest in autumn at latest delayed harvest requires considerably less technical effort to achieve comparable reductions of elements detrimental for combustion. However, some further aspects may complicate the practical application of delayed harvest in semi-natural grasslands. Lodging of the vegetation over winter especially in mountain regions with high snow-fall may lead to DM yield losses of over 0.7 (Tonn et al., 2007). These losses may be due to respiration and leaching or they arise from mechanical treatments (McGechan, 1989) leading to stem and leaf detachment (McGechan, 1989; Christian et al., 2006). Lindh et al. (2009) found total harvest losses for *Phalaris arundinacea* in Finland of 0.2-0.4 under optimum conditions and 0.5-0.6 under worst conditions. Proportional DM yield losses of *Phalaris arundinacea* between a conventional harvest in

September and a delayed harvest in March were reported to be on average 0.3 in southern England (Christian et al., 2002) and 0.18 for the same crop grown in Sweden (Landström et al., 2006).

Compared to pure swards of perennial grasses, semi-natural grasslands usually contain higher proportions of legumes and forbs which are more susceptible to DM losses as grass species (Dernedde and Honig, 1979; Rees, 1982). Harvesting generally requires soil conditions which allow the traffic of harvesting machinery. As semi-natural grasslands often occur at marginal sites with wet soil conditions during winter this may impose restrictions on a delayed harvest in winter or early spring, unless ground frost stabilises the soil. Limited trafficability in winter could be a problem on all of the sites of this study due to high annual rainfall (Wachendorf et al., 2009). In addition the uncertainty of suitable weather conditions for hay making during winter or early spring have to be considered.

There is evidence that disturbance in the form of mowing has a great impact on species diversity as it prevents competitive exclusion by permanently reducing the competitive ability of the dominants (Huston, 1979, 1994). Jacquemyn et al. (2003) state that in vulnerable grassland communities, like calcareous grassland, removal of above-ground phytomass appears to be a prerequisite for the maintenance of a high diversity of plant species. They found that species diversity declined by about 30% after only 2 years and decreased further during the following years without any form of management. There is no specific information available on the effects of delayed harvest on species diversity of semi-natural grassland, but it is to be assumed that, albeit less pronounced than in abandoned grasslands, prolonged periods of undisturbed growth lead to a decrease in light availability resulting from high population growth rates of tall forbs and grass species (Jacquemyn et al., 2003). These may outcompete less competitive species whereas the decreased light penetration to subcanopy plants such as seedlings and low growing species may further lead to a decline in species richness because the decreasing light availability may result in greater mortality of subcanopy plants (Newman, 1973; Goldberg and Miller, 1990). Thus, delayed harvest may be an option for perennial grasses, when soil conditions are suitable and species diversity is not an objective, but it is questionable if such a management complies with the natural conditions and conservation goals of semi-natural grasslands.

The HHV of the parent material (17.82 MJ kg<sup>-1</sup> DM) was of similar magnitude as those reported for wheat straw (17.94 MJ kg<sup>-1</sup> DM, Jenkins et al., 1998) and *Festuca arundinacea* (17.8 MJ kg<sup>-1</sup> DM, Hartmann, 2001). The slightly higher HHV of the press cakes of the

5°C treatments (18.05 MJ kg<sup>-1</sup> DM) were in the range of those reported for *Lolium perenne* (18.0 MJ kg<sup>-1</sup> DM, Hartmann, 2001) and *Panicum virgatum* (18.06 MJ kg<sup>-1</sup> DM, Jenkins et al., 1998). Even higher were the HHV of the press cakes from the 60 and 80°C treatments (18.41 and 18.33 MJ kg<sup>-1</sup> DM, respectively), which was comparable to alfalfa stems (18.67 MJ kg<sup>-1</sup> DM, Jenkins et al., 1998) and wheat grains (18.4 MJ kg<sup>-1</sup> DM, Hartmann, 2001). A comparable increase in HHV from 18.2 to 18.7 MJ kg<sup>-1</sup> DM was reported from the multistep-dewatering of banagrass (Turn et al., 1997). However, the HHV of the IFBB press cakes were still somewhat lower than those of short rotation coppice, like wood from willow or hybrid poplar with 19.59 MJ kg<sup>-1</sup> and 19.02 MJ kg<sup>-1</sup> DM, respectively (Jenkins et al., 1998).

#### 5.4.2 Energy and greenhouse gas balance

The conversion of semi-natural grassland biomass into energy using the IFBB procedure was more efficient than WCD, even when complete waste heat utilization was assumed in the latter system, which is barely achievable in practice. This is mainly due to the low digestibility of mature grassland biomass in WCD, which results in low methane yields and low energy outputs (Lemmer and Oechsner, 2001; Richter et al., 2009b). Much higher energy yields (21-33 MWh ha<sup>-1</sup> compared to 6-10 MWh ha<sup>-1</sup> in this study) can be obtained by high-input grassland, which is cut 2-3 times per year and has a higher digestibility (Rösch et al., 2009). In contrast, the press fluid from the IFBB process is easily digestible with high specific methane yields (Richter et al., 2009b) and the press cakes show good HHV, which results in higher total energy outputs.

Bühle et al. (2009) calculated net energy productions of up to 63 MWh ha<sup>-1</sup> for maize and rye silage converted with IFBB, which is about four-fold higher than the net energy production in this study. Considering the fact that the dry matter yields of the biomass were also about four-fold higher than in this study, it can be assumed that the net energy production is comparable for the IFBB conversion of biomass from arable crops and from semi-natural grassland. The corresponding conversion efficiencies for maize and rye silage were up to 0.60, which is slightly higher than the data in this study, where values of up to 0.51 were observed. In contrast, WCD conversion of maize and rye silage achieved conversion efficiencies of 0.44 at maximum (Bühle, 2008), while in this study only 0.22 of the gross energy in the feedstock could be converted into net energy. This suggests that IFBB may be more efficient than WCD in the case of low digestible biomasses, such as semi-natural grasslands with a delayed cut, whereas for easily digestible biomasses, such as maize or rye silages harvested at an

appropriate date, the differences may vanish. However, higher net energy yields might be obtainable for WCD, if the digestate was used as solid fuel, which is not of practical relevance at present due to very high elemental concentrations in the digestate and a high energy demand for drying. Therefore it was not evaluated in this study.

With regard to humus balances it is noteworthy that for WCD much more carbon is recycled to the field than for IFBB, where most of the carbon is converted into biogas and solid fuels. Prochnow et al. (2009a) calculated for biogas production from extensively managed grassland that in total a proportion of 0.5 of the carbon in the above-ground biomass is returned to the system in the form of harvest residues (0.1 of the carbon in the above-ground biomass) and the digestate (0.4). In the IFBB system 0.2-0.3 of the organic matter of the harvested biomass is transferred into the press fluid (Wachendorf et al., 2009) and on average 0.1 of the organic matter of the press fluid is not transformed into biogas, but remains in the digestate (Richter et al., 2009b). Thus, as a rough estimate, proportionally 0.02-0.03 of the carbon of the above-ground biomass is returned to the system in form of the digestate, but together with harvest residues a total of 0.12-0.13 of the carbon of the above-ground biomass is cycling within the system. However, pasture plants transfer a proportion of 0.3 to 0.5 of assimilates below ground (Kuzyakov and Domanski, 2000), accomplishing a substantial replenishment of soil organic matter (Oades, 1988). Unlike arable land where intensive carbon mineralization occurs due to cultivation, mineralization processes on permanent grassland occur at very low rates, resulting in a net accumulation of carbon in the soil.

In comparison to the IFBB procedure, CH was characterized by a lower energy input, as solar radiation provided the energy for the field curing of hay, resulting in higher conversion efficiencies under conventional harvest conditions. Comparable net energy yields for the combustion of hay were also found by Rösch et al. (2009). The quality of the hay as solid fuel is, however, low due to the presence of detrimental compounds, therefore the energy output is only achievable under the assumption that the hay can be used in co-combustion with high-quality fuels. Through delayed harvest in winter or spring of the next year hay of a better fuel quality can be produced, which is comparable to the press cakes of the IFBB procedure.

The delayed harvest scenario (CH with a DM field loss of 0.54), however, obtained a lower net energy yield and conversion efficiency than all IFBB scenarios. In order to achieve the same net energy yield as the IFBB scenario with a hydrothermal conditioning at 60°C (14.10 MWh ha<sup>-1</sup>), DM field losses in CH may not exceed 0.40. The direct comparison of yields of different forms of energy (heat versus electricity) is problematic. WCD generates about four



times as much electricity as the IFBB procedure, which generates for the most part thermal energy in the form of press cake pellets, while CH is primarily used to generate thermal energy. Thus, for a final evaluation of such different bioenergy systems, the specific situations of energy demand, desired form of the energy, energy supply, environmental standards and economic conditions in a certain region or country have to be considered (McKendry, 2002).

With regard to GHG mitigation, the IFBB procedure out-performed WCD. The difference between the two procedures however, was smaller than in terms of energy. This is due to the larger proportion of electric energy produced in WCD and the pronounced GHG saving for the substitution of electric energy from fossil fuels which is almost double the GHG saving for the substitution of thermal energy. CH showed higher GHG mitigation than the IFBB procedure under conventional harvest conditions (DM field loss of 0.12-0.30), but lower GHG mitigation under delayed harvest conditions (DM field loss of 0.54).

Because of the lower proportion of electric energy output, the DM field loss in CH has to be lower than 0.37 in order to achieve the same GHG mitigation as the IFBB scenario with a hydrothermal conditioning at 60°C (-4.4 t CO<sub>2</sub>eq ha<sup>-1</sup>). The latter value was only about one third of this calculated by Bühle et al. (2009) for maize and rye silage converted through the IFBB procedure, but comparable to the values of a practice-related model plant (5-7 t CO<sub>2</sub>eq ha<sup>-1</sup>) based on maize WCD without any utilization of the waste heat (Berenz, 2008). The calculated GHG reduction potential of hay pellets from a low-input permanent grassland was -5.13 t CO<sub>2</sub>eq ha<sup>-1</sup> at a DM yield of 3.9 t ha<sup>-1</sup> (Rösch et al., 2009), which is comparable to the values of the CH scenarios (-6.17 to -4.91 t CO<sub>2</sub>eq ha<sup>-1</sup>) at a slightly higher mean DM yield of 5.6 t ha<sup>-1</sup>.

## 5.5 Conclusions

### 5.5.1 Specific Conclusions

(i) Compared to the grassland silages used as parent material, the press cakes generated using hydrothermal conditioning and subsequent mechanical dehydration showed significantly ( $P < 0.05$ ) lower concentrations of elements detrimental for combustion. Overall, the conditioning temperature of 60°C obtained the lowest concentrations with 2.43 (K), 1.22 (Mg), 0.93 (Cl) and 0.98 g kg<sup>-1</sup> DM (S). Decreases of Ca and N in the 60°C treatment to concentrations of 7.41 and 11.46 g kg<sup>-1</sup> DM, respectively, were not significant. As a result of the changed chemical composition, the ash softening temperature of the press cakes increased

significantly ( $P < 0.05$ ) for the 60 and 80°C treatments and reached 1250°C (60°C treatment), which is comparable to beech wood. The increase in the higher heating value of these press cakes (18.41 MJ kg<sup>-1</sup> DM, 60°C treatment) was not statistically significant.

(ii) Conversion efficiencies of the IFBB procedure ranged from 0.43 (5°C treatment) to 0.51 (60°C treatment) of the gross energy contained in the grassland biomass. Conventional anaerobic digestion of whole crop silages from the same biomass obtained conversion efficiencies of 0.07 to 0.22, dependent on the proportion (0 to 1) of waste heat utilised. Highest conversion efficiencies of 0.59 to 0.74 were obtained with the combustion of hay, when 0.30 to 0.12 of the DM yield was lost during field curing of the hay, although the fuel quality was quite low. The delayed harvest of hay achieved a conversion efficiency of 0.39 at a DM field loss over winter of 0.54 with a fuel quality comparable to IFBB press cakes.

(iii) Greenhouse gas mitigation through the substitution of fossil energy by electric and heat energy generated from biomass was up to -4.40 t CO<sub>2</sub>eq ha<sup>-1</sup> for the IFBB procedure (60°C treatment), -2.24 to -0.95 t CO<sub>2</sub>eq ha<sup>-1</sup> for the WCD digestion (proportion of waste heat utilization of 1 to 0, respectively) and -6.17 to -3.23 t CO<sub>2</sub>eq ha<sup>-1</sup> for the combustion of hay (proportion of DM as field loss of 0.12 to 0.54, respectively).

### 5.5.2 General Conclusions

In landscapes with high proportions of semi-natural grasslands the IFBB procedure may open up new perspectives for a regular management of such vegetations which are of high ecological importance due to species diversity. Compared to conventional biogas production,

- IFBB has lower demands in terms of biomass quality and can convert biomasses of a low digestibility into utilizable energy much more efficiently,

- IFBB comprises a complete and year-round utilization of the waste heat, which is favourable in rural areas, where municipal and industrial heat demand is low and where access to the gas distribution system may be limited.

Similar to the combustion of hay the major proportion of energy is retained in a storable solid fuel. While IFBB fuels are of superior quality compared to conventional hay, a delayed harvest of hay may result in a comparable fuel quality. Such fuels can be used flexibly and may support a local value added, when traded as a renewable substitute for fossil heat carriers.

The balance calculations presented in this paper partially used data from small-scale experimental devices or from literature. Although measures were taken to correct for scaling-up effects, some uncertainty remains until a pilot plant is built. It will then be subject to a comprehensive system analysis (considering actual costs for construction, transportation and for alternative solid fuels, etc.) to determine if the claimed advantages of the IFBB technique can compensate for the increased technical demands and costs of the concept.

## **6 Influence of sward maturity and pre-conditioning temperature on the energy production from grass silage through the integrated generation of solid fuel and biogas from biomass (IFBB): 1. The fate of mineral compounds**

**Abstract** The novel IFBB process is aimed at converting biomass from low-input high-diversity grasslands into energy, which is problematic for conventional conversion techniques. In the IFBB process, biomass is separated into a press fluid for biogas production and a press cake for combustion. Herbage from a lowland hay meadow (*Arrhenaterion*) was sampled and ensiled on eight dates between 24 April and 21 June 2007. The silage from each date was processed in six treatments without and with hydrothermal conditioning at different temperatures. The effects on mass flows of plant compounds and on elemental concentrations in the press cake were investigated. Elements detrimental for combustion were significantly reduced in the press cake compared to the silage. Mass flows and elemental concentrations in the press cake were strongly influenced by temperature of hydrothermal conditioning as well as concentration of neutral detergent fibre and dry matter in the silage ( $R^2$  from 0.70 to 0.99).

### **6.1 Introduction**

Energy from biomass is a contemporary issue in science and politics. The growing demand for energy worldwide, as well as the limited and unstable supply of fossil fuels, is responsible for increasing energy prices. Furthermore, constantly increasing CO<sub>2</sub> emissions are intensifying climate change. Thus, both from an economic and an ecological point of view, there is an urgent need to develop sustainable, eco-friendly energy supplies.

Grassland plays an important role as part of the world's biomass potential, since 26% of the total land area and 68% of the agricultural area is covered by permanent pastures and meadows (FAOSTAT, 2009). In many European countries, the traditional management of large areas of permanent grassland as a feed source in animal husbandry is at risk of being abandoned, because the herbage does not meet the yield and quality demands of intensive livestock production (Isselstein et al., 2005). However, regular management is necessary in order to maintain these grasslands and the benefits that emanate from them, such as high biodiversity, protection of soils, carbon storage, possibilities for human recreation and rural development. The utilization of grassland biomass for energy purposes might be an

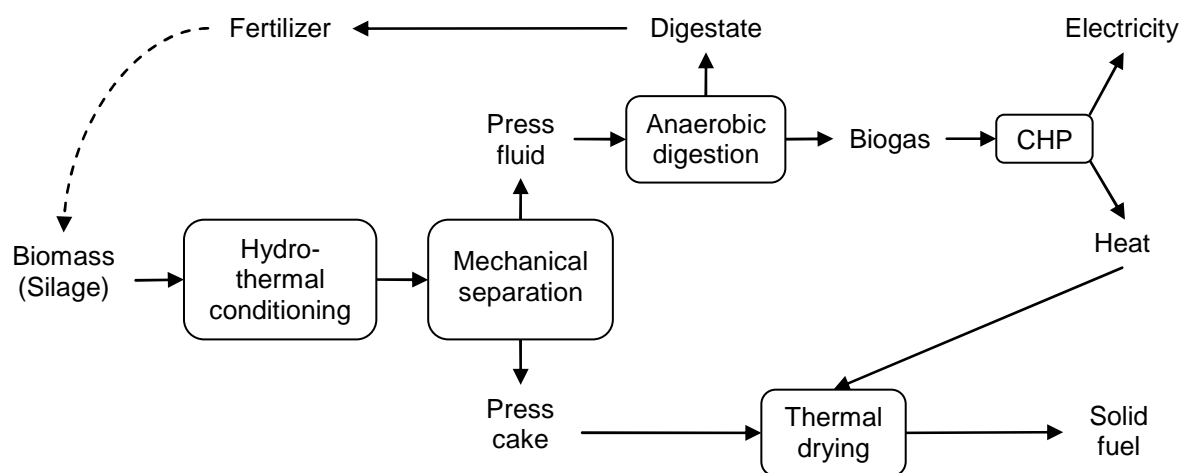
alternative management option for permanent grassland and has been extensively investigated for the two most common conversion processes, i.e. biogas production from grass silage (Prochnow et al., 2009a) and combustion of hay (Prochnow et al., 2009b).

However, both techniques face important limitations with regard to low-input high-diversity (LIHD) grassland. In the case of biogas production, these are associated with ligno-cellulose compounds, which are resistant to anaerobic digestion and inhibit the fermentation of readily digestible compounds through their inclusion. In consequence, the methane yields and the conversion efficiencies are low (Shiralipour and Smith, 1984). Furthermore, in order to achieve satisfactory conversion efficiencies, the waste heat from combined heat and power plants (CHP), which accounts for more than 0.60 of the total energy contained in the biogas, must be used. This is difficult to achieve in rural areas, where the demand for heat is low compared to industrialised urban areas.

As for the combustion of hay, major limitations result from the production and from the chemical composition of hay. The process of haymaking is strongly dependent on good weather conditions for achieving dry matter (DM) contents of about  $850 \text{ g kg}^{-1}$ , which are necessary to prevent microbial deterioration during storage. Besides, field losses during haymaking are usually higher than for silage production, which results in reduced yields of energy. Compared to wood as the most common solid biofuel, hay contains more N, K, Mg and Cl. During combustion, N is almost completely transformed into nitrogen oxides ( $\text{NO}_x$ ), which are a major air pollutant (Greul, 1998). K and Cl are significantly involved in corrosion processes in the furnace and K, together with Mg, promotes melting of the ash at high temperatures, while Ca works against it (Van Loo and Koppejan, 2001). Melting of the ash leads to slagging and fouling inside the combustion chamber, which reduces the life span of the boiler (Oberberger et al., 2006). Complex technologies are required to prevent adherence of the ash to iron walls within the furnace and to reduce the emission of particles and chemical compounds harmful to the environment.

With the aim of overcoming the problems outlined above, the Integrated Generation of Solid Fuel and Biogas from Biomass (IFBB) was developed (Wachendorf et al., 2009), which separates the easily digestible constituents of grass silage into a liquid form, to be converted into biogas, from the more fibrous parts, which are processed to form a solid fuel (Figure 6.1). The first step is a hydrothermal conditioning of the silage in which silage and water are mixed and heated under continuous stirring for a short time. This treatment is designed to macerate cell walls and produces a mash which is then mechanically dehydrated by a screw press. As

an effect of the conditioning and dehydration, several minerals (e.g. K, Mg, P and Cl) and organic compounds (e.g. carbohydrates, proteins and lipids) are transferred into the press fluid (PF), which is an excellent feedstock for biogas production, with specific methane yields of up to 500 normal litres ( $L_N$ ) per kg volatile solids (VS) (Richter et al., 2009b). The remaining press cake (PC) is rich in cellulose and hemicellulose, and contains relatively low concentrations of detrimental elements, which results in an improved quality as a biofuel (Richter et al., 2010a).



**Figure 6.1** Flow chart of the integrated generation of solid fuel and biogas from biomass (IFBB). CHP refers to a combined heat and power plant.

The PC achieves DM concentrations of about 450-500  $g\ kg^{-1}$  through mechanical dehydration, but needs to be dried to a DM content of about 850  $g\ kg^{-1}$  in order to be suited for pelleting and storage (Hartmann, 2001). Thus, the IFBB process has a year-round demand for heat produced in the CHP from PF biogas. Richter et al. (2010a) calculated that proportionally around 0.5 of the gross energy contained in the biomass from semi-natural grassland was converted into electricity (0.06) and heat (0.94) through the IFBB process, whereas only 0.25 was recovered through conventional whole-crop fermentation, even when all waste heat from the CHP was utilized. The IFBB conversion of semi-natural grasslands to energy results in three major products: (i) a solid fuel of acceptable quality, which is suitable for combustion, gasification or subsequent processing to, for example, synthetic fuels; (ii) electricity produced in the CHP from the combustion of biogas, which can be fed into the national electricity grid and (iii) a digestate from the biogas plant with high concentrations of readily available nutrients, which can be used as liquid fertilizer.

Previous studies on the conversion of extensively managed grassland vegetations by the IFBB technique only comprised single sampling dates and a low range of temperatures during hydrothermal conditioning (Wachendorf et al, 2009; Richter et al., 2009a; Richter et al., 2009b; Richter et al., 2010a). Furthermore, the data available do not provide information on the effect of sward maturity, as it cannot be separated from the effect of grassland vegetation. The present study seeks to fill these gaps of knowledge by sampling biomass from a botanically well defined sward on consecutive dates over a prolonged spring growth and by including IFBB treatments without hydrothermal conditioning and hydrothermal conditioning at temperatures of 10, 30, 50, 70 and 90°C. Hence, the objective of this study was to determine the influence of sward maturity and hydrothermal conditioning on (i) mass flows of plant compounds into the PF and on (ii) concentrations of combustion-relevant elements in the PC. A further goal was (iii) to identify chemical parameters in the parent grassland silage which might possibly explain the mass flow and the concentration in the PC for several plant compounds at the same time.

## 6.2 Material and Methods

### 6.2.1 Grass Silage

The grass silage consisted of herbage from a permanent sub-montane hay meadow (*Arrhenaterion*) on a loamy soil in Northern Hesse, Germany (51°23' N, 9°54' E, 284 m above sea level). During the experimental year (2007), the annual precipitation was 619 mm and the mean annual temperature was 7.8°C. Botanical composition of the sward and estimated species' contributions to the DM yield are shown in Table 6.1. The grassland has been managed with two annual cuts for hay making (late first cut at the beginning of June, second cut in August), followed by grazing with sheep and no fertilizer or further treatment for more than 10 years.

Biomass was sampled and ensiled on eight consecutive dates of a prolonged spring cut (27/04, 02/05, 09/05, 16/05, 24/05, 31/05, 11/06, 21/06). Due to weather conditions, the interval between samplings varied from seven to twelve days. The grassland was divided into three blocks and each block was randomly divided into eight plots of 60 m<sup>2</sup>, one for each sampling date. Three plots, one in each block, were harvested at each date with a finger-bar mower at a cutting height of 5 cm after samples for the determination of fresh matter (FM) and dry matter (DM) had been taken from three randomly located sub-plots of 0.25 m<sup>2</sup> in each plot. About 50 kg FM from each plot were chopped into 1-3 cm long pieces with a modified

maize chopper, compacted and ensiled for three months in two 50 L polyethylene barrels, with no additives being applied. After opening the barrels, fermentation quality was checked for colour, texture and smell (Wilkinson, 2005). All attributes indicated that silages had been well preserved.

**Table 6.1** Species DM contribution in the experimental sward.

Species	DM Contribution (%)†
Grasses:	
<i>Alopecurus pratensis</i>	28
<i>Arrhenaterum elatius</i>	5
<i>Dactylis glomerata</i>	0.4
<i>Festuca rubra</i>	6
<i>Holcus lanatus</i>	30
<i>Lolium multiflorum</i>	4
Sum:	73.4
Legumes:	
<i>Trifolium pratense</i>	8
<i>Trifolium repens</i>	1
<i>Vicia sepium</i>	0.2
Sum:	9.2
Herbs:	
<i>Glechoma hederacea</i>	0.1
<i>Heracleum sphondylium</i>	3
<i>Hieracium spec.</i>	0.1
<i>Ranunculus repens</i>	0.1
<i>Rumex acetosa</i>	4
<i>Taraxacum officinale</i>	10
<i>Veronica chamaedrys</i>	0.1
Sum:	17.4

†Visual estimation on 02 May 2007

### 6.2.2 Hydrothermal conditioning and mechanical dehydration

The six barrels of silage from each sampling date were pooled, thoroughly mixed and subjected to six different treatments of hydrothermal conditioning. One treatment was the direct mechanical dehydration without hydrothermal conditioning, the other five treatments comprised hydrothermal conditioning at different temperatures (10°C, 30°C, 50°C, 70°C, 90°C) and subsequent mechanical dehydration. For the hydrothermal conditioning, 20 kg FM of the silage were mixed with 80 kg tempered water in a modified concrete mixer with a



volume of 700 L, resulting in a mash with a silage:water ratio of 1:4. The mash was kept at a constant temperature with gas burners and stirred for 10 min in order to thoroughly rinse the silage with water. Subsequent mechanical dehydration of the silage was conducted with a screw press (type AV, Anhydro Ltd., Kassel, Germany). The conical screw had a pitch of 1:6 and a rotational speed of 6 revolutions  $\text{min}^{-1}$ . The cylindrical screen encapsulating the screw had a perforation of 1.5 mm. Sub-samples of the silage and the PF were stored at 4°C for anaerobic digestion experiments, sub-samples of the silage and the PC were immediately dried at 60°C for 48 h for chemical analysis. DM content of all sub-samples of the silage, the PF and the PC was determined by oven-drying at 105°C for 48 h. Ash concentration of all sub-samples of silage, PF and PC was determined by combustion at 550°C for 48 h.

### 6.2.3 Chemical analyses and mass flow calculation

The silage and the PC were analyzed for K, Mg, Ca, Cl, S and P by X-ray fluorescence analysis, for N using an elemental analyzer (EA 1106, Carlo Erba Ltd., Rodano, Italy) and for the organic constituents crude protein (CP), ether extract (EE), neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) according to standard methods (Bassler, 1976). Based on the concentrations of K, Ca and Mg ( $\text{g kg}^{-1}$  dry matter, DM), the ash softening temperature (AST; in °C), an important indicator to describe the ash melting behaviour during combustion, was calculated according to Hartmann (2001):

$$AST = 1172 - 5.39 * K + 25.27 * Ca - 78.84 * Mg$$

The concentration of any chemical compound (represented by Z;  $\text{g kg}^{-1}$  DM) in the PF was calculated from the concentration of Z in the silage (SIL) and the PC and the DM contents of the PC, the PF and the silage after hydrothermal conditioning (SILC) according to:

$$Z_{PF} = \frac{DM_{SILC} * Z_{SIL} - Y * DM_{PC} * Z_{PC}}{X * DM_{PF}}$$

where X and Y are the quantities of the PF and the PC as a proportion of the silage after hydrothermal conditioning, respectively, which were calculated by:

$$X = \frac{DM_{PC} - DM_{SILC}}{DM_{PC} - DM_{PF}} \quad Y = 1 - X$$

The mass flow (MF) of a substance into the PF is defined as the proportion of this substance's concentration in the silage, which is transferred into the PF after mechanical dehydration and

is given in a dimensionless figure between 0 and 1. The MF of DM and of any other compound (Z) from the silage into the PF was determined by:

$$MF_{DM_{PF}} = \frac{X * DM_{PF}}{DM_{SILC}} \qquad MF_{Z_{PF}} = \frac{X * DM_{PF} * Z_{PF}}{DM_{SILC} * Z_{SILC}}$$

A full derivation of these equations is given elsewhere (Wachendorf et al., 2009). In this paper, the term ‘mass flow’ means mass flow into the PF.

#### 6.2.4 Statistical analysis

Analysis of variance was performed to test the effect of hydrothermal conditioning before mechanical dehydration on the mass flows of plant compounds into the PF and on elemental concentrations in the PC. This was done by the comparison of two treatments, mechanical dehydration without conditioning and mechanical dehydration after conditioning at 10°C, which was also the ambient temperature at which the mechanical dehydration was conducted. As there was only one observation for each combination of treatment and sampling date, the sampling dates were allocated to four periods with two consecutive samplings each, allowing an analysis of variance (ANOVA), with the two samplings used as replications. The procedure MIXED in SAS (SAS Institute, 1996) was used with treatment (T), sampling period (SP) and T x SP as fixed effects and SP as repeated subject. Due to the lack of replications, the ANOVA results are only tentative and should be interpreted cautiously.

Multiple regression analysis was performed to test the effect of chemical constituents in the parent silage on mass flows during mechanical dehydration and on the elemental concentrations in the PC, using the procedure GLM in SAS (SAS Institute, 1996). As the sampling date is a year-specific variable without much biological information and due to some insight from previous experiments, the concentration of neutral and acid detergent fibre fractions (NDF, ADF) and acid detergent lignin (ADL) were included in the starting model for describing the maturity of grassland swards, as well as the DM concentration of the silage and the conditioning temperature.

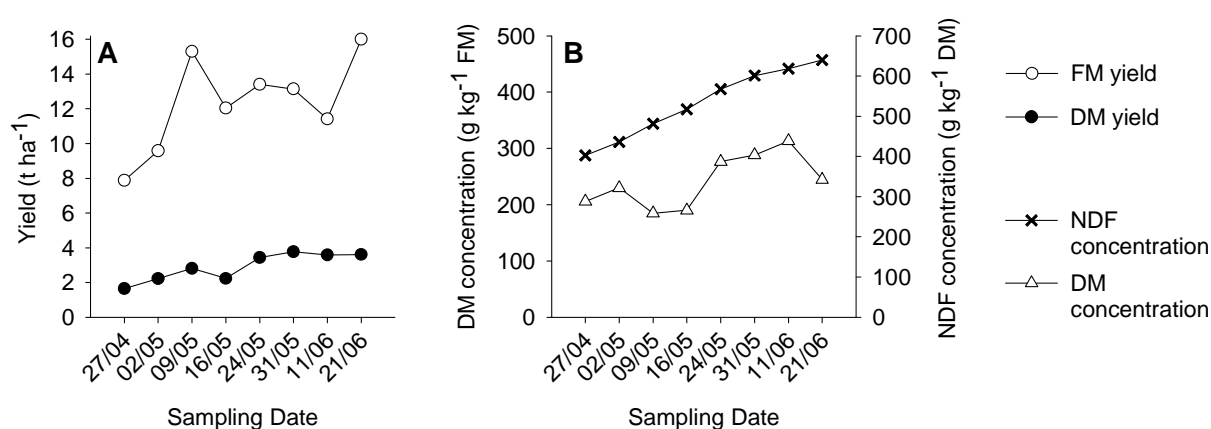
The selection of terms for inclusion in the model either as main effects, quadratic term or twofold interaction depended on standard statistical model selection methods (Draper and Smith, 1998) and obeyed the rules of hierarchy and marginality (Nelder and Lane, 1995). Effect terms were included if their significance exceeded the 5% level. The rules of hierarchy ensured that if an interaction was included in the model, then the variables involved in the interaction were also included separately. The marginality principle (Nelder and Lane, 1995)

implies that if a term appears as part of a more complex element in the model then, in general, the term itself is not tested for significance, because the meaning of such terms is open to misinterpretation (Connolly and Wachendorf, 2001). Thus, *P* values for model coefficients where the term is involved in a higher-order term in the model were generally omitted. In order to avoid multicollinearity, Pearson's correlation was performed with the CORR procedure in SAS (SAS Institute, 1996) to test for correlation between the fibre fractions and the DM concentration in the silage. The models, expressed as estimates of multiple regression coefficients (Table 6.4 and Table 6.6), are presented graphically, following a procedure described by Connolly and Wachendorf (2001). Predictions for variables are plotted as lines, one each for different levels of one variable. The range chosen for prediction from the independent variables was selected to exclude values close to the observed minimum and maximum of the variable, and predictions outside the range of the observed data were excluded.

## 6.3 Results and Discussion

### 6.3.1 Biomass yield and chemical composition of silage

Fresh matter (FM) yield of the sward increased from 7.9 t ha<sup>-1</sup> at the first sampling date to a maximum of 16.0 t ha<sup>-1</sup> at the final sampling date (Figure 6.2A) and DM concentration in the silage varied between 175.1 and 390.8 g kg<sup>-1</sup> FM (Figure 6.2B). While FM yield and DM concentration showed tremendous fluctuations during growth, DM yield increased until 24/05 and then levelled off at approximately 3.6 t ha<sup>-1</sup>, while NDF concentration strictly increased from the first to the last sampling (402.7 to 639.7 g kg<sup>-1</sup> DM).



**Figure 6.2** (A) FM and DM yield of the grassland and (B) concentration of NDF and DM concentration in the silage at eight consecutive sampling dates.

Ash concentration of the silage was in a range from 76.5 to 101.2 g kg<sup>-1</sup> DM, with the highest values at the third and fourth sampling dates, when the DM concentrations were lowest (Table 6.2). The accumulation of biomass in t DM ha<sup>-1</sup> was comparable to that in a two-year experiment in Northern Germany on a *Molinio-Arrhenatherethea*, where the DM yield was 0.5 t ha<sup>-1</sup> on 27 April, increased to 3.5 t ha<sup>-1</sup> on 05 June and stayed on this level until 21 June (Kornher et al., 1997). The DM concentration of the herbage did not increase linearly with increasing maturity of the sward. Other effects such as weather conditions (Haigh, 1990) and the time of day (Delagarde et al., 2000) have strong influences on the DM concentration of grassland herbage, which in turn has a strong influence on the DM concentration of the silage (Haigh, 1990). The NDF concentration increased continuously over time, which was also found by Čop et al. (2009) for five different grass species with NDF concentrations increasing from 340-390 g kg<sup>-1</sup> DM on 18 April to 570-700 g kg<sup>-1</sup> DM on 6 June. Due to this steady increase, the NDF concentration was considered as a parameter representing the maturity of the sward in regression models.

A continuous decrease with proceeding growth was detected for the concentrations of K (26.8 to 17.4 g kg<sup>-1</sup> DM), Ca (5.4 to 3.6 g kg<sup>-1</sup> DM), N (26.5 to 12.5 g kg<sup>-1</sup> DM), S (2.0 to 1.5 g kg<sup>-1</sup> DM) and P (3.1 to 2.3 g kg<sup>-1</sup> DM). In contrast, the concentration of Cl increased slightly with progressive sampling date from 2.0 to 2.5 g kg<sup>-1</sup> DM. The concentration of Mg (1.7-1.5 g kg<sup>-1</sup> DM) did not show any directed development over time.

Few studies have dealt with the concentrations of mineral elements in grassland swards at different stages of maturity (Wilman et al., 1994). In a sampling of *Lolium perenne* (perennial ryegrass) at five consecutive development stages (from unemerged to dead leaves), Wilman et al. (1994) showed that concentrations of K and N declined, while concentrations of Mg and Ca increased. In an experiment by Pritchard et al. (1964), where whole plants of *Phleum pratense* (Timothy) were cut at five consecutive dates from 25 May to 29 June, the concentrations of K, Mg and Ca decreased from 25 to 15 g kg<sup>-1</sup> DM, from 1.0 to 0.8 g kg<sup>-1</sup> DM and from 3.7 to 2.5 g kg<sup>-1</sup> DM, respectively. Apart from Mg, the values are of the same magnitude as in the present study, where concentrations of K, N and Ca declined, while Mg concentration did not change. Chiy and Phillips (1997) found an increase in Cl concentrations of *Lolium perenne* leaves with advancing leaf maturity, which was also found in this study.

**Table 6.2** Dry matter concentration (g kg<sup>-1</sup> FM), ash concentration (g kg<sup>-1</sup> DM) and elemental composition (g kg<sup>-1</sup> DM) of the silage, the press cake (PC) and press fluid (PF) with (+hc) and without (-hc) hydrothermal conditioning at eight consecutive sampling dates.

		27/04	02/05	09/05	16/05	24/05	31/05	11/06	21/06
DM	Silage	266.3	301.7	175.1	206.4	339.3	290.2	390.8	252.6
	PC <sub>-hc</sub>	390.1	420.9	378.9	406.5	481.4	453.0	500.1	459.2
	PC <sub>+hc</sub> †	392.3±7.2	401.7±6.8	388.4±7.5	414.8±7.8	420.3±11.5	451.5±6.8	415.3±10.4	435.2±11.6
	PF <sub>-hc</sub>	120.9	136.1	63.5	77.5	114.4	89.4	118.3	74.3
	PF <sub>+hc</sub> †	26.6±0.7	29.2±0.9	21.3±0.4	23.4±0.6	27.5±0.7	24.6±0.7	26.0±0.9	20.5±0.4
Ash	Silage	89.2	82.6	95.5	101.2	84.9	85.9	76.5	85.4
	PC <sub>-hc</sub>	82.8	81.4	86.0	95.8	77.9	79.1	72.0	82.6
	PC <sub>+hc</sub>	59.2±2.4	53.4±0.7	58.9±0.8	66.5±1.3	55.5±0.6	52.7±1.0	53.9±0.5	60.9±1.6
	PF <sub>-hc</sub>	113.5	87.8	126.7	119.2	131.5	128.4	123.9	100.1
	PF <sub>+hc</sub>	123.7±1.0	119.3±1.8	121.4±1.6	130.8±1.9	136.5±2.3	137.8±2.3	132.4±1.6	126.5±3.7
K	Silage	26.8	26.8	27.4	24.3	22.4	19.6	18.7	17.4
	PC <sub>-hc</sub>	16.4	17.2	10.9	10.0	13.6	10.1	12.2	8.4
	PC <sub>+hc</sub>	7.0±1.12	6.0±0.55	3.5±0.34	3.6±0.29	4.6±0.25	3.4±0.28	4.0±0.14	2.5±0.12
	PF <sub>-hc</sub>	66.2	68.1	81.3	72.6	81.0	79.0	87.2	65.4
	PF <sub>+hc</sub>	49.7±0.4	52.9±1.1	44.3±0.7	42.0±0.8	53.7±1.2	45.0±0.8	55.4±1.5	42.3±0.6
Mg	Silage	1.7	1.5	1.8	1.6	1.5	1.4	1.5	1.5
	PC <sub>-hc</sub>	1.3	1.1	0.9	0.9	1.1	0.9	1.0	0.9
	PC <sub>+hc</sub>	1.1±0.02	0.9±0.02	0.7±0.02	0.7±0.01	0.8±0.02	0.7±0.02	0.8±0.02	0.6±0.02
	PF <sub>-hc</sub>	3.2	3.2	4.7	4.0	4.2	4.5	6.8	4.7
	PF <sub>+hc</sub>	2.4±0.04	2.3±0.05	2.6±0.03	2.4±0.04	2.8±0.04	2.6±0.03	3.3±0.02	2.9±0.04
Ca	Silage	5.4	4.8	5.3	4.7	4.5	4.1	3.8	3.6
	PC <sub>-hc</sub>	4.8	4.3	4.0	3.5	3.6	3.1	3.0	2.5
	PC <sub>+hc</sub>	4.4±0.06	4.0±0.05	3.3±0.17	3.0±0.12	3.1±0.07	2.5±0.04	2.6±0.03	2.0±0.05
	PF <sub>-hc</sub>	7.7	7.0	9.6	8.8	10.5	10.4	12.2	9.5
	PF <sub>+hc</sub>	6.5±0.07	5.9±0.09	6.7±0.11	6.1±0.09	6.9±0.08	6.6±0.09	6.8±0.1	6.3±0.06
Cl	Silage	2.0	2.0	2.1	2.3	2.3	2.3	2.3	2.5
	PC <sub>-hc</sub>	1.2	1.2	0.8	0.8	1.3	1.1	1.4	1.1
	PC <sub>+hc</sub>	0.4±0.06	0.3±0.02	0.2±0.02	0.3±0.02	0.4±0.01	0.3±0.02	0.4±0.01	0.3±0.01
	PF <sub>-hc</sub>	5.0	5.4	6.4	7.4	9.0	9.8	11.8	10.0
	PF <sub>+hc</sub>	3.8±0.04	4.1±0.11	3.4±0.05	4.0±0.09	5.6±0.14	5.4±0.13	7.0±0.21	6.2±0.11
N	Silage	26.5	22.3	19.0	17.3	15.0	13.0	13.0	12.5
	PC <sub>-hc</sub>	19.9	17.3	13.4	11.7	12.0	9.7	10.9	9.4
	PC <sub>+hc</sub>	15.3±0.83	12.8±0.28	11.1±0.08	10.0±0.11	9.5±0.14	7.7±0.19	9.0±0.09	7.8±0.10
	PF <sub>-hc</sub>	51.5	44.0	37.1	36.3	34.9	33.6	35.2	28.8
	PF <sub>+hc</sub>	39.3±0.3	34.3±0.4	24.5±0.2	23.6±0.3	24.7±0.3	21.3±0.3	23.0±0.3	20.2±0.2
S	Silage	2.0	1.7	1.7	1.6	1.6	1.5	1.6	1.5
	PC <sub>-hc</sub>	1.4	1.2	1.2	1.0	1.1	1.0	1.2	1.0
	PC <sub>+hc</sub>	1.1±0.05	0.9±0.04	0.8±0.04	0.8±0.02	0.8±0.02	0.6±0.02	0.8±0.01	0.7±0.01
	PF <sub>-hc</sub>	4.3	3.9	3.3	3.6	4.9	4.6	5.8	4.2
	PF <sub>+hc</sub>	3.0±0.02	2.7±0.03	2.3±0.02	2.3±0.02	3.0±0.05	2.8±0.04	3.6±0.09	2.8±0.04
P	Silage	3.1	2.9	2.9	2.8	2.6	2.5	2.4	2.3
	PC <sub>-hc</sub>	1.8	1.6	1.2	1.2	1.5	1.2	1.5	1.1
	PC <sub>+hc</sub>	0.9±0.12	0.7±0.05	0.6±0.05	0.6±0.04	0.6±0.03	0.5±0.04	0.6±0.02	0.5±0.02
	PF <sub>-hc</sub>	8.0	8.5	8.5	8.2	9.9	10.6	11.9	8.7
	PF <sub>+hc</sub>	5.7±0.02	5.7±0.11	4.5±0.05	4.7±0.07	6.1±0.13	5.6±0.09	6.8±0.18	5.2±0.08

†Mean value ± s.e. of mean of five PC/PF after hydrothermal conditioning at 10, 30, 50, 70, and 90°C

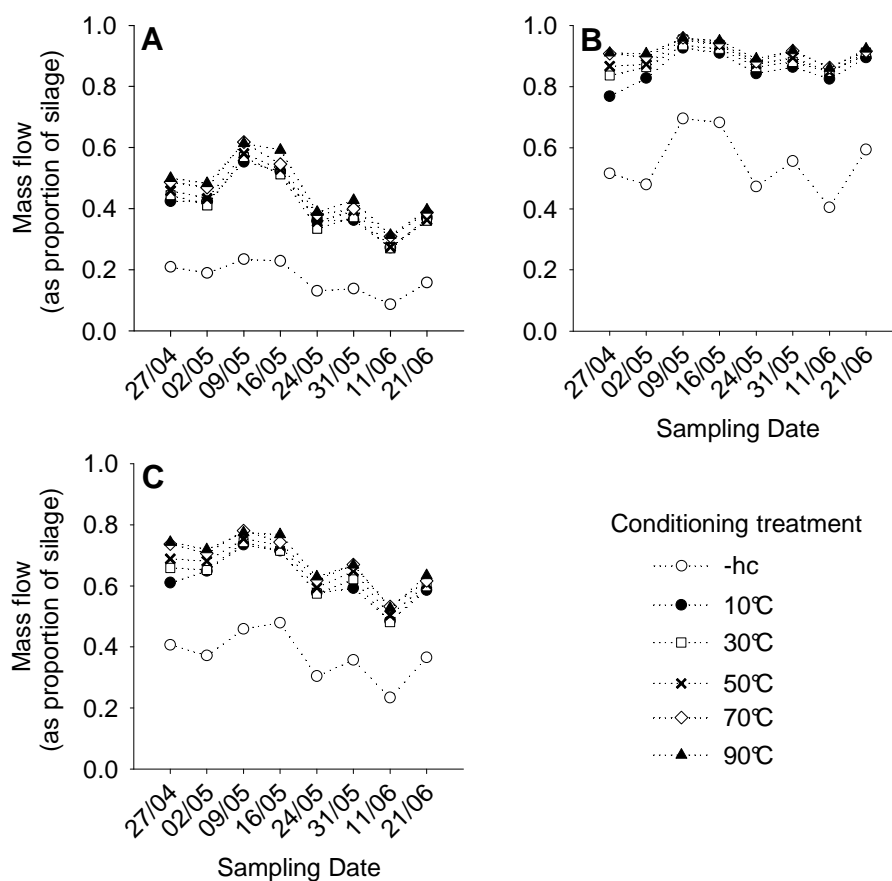
### 6.3.2 Influence of sward maturity and hydrothermal conditioning on mass flows into the PF

PF after mechanical dehydration without hydrothermal conditioning (PF<sub>-hc</sub>) had DM concentrations of 63.5-136.1 g kg<sup>-1</sup> FM and ash concentrations of 87.8-131.5 g kg<sup>-1</sup> DM (Table 6.2). Due to the addition of water, PF obtained through mechanical dehydration with prior hydrothermal conditioning (PF<sub>+hc</sub>) had lower DM concentrations of 20.5-29.2 g kg<sup>-1</sup> FM. Ash concentrations were slightly higher (119.3-137.8 g kg<sup>-1</sup> DM). Concentrations of elements were higher in the PF compared to the silage and higher in PF<sub>-hc</sub> compared to PF<sub>+hc</sub>. Compared to the silage, mean concentration in PF<sub>+hc</sub> was 1.6-3.0 times higher for K, 1.4-2.2 for Mg, 1.2-1.8 for Ca, 1.6-3.1 for Cl, 1.3-1.8 for N, 1.4-2.3 for S and 1.6-2.8 for P.

Proportionately, 0.09-0.23 of the DM contained in the silage was transferred into the PF during mechanical dehydration without hydrothermal conditioning (PF<sub>-hc</sub>), while 0.27-0.62 of the DM was transferred when a hydrothermal conditioning (10-90°C) was applied beforehand (Figure 6.3A). The mass flows of organic compounds (CP, EE, NDF, ADF, ADL) were comparable to those of DM regarding 10-90°C, but somewhat lower regarding PF<sub>-hc</sub> (data not shown). The highest mass flows were obtained for K (0.40-0.70 for -hc and 0.77-0.96 for 10-90°C, Figure 6.3B) and Cl (0.44-0.73 for -hc and 0.86-0.96 for 10-90°C, data not shown). Mass flows of N were between 0.23 and 0.48 (-hc) and between 0.48 and 0.78 (10-90°C) (Figure 6.3C). Mass flows of the other elements (Mg, Ca, S, P) and the ash were in the range between N and K (data not shown). For all plant compounds, mass flows were maximum at the third sampling date and lowest at the seventh sampling date.

Mass flows of DM and ash were higher compared to an experiment with five semi-natural grassland swards, where the biomass was sampled in July and August at a higher stage of maturity, and mass flows of 0.3 and 0.44 were obtained for DM and ash, respectively, in a treatment with hydrothermal conditioning at 60°C (Wachendorf et al., 2009). In the present study, K mass flows were in the same range, while N mass flows were considerably higher compared to Wachendorf et al. (2009), where a mean N mass flow of 0.42 was determined. N occurs in a plant in two forms, protein-N (PN) and non-protein-N (NPN), comprising inorganic-N compounds (e.g. nitrate) or soluble organic-N compounds (e.g. amino-acids). In young plant tissue, the concentrations of NPN and soluble PN are much higher than in mature tissue, where N occurs predominantly in structurally insoluble proteins (Mattson, 1980). Due to this occurrence of N in different forms, the N mass flow, which describes the soluble N fraction that is transferred into the PF, is higher in the younger plant tissues of the present

study (sampled between April and June) compared to the older plant tissue described in Wachendorf et al. (2009). N mass flows of the first four sampling dates were also higher than those of the last four sampling dates. In contrast to N, the occurrence of K in the cell is predominantly found as ions in the vacuole and in the cytosol of plant cells (Walker et al., 1996). Therefore, it is highly mobile, even in mature plant tissue and can easily be transferred into the PF during mechanical dehydration.



**Figure 6.3** Mass flows of (A) DM, (B) K and (C) N into the press fluid without hydrothermal conditioning (-hc) and with hydrothermal conditioning at 10, 30, 50, 70, 90°C at eight consecutive sampling dates.

In order to conserve the productivity of grasslands over a long time, nutrients exported with the harvest need to be replaced. The transformation of carbon from PF into methane and from PC into solid fuel does not allow an appreciable return of carbon. However, as grassland plants transfer proportionally 0.3 to 0.5 of assimilates below ground and mineralisation processes on permanent grassland occur at very low rates, a net accumulation of carbon in the soil is accomplished (Kuzuyakov and Domanski, 2000). Mass flows of elements into the PF indicate the amount of each element in the herbage that becomes available in the digestate as a fertilizer after biogas production. As regulations in many European countries restrict the

application of ash as grassland fertilizer or even prohibit it, high mass flows of N and minerals into the PF are permissible in order to achieve a neutral nutrient balance, which is an essential aspect of sustainable sward management.

Regarding essential plant nutrients, 0.62-0.68 of N, 0.84-0.90 of P, 0.86-0.91 of K, 0.70-0.75 of Mg, 0.59-0.65 of Ca, and 0.70-0.75 of S in the herbage were transferred into the PF and were thus available for the replacement of nutrients withdrawn with the sampling. Through the anaerobic digestion process, organically bound elements are mineralised and increase the short-term fertilization effect in comparison to conventional organic fertilizers, such as slurry or manure from animal husbandry (Weiland, 2010). Moreover, the low dry matter concentration of the PF digestate improves the flow properties, which in turn reduces the adhesion to plant surfaces and supports a fast penetration into the soil, hence resulting in a reduced risk of nitrogen losses through ammonia emissions (Weiland, 2010).

In a monitoring program of 96 Austrian biogas plants, mean concentrations in the digestates were determined for K ( $66.5 \text{ g kg}^{-1} \text{ DM}$ ), Mg ( $12.8 \text{ g kg}^{-1} \text{ DM}$ ), Ca ( $30.7 \text{ g kg}^{-1} \text{ DM}$ ) and N ( $81 \text{ g kg}^{-1} \text{ DM}$ ) (Pötsch et al., 2004). Most of these biogas plants were fed by animal slurry with K, Mg, Ca and N concentrations of 50.1, 7.3, 19.2 and  $42.7 \text{ g kg}^{-1} \text{ DM}$  (average of 1639 cattle slurry samples; Pötsch et al., 2004), respectively, which is higher than the grassland silage used in the present study. The IFBB PF had higher element concentrations when obtained through mechanical dehydration without prior hydrothermal conditioning. Regarding K concentrations, these PF were in a comparable range ( $65.4\text{-}87.2 \text{ g kg}^{-1} \text{ DM}$ ) to biogas slurry, while hydrothermal conditioning resulted in lower K concentrations of  $42.3\text{-}55.4 \text{ g kg}^{-1} \text{ DM}$ , which is comparable to cattle slurry. Concerning the other nutrients, concentrations in PF<sub>-hc</sub> were 0.44-0.93 (Mg), 0.40-0.64 (Ca) and 0.67-1.21 (N) of those of cattle slurry, whereas concentrations in PF<sub>+hc</sub> were slightly lower, at 0.32-0.45 (Mg), 0.31-0.36 (Ca) and 0.47-0.92 (N) of those of cattle slurry.

The repeated measurement ANOVA showed significant treatment (T) and sampling period (SP) effects for mass flows of all plant compounds in PF (Table 6.3). Significant T x SP interactions indicate variable conditioning effects for EE, NDF, ADF, K, Cl and P, depending on the sampling date. For ash, ADL, NFC, Mg, N and S, the repeated statement was removed from the model as the null model likelihood ratio test was not significant.



**Table 6.3** Levels of significance of ANOVA for the effects of treatment (T), sampling period (SP) and T x SP, and for the estimate of the repeated measurement effect (LRT) on mass flow of plant compounds into the press fluid with (10°C) and without (-hc) hydrothermal conditioning of grassland silage.

Plant compound	Treatment†		Level of significance (P-value)			
	-hc	10°C	LRT‡	Treatment (T)	Sampling period (SP)	T x SP
DM	0.17 ± 0.02	0.41 ± 0.03	*	**	**	ns
Ash	0.22 ± 0.02	0.60 ± 0.03	ns	***	**	ns
EE	0.14 ± 0.02	0.40 ± 0.03	**	**	**	*
NDF	0.06 ± 0.01	0.26 ± 0.03	*	***	**	**
ADF	0.07 ± 0.01	0.30 ± 0.03	*	**	**	*
ADL	0.23 ± 0.02	0.48 ± 0.04	ns	***	**	ns
NFC	0.30 ± 0.03	0.60 ± 0.03	ns	**	*	ns
K	0.55 ± 0.04	0.86 ± 0.02	**	*	**	*
Mg	0.46 ± 0.03	0.70 ± 0.03	ns	***	**	ns
Ca	0.35 ± 0.02	0.59 ± 0.02	***	*	**	ns
Cl	0.58 ± 0.04	0.89 ± 0.01	*	*	*	*
N	0.37 ± 0.03	0.62 ± 0.03	ns	***	**	ns
S	0.43 ± 0.02	0.70 ± 0.02	ns	***	*	ns
P	0.57 ± 0.03	0.84 ± 0.01	***	*	*	*

†Values represent least square means (± s.e. of mean) per treatment

‡LRT, null model likelihood ratio test; the null model, which excludes the specified covariance structure of repeated measurements, was used if not significant ( $P > 0.05$ )

ns, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

As part of the dehydration of the silage, plant cells which contain the liquid phase have to be macerated before the separation of solid and liquid can take place. Screw presses, which provide a high degree of maceration of cell walls due to axial movement and abrasion of the tissue under high pressure, are also used in bio-refineries, where lactic acids and amino-acids are extracted from herbage (Kromus et al., 2004). In order to enhance the effect of plant cell maceration, the hydrothermal conditioning treatment was introduced prior to mechanical separation with the screw press (Wachendorf et al., 2009). Previous studies have shown higher mass flows of most plant compounds into the PF at higher conditioning temperatures (60-80°C) compared to those of ambient conditioning temperatures (5-15°C) (Richter et al., 2009a, Wachendorf et al., 2010). In this study, however, differences between the five temperature treatments (10-90°C) were pronounced at the beginning, but rather low at the end of the sampling period. In contrast, mass flows of the treatment without hydrothermal conditioning were significantly ( $P < 0.05$ ) lower for all plant compounds.

When sampling date was replaced by fibre concentration, prediction accuracy of regression equations was highest when NDF was used as a variable representing sward maturity. Pearson's correlation test between NDF and DM in the silage showed a low correlation coefficient ( $r = 0.37$ ), so both variables were used in the analysis. Mass flows of different plant compounds could be explained with a high accuracy by including the NDF concentration of the silage, the temperature of hydrothermal conditioning and the DM concentration of the silage, as well as their quadratic terms and twofold interactions in the regression model (Table 6.4). Coefficients of determination ranged between  $R^2 = 0.86$  for CI and 0.99 for DM.

**Table 6.4** Coefficient of determination and parameter estimates for the models of mass flow of plant compounds into the press fluid.

	<b>DM</b>	<b>Ash</b>	<b>EE</b>	<b>NDF</b>	<b>ADF</b>	<b>ADL</b>	<b>NFC</b>
$R^2$	0.99***	0.94***	0.92***	0.96***	0.98***	0.93***	0.95***
F value	488.18	71.18	59.84	144.46	311.15	114.30	89.00
Estimates:							
Intercept	-0.0225ns	0.1665ns	-0.4565ns	0.4288ns	0.3747*	0.2162ns	-1.0173**
NDF†	0.0036***	0.0041***	0.0048***	0.0021**	0.0027***	0.0026*	0.0067***
NDF <sup>2</sup>	-0.000004***	-0.000006***	-0.000005***	-0.000003***	-0.000004***	-0.000003**	-0.000004***
T‡	0.0010*	0.0029***	0.0008ns	-0.0009ns	0.0005ns	0.0019***	0.0035***
T <sup>2</sup>	0.000008**	Ns	0.00002*	0.00001**	0.00001**	ns	ns
DM¶	-0.0019***	-0.0010ns	-0.0028**	-0.0037***	-0.0040***	-0.0008***	-0.0006ns
DM <sup>2</sup>	0.000002***	-0.000005*	ns	ns	ns	ns	0.000007***
NDF x T	-0.000002**	-0.000004**	ns	ns	ns	ns	-0.000004*
NDF x DM	ns	0.000006*	0.000003*	0.000005***	0.000005***	ns	-0.000008**
	<b>K</b>	<b>Mg</b>	<b>Ca</b>	<b>CI</b>	<b>N</b>	<b>S</b>	<b>P</b>
$R^2$	0.93***	0.95***	0.95***	0.86***	0.98***	0.92***	0.88***
F value	68.93	94.58	168.39	33.61	228.38	42.33	50.45
Estimates:							
Intercept	0.3343**	-0.4007ns	-0.0479ns	0.4781**	-0.2152ns	0.1735ns	0.2040ns
NDF	0.0021***	0.0054***	0.0033***	0.0014***	0.0037***	0.0027**	0.0026***
NDF <sup>2</sup>	-0.000002***	-0.000003***	-0.000003***	ns	-0.000004***	-0.000004***	-0.000002***
T	0.0035***	-0.0006ns	0.0008***	0.0023***	0.0023***	0.0017*	0.0026***
T <sup>2</sup>	-0.000007*	0.00001**	ns	ns	ns	0.000009*	ns
DM	-0.0005***	-0.0017**	-0.0012***	0.00005ns	0.0005ns	-0.0009ns	-0.0003***
DM <sup>2</sup>	ns	0.000007***	ns	0.000003**	-0.000002***	-0.000004*	ns
NDF x T	-0.000004***	Ns	ns	-0.000004***	-0.000003**	-0.000004**	-0.000004***
NDF x DM	ns	-0.000006**	ns	-0.000004**	ns	0.000004*	ns

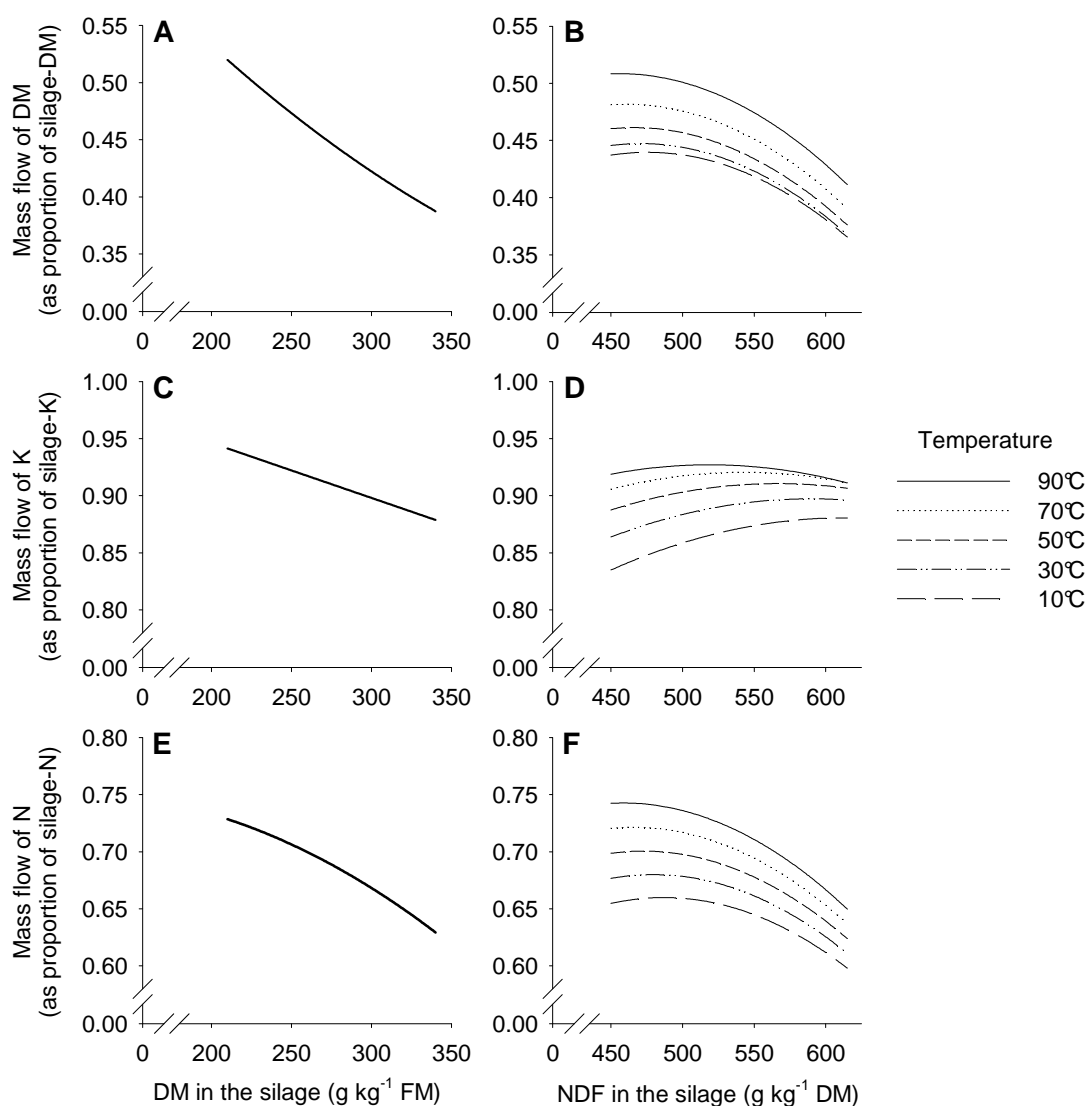
†NDF in the silage in  $\text{g kg}^{-1}$  DM

‡T in °C

¶DM in the silage in  $\text{g kg}^{-1}$  FM

ns, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

DM in the silage had a negative impact on mass flow in the PF of DM, K and N (Figure 6.4), whereas NDF was negatively related to mass flow of DM and N, but showed hardly any impact on mass flow of K. Temperature during hydrothermal conditioning had a positive impact on mass flow of all plant compounds, but was more pronounced for DM, K and N at low levels of NDF (Figure 6.4B, D, F).



**Figure 6.4** Predictions of mass flow of DM, K and N: (A, C, E) DM concentration in the silage ( $\text{g kg}^{-1}$  FM) at a mean temperature of hydrothermal conditioning of  $50^{\circ}\text{C}$  and a mean NDF concentration in the silage of  $533 \text{ g kg}^{-1}$  DM and (B, D, F) the interaction of NDF concentration in the silage with temperature of hydrothermal conditioning at a mean DM concentration in the silage of  $280 \text{ g kg}^{-1}$  FM.

The multiple regression models showed that the main parameters influencing the mass flow are the temperature of hydrothermal conditioning as well as the NDF and DM concentrations of the silage. High temperatures of hydrothermal conditioning might contribute to a better maceration of the cell walls compared to low temperatures, which was also found by

Wachendorf et al. (2009) and for lignocellulosic material in general by Garotte et al. (1999). High water content in the silage may lead to a soaked condition of the cell walls, which results in a higher degree of maceration during the press process. In contrast to conditioning water, which affects the plant tissue mainly from the outside of the cells, escape of cell water, which is contained mainly in the vacuole and cytoplasm, may further contribute to the rupture of cell walls during compression, resulting in an enhanced discharge of cell compounds.

The reason for lower mass flows of N with increasing NDF concentrations may be the higher concentration of soluble N fractions at an early development stage of plants, whereas the mass flow of the highly mobile K is not affected by NDF concentration. Mass flows were highest at the highest temperature of hydrothermal conditioning, the lowest DM concentration in the silage and - with the exception of K mass flow - the lowest NDF concentration in the silage.

### **6.3.3 Influence of sward maturity and hydrothermal conditioning on fuel-relevant properties of press cakes**

Through mechanical dehydration without hydrothermal conditioning, the DM concentration of the press cake (PC<sub>-hc</sub>) was increased compared to the silage by up to 206.6 g kg<sup>-1</sup> FM to values between 378.9 and 500.1 g kg<sup>-1</sup> FM (Table 6.2). Compared to the silage fuel, quality was improved by reduced concentrations of ash (reduction of 0.11-0.10), K (0.35-0.60), Mg (0.24-0.50), Cl (0.40-0.65), N (0.16-0.32) and S (0.25-0.38). Ca (0.11-0.31) and P (0.38-0.59) concentrations were also reduced, but this did not have any positive effect on fuel quality. PC produced by hydrothermal conditioning prior to mechanical dehydration (PC<sub>+hc</sub>) led to DM concentrations of 388.4 to 451.5 g kg<sup>-1</sup> FM on average of five temperature treatments. Low standard errors of mean (6.8-11.6 g kg<sup>-1</sup> FM) suggested a low variation between the different temperature treatments. Improvement of fuel quality was higher than in PC<sub>-hc</sub>, reductions of ash (0.42-0.52), K (0.74-0.84), Mg (0.33-0.63), Ca (0.17-0.44), Cl (0.79-0.89), N (0.31-0.43), S (0.44-0.57) and P (0.71-0.80) being achieved, again with standard errors of mean indicating a low variation between temperature treatments.

Hydrothermal conditioning resulted in a significant ( $P < 0.05$ ) reduction of ash, K, Mg, Cl and P of 30.91, 6.72, 0.23, 0.71 and 0.64 kg<sup>-1</sup> DM in PC<sub>+hc</sub> compared to PC<sub>-hc</sub> (Table 6.5), whereas Ca, N and S concentrations were not reduced significantly. Except for ash and S, element concentrations decreased significantly with the delay of sampling. Ash softening temperature (AST) in PC<sub>+hc</sub> (1138-1192°C) was significantly higher than in PC<sub>-hc</sub> (1101-1143°C) and

more so than in the silage (1016-1060°C) (data not shown). While AST in the silage slightly increased during growth, there was no consistent effect of sampling date on AST in PC.

**Table 6.5** Levels of significance of ANOVA for the effects of treatment (T), sampling period (SP) and T x SP, and for the estimate of the repeated measurement effect (LRT) on concentrations (g kg<sup>-1</sup> DM) of ash and various elements in the press cake with (10°C) and without (-hc) hydrothermal conditioning of grassland silage.

Variable	Treatment†		Level of significance (P-value)			
	-hc	10°C	LRT‡	Treatment (T)	Sampling period (SP)	T x SP
ash	82.19 ± 2.44	58.28 ± 1.69	ns	***	ns	ns
K	12.35 ± 1.12	5.63 ± 0.90	**	*	**	ns
Mg	1.01 ± 0.05	0.78 ± 0.05	ns	**	*	ns
Ca	3.60 ± 0.26	3.21 ± 0.27	ns	Ns	**	ns
Cl	1.11 ± 0.08	0.40 ± 0.03	ns	***	*	ns
N	13.04 ± 1.31	11.11 ± 1.16	***	Ns	**	ns
S	1.14 ± 0.05	0.85 ± 0.06	*	Ns	ns	ns
P	1.39 ± 0.09	0.75 ± 0.07	ns	***	*	ns

†Values represent least square means (± s.e. of mean) per treatment

‡LRT, null model likelihood ratio test; the null model, which excludes the specified covariance structure of repeated measurements, was used if not significant ( $P > 0.05$ )

NS, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

Although the parameter settings of the conditioning unit and screw press were identical, the DM concentrations of the PC<sub>+hc</sub> were somewhat lower than in previous studies with five semi-natural montane grasslands, where DM concentrations of 464-543 g kg<sup>-1</sup> FM were found (Richter et al., 2010a), and with three semi-natural alluvial grasslands, where 428-499 g kg<sup>-1</sup> FM were determined (Richter et al., 2009a). Overall, DM concentrations of PC were 0.6 higher than in the parent silage. DM concentration of PC<sub>+hc</sub> was slightly lower than that of PC<sub>-hc</sub>, which may be due to the addition of water as a conditioning liquid. Hydrothermal conditioning significantly enhanced the reduction in ash and single elements compared to dehydration without prior conditioning. Woodchips of spruce, a common biofuel, have ash concentrations of 10-25 g kg<sup>-1</sup> DM, K concentrations of 0.9-1.5 g kg<sup>-1</sup> DM, Mg concentrations of 0.3-0.8 g kg<sup>-1</sup> DM and Ca concentrations of 2.9-7.0 g kg<sup>-1</sup> DM (Van Loo and Koppejan, 2008). In both PC<sub>+hc</sub> and PC<sub>-hc</sub>, ash and K concentrations were higher, whereas Mg and Ca concentrations were in the same range.

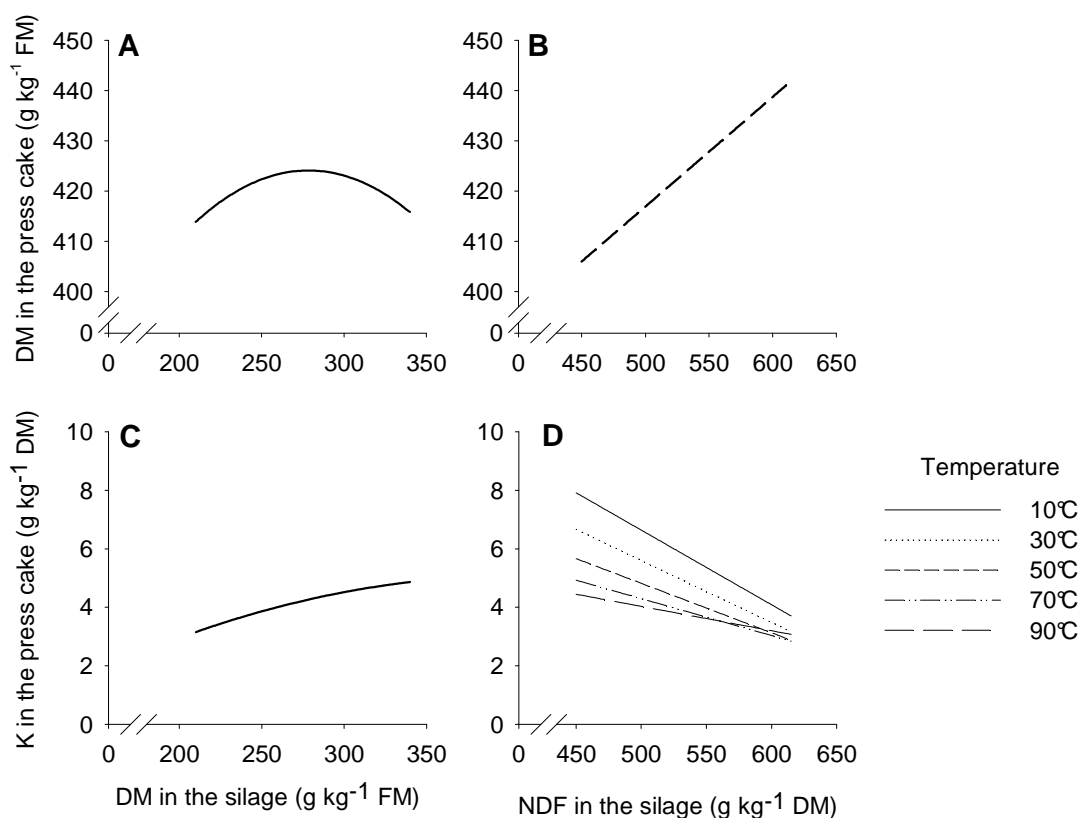
According to Obernberger et al. (2006), problems with ash melting occur at K concentrations in the ash above 70 g kg<sup>-1</sup> ash and Ca concentrations in the ash below 150 g kg<sup>-1</sup> ash. Only at the eighth sampling date, the K concentration in all PC<sub>+hc</sub> treatments (35-49 g kg<sup>-1</sup> ash) was

below the limit, whereas the K concentration in PC<sub>-hc</sub> (102-211 g kg<sup>-1</sup> ash) was above the limit at all sampling dates. Regarding Ca, both PC<sub>-hc</sub> (30-58 g kg<sup>-1</sup> ash) and PC<sub>+hc</sub> (28-91 g kg<sup>-1</sup> ash) had concentrations in the critical range. However, AST of PC<sub>+hc</sub> as estimated according to Hartmann (2001) were strongly increased compared to PC<sub>-hc</sub> and more so to the parent material, which is due to the significantly reduced K and Mg concentrations and the increased Ca concentration. There was a sharp decline in K and Mg concentrations at the start of May, causing a maximum for AST at the third sampling date. AST decreased thereafter, as the concentration of Ca declines with advancing sward maturity, whereas K and Mg remained on a constant low level. At 1138-1192°C, AST for PC<sub>+hc</sub> are close to the range of woodchips from willow (1200-1300°C; Hartmann, 2001).

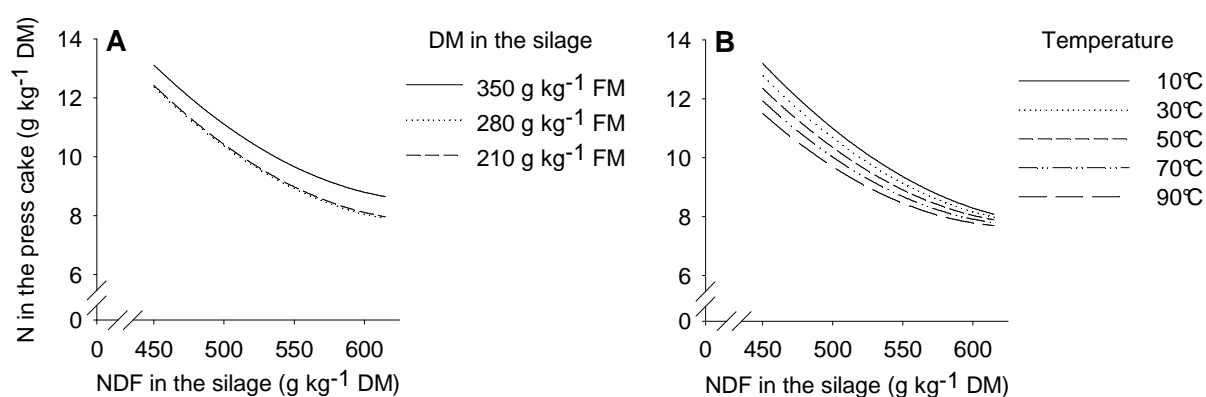
According to Launhardt and Thoma (2000), emission related problems during biomass combustion occur at concentrations of Cl (emission as HCl and PCDD/F) above 1.5 g kg<sup>-1</sup> DM, of N (emissions as NO<sub>x</sub>) above 6 g kg<sup>-1</sup> DM and of S (emissions as SO<sub>x</sub>) above 1 g kg<sup>-1</sup> DM (Oberberger et al., 2006). Compared with woodchips of spruce, where Cl, N and S concentrations (0.05-0.06, 0.9-1.7 and 0.07-1.0 g kg<sup>-1</sup> DM, respectively; Van Loo and Koppejan, 2008) are well below the threshold, both PC<sub>-hc</sub> and PC<sub>+hc</sub> were below the threshold regarding Cl, but above it regarding N. However, at the eighth sampling date, the N concentration of PC<sub>+hc</sub> was close to the critical value. The S concentration was below the limit only for PC<sub>+hc</sub>, apart from the first sampling date.

DM concentration in the silage had no marked effect on the DM level in PC (Figure 6.5A). The latter, however, increased by 36 g kg<sup>-1</sup> FM, with an increase in NDF concentration in the silage from 450 to 615 g kg<sup>-1</sup> DM (Figure 6.5B). DM content in the silage had a slightly positive effect on K concentration in PC (Figure 6.5C). NDF interacted significantly with the level of temperature in hydrothermal conditioning, with K concentration showing a negative response to increased NDF (Figure 6.5D). At low levels of NDF, reduction in K due to increased temperature from 10 to 90°C was much stronger than at high levels of NDF.

NDF in the silage had a strong negative effect on N concentration in PC, but interacted significantly with DM concentration in the silage and temperature of hydrothermal conditioning (Figure 6.6). N in PC decreased with decreasing DM content in the silage (Figure 6.6A) and with increasing temperature (Figure 6.6B). Reduction due to temperature was more pronounced at low levels of NDF.



**Figure 6.5** Predictions of DM and K concentration in the press cake: (A, C) DM concentration in the silage ( $\text{g kg}^{-1}$  FM) at a mean temperature of hydrothermal conditioning of  $50^{\circ}\text{C}$  and a mean NDF concentration in the silage of  $533 \text{ g kg}^{-1}$  DM; (B) NDF concentration in the silage ( $\text{g kg}^{-1}$  DM) at a mean temperature of hydrothermal conditioning of  $50^{\circ}\text{C}$  and a mean DM concentration in the silage of  $280 \text{ g kg}^{-1}$  FM; (D) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}$  DM) with temperature of hydrothermal conditioning ( $^{\circ}\text{C}$ ) at a mean DM concentration in the silage of  $280 \text{ g kg}^{-1}$  FM.



**Figure 6.6** Predictions of N concentration in the press cake: (A) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}$  DM) with DM concentration in the silage ( $\text{g kg}^{-1}$  FM) at a mean temperature of  $50^{\circ}\text{C}$ ; (B) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}$  DM) with temperature of hydrothermal conditioning ( $^{\circ}\text{C}$ ) at a mean DM concentration in the silage of  $280 \text{ g kg}^{-1}$  FM.

The models obtained by multiple regression analysis for DM, ash and Cl concentration in PC had low  $R^2$  of 0.50, 0.36 and 0.70, respectively (Table 6.6).  $R^2$  for K, Mg, Ca, N, S and P ranged between 0.84 and 0.96.

**Table 6.6** Coefficient of determination and parameter estimates for the models of concentration of plant compounds in the press cake.

	DM	Ash	K	Mg	Ca	Cl	N	S	P
$R^2$	0.50***	0.36***	0.93***	0.87***	0.95***	0.70***	0.96***	0.88***	0.84***
F value	11.85	21.42	75.51	38.49	213.39	12.85	135.78	39.00	27.86
Estimates:									
Intercept	138.315*	70.6399***	13.6559***	6.1637***	7.5308***	1.8157**	73.2710***	5.7572***	5.0457***
NDF <sup>†</sup>	0.2183***	ns	-0.0277***	-0.0184***	-0.0096***	-0.0045**	-0.1836***	-0.0150***	-0.0125***
NDF <sup>2</sup>	ns	ns	ns	0.00001***	ns	ns	0.0001***	0.00001***	0.00001**
T <sup>‡</sup>	ns	ns	-0.1725***	-0.0009*	-0.0029**	-0.0074***	-0.0877***	-0.0064**	-0.0143***
T <sup>2</sup>	ns	ns	0.0003**	ns	ns	ns	ns	ns	ns
DM <sup>¶</sup>	1.2175**	-0.0468***	0.0406***	-0.0016ns	0.0030***	-0.0006ns	-0.0395**	-0.0037*	-0.0037*
DM <sup>2</sup>	-0.0022**	ns	-0.0001***	-0.00002**	ns	-0.00001*	ns	ns	ns
NDF x T	ns	ns	0.0002**	ns	ns	0.00001***	0.0001***	0.00001*	0.00002***
NDF x DM	ns	ns	ns	0.00002**	ns	0.00001*	0.00008**	0.00001**	0.00001*

<sup>†</sup>NDF in the silage in g kg<sup>-1</sup> DM

<sup>‡</sup>T in °C

<sup>¶</sup>DM in the silage in g kg<sup>-1</sup> FM

ns, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

## 6.4 Conclusions

- (i) Mass flows of plant compounds into the PF increased with increasing temperature, whereas sampling date showed inconsistent effects.
- (ii) Concentrations of elements detrimental for combustion were lower in PC compared to the silage. Increasing temperatures during hydrothermal conditioning and delayed sampling further reduced elemental concentrations in PC.
- (iii) NDF and DM in the silage and temperature during hydrothermal conditioning were key variables in determining mass flow ( $R^2$  of 0.86 to 0.99) and elemental concentration in PC ( $R^2$  of 0.36 to 0.96). Lowest PC concentrations were predicted at a combination of high temperatures, low DM concentrations and high NDF concentrations.



## **7 Influence of sward maturity and pre-conditioning temperature on the energy production from grass silage through the integrated generation of solid fuel and biogas from biomass (IFBB): 2. Properties of energy carriers and energy yield**

**Abstract** In order to determine influencing parameters on energy production of the IFBB process, herbage from a lowland hay meadow (*Arrhenaterion*) was sampled and ensiled at eight dates between 27 April and 21 June 2007. The silage from each date was processed in six IFBB treatments with and without hydrothermal conditioning at different temperatures. Methane yields and higher heating values were determined and an energy balance was calculated with whole-crop digestion (WCD) of the silage as reference system. Maximum net energy yields were 10.2 MWh ha<sup>-1</sup> for the IFBB treatment without hydrothermal conditioning and 9.0 MWh ha<sup>-1</sup> for the treatment with hydrothermal conditioning at 50°C. WCD achieved a maximum net energy yield of 3.7 MWh ha<sup>-1</sup>. Energy conversion efficiency ranged from 0.24 to 0.54 and was predicted with high accuracy by temperature of hydrothermal conditioning as well as concentration of neutral detergent fibre and dry matter in the silage ( $R^2=0.90$ ).

### **7.1 Introduction**

Due to the problems associated with global climate change and the finite nature and instability of fossil fuel supply, energy production from biomass has become an important part of agricultural business. In this context, crucial questions have arisen about competition with food production for a finite land resource, the efficiency of energy production from agricultural land and the extent of green house gas (GHG) mitigation. Competition with food production can be avoided if residual agricultural biomass is used. Beside the use of harvest residues (e.g. combustion of straw; Frandsen, 2005) or waste materials from food processing (e.g. in anaerobic co-digestion; Murto et al., 2004), an increasing amount of biomass from grasslands will become available in the future due to abandonment as a feed source for modern livestock production (Isselstein et al., 2005). These permanent grasslands are often characterized by extensive management and high biodiversity, which decreases dramatically in the case of abandonment, due to the formation of shrubs and woods in the course of natural succession (Poschlod et al., 2005). A site-adapted management for energy production purposes may help in the conservation of these low-input high-diversity (LIHD) grasslands.

However, if common conversion technologies such as anaerobic digestion are used, the energy conversion efficiency will often be low, because of high fibre concentrations, especially when the grassland biomass is harvested late in the summer due to conservation objectives (Prochnow et al., 2005; Richter et al., 2009b). The use of this biomass for combustion, another common technique, is limited due to high concentrations of elements that cause corrosion inside the combustion chamber, ash softening and hazardous emissions (Jenkins et al., 1998).

The development of the integrated generation of solid fuel and biogas from biomass (IFBB, Wachendorf et al., 2009) is aimed at increasing the efficiency of converting biomass into energy, with special regard to biomass from LIHD grasslands. In the IFBB procedure, the grassland biomass is subjected to hydrothermal conditioning and a subsequent separation with a screw press into a press fluid (PF) for biogas production and a press cake (PC) for direct combustion as solid fuel (Wachendorf et al., 2009). With the need to dry the PC to dry matter (DM) concentrations above 850 g kg<sup>-1</sup> FM in order to make it suitable for storage and pelleting, the IFBB system provides a year-round demand for heat produced in the combined heat and power plant (CHP), where the biogas is combusted. Recent research has shown that temperatures of hydrothermal conditioning and stage of sward maturity affect the properties of PC and PF as energy carriers (Richter et al., 2010b), but the effect on the internal energy fluxes and the resulting energy yield is still unclear. Richter et al. (2010b) proved that neutral detergent fibre (NDF) concentration of the silage, as a parameter representing sward maturity, together with temperature of hydrothermal conditioning and DM concentration of the silage, was able to predict mass flows of DM and various elements and plant compounds into the PF as well as their concentrations in the PC with a high accuracy.

Hence, the objective of this study was to determine the influence of sward maturity and hydrothermal conditioning on (i) the properties of PF and PC as energy carriers and (ii) the energy yield of the IFBB process as a whole. A further goal was to (iii) prove the predictive power of NDF and DM in the silage for different energy parameters.

## **7.2 Material and Methods**

### **7.2.1 Biomass processing according to IFBB**

The grass silage consisted of herbage from a permanent sub-montane hay meadow (*Arrhenaterion*) on a loamy soil in Northern Hesse, Germany (51°23' N, 9°54' E, 284 m

above sea level). During the experimental year (2007), the annual precipitation was 619 mm and the mean annual temperature was 7.8°C. The grassland has been managed with two annual cuts for hay making (late first cut at the beginning of June, second cut in August), followed by grazing with sheep and no fertilizer or further treatment for more than 10 years. Biomass was sampled and ensiled on eight consecutive dates of a prolonged spring cut (27/04, 02/05, 09/05, 16/05, 24/05, 31/05, 11/06, 21/06). Due to weather conditions, the interval between samplings varied from seven to twelve days. The botanical composition of the grassland, the methods of sampling and ensiling and the chemical composition of the silages are described elsewhere (Richter et al., 2010b). The silage from each sampling date was subjected to six different treatments. One treatment was direct mechanical dehydration without hydrothermal conditioning, the other five treatments comprised hydrothermal conditioning at different temperatures (10°C, 30°C, 50°C, 70°C, 90°C) and subsequent mechanical dehydration. The methods of hydrothermal conditioning and mechanical dehydration are described elsewhere (Richter et al., 2010b).

### 7.2.2 2.2. Chemical composition analysis

The silage and the PC were analyzed for C, H and N using an elemental analyzer (EA 1106, Carlo Erba Ltd., Rodano, Italy) and for the organic constituents crude protein (CP), ether extract (EE), neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) according to standard methods (Bassler, 1976). Non-fibre carbohydrate (NFC) was calculated as follows:

$$NFC = 1000 - ash - CP - EE - NDF \quad [\text{g kg}^{-1} \text{ DM}]$$

Based on the concentrations of C, H and N ( $\text{g kg}^{-1} \text{ DM}$ ), the higher heating value (HHV) was calculated using the following empirical equation developed by Friedl et al. (2005) for biofuels:

$$HHV = 0.0355 * C^2 - 23.2 * C - 223 * H + 0.512 * C * H + 13.1 * N + 20600 \quad [\text{kJ kg}^{-1} \text{ DM}]$$

The concentration of any chemical compound (represented by Z;  $\text{g kg}^{-1} \text{ DM}$ ) in the PF was calculated from the concentration of Z in the silage (SIL) and the PC and the DM concentration of the PC, the PF and the silage after hydrothermal conditioning (SILC) according to:

$$Z_{PF} = \frac{DM_{SILC} * Z_{SIL} - Y * DM_{PC} * Z_{PC}}{X * DM_{PF}}$$

where X and Y are the quantities of the PF and the PC as a proportion of the silage after hydrothermal conditioning, respectively, which were calculated by:

$$X = \frac{DM_{PC} - DM_{SILC}}{DM_{PC} - DM_{PF}} \quad Y = 1 - X$$

Volatile solids (VS) equates to the dry organic matter, which is DM without the ash.

### 7.2.3 Anaerobic digestion experiments

Determination of biogas production of the silage in whole-crop anaerobic digestion (WCD) and the PF was performed in batch experiments in accordance with the German Standard (VDI 4630, 2004), based on a method described by Richter et al. (2009b) with two replicates. Fermentation of the substrates took place in gas-proof 20 litre polyethylene containers. Mixing of the fermenter content was carried out for 15 minutes every three hours. The experiments were performed in a mesophile temperature range of 37°C, with a fluctuation of  $\pm 1^\circ\text{C}$ . Digested slurry from a biogas plant was used as inoculum (15 kg fresh matter, FM) and either 500 g FM of silage or 1 kg FM of PF was used as feedstock. The fermentation time was 35 days for the silage and 15 days for the PF.

Measurement of gas fluxes started 24 hours after incubation and was repeated once a day. The total daily biogas volume was determined with a wet drum gas meter (TG1, Ritter Ltd., Germany). The biogas composition (the percentages of CH<sub>4</sub>, CO<sub>2</sub> and O<sub>2</sub>) was measured by infrared detection with a landfill gas analyser (LFG 20, Bernt Ltd., Germany). Methane volumes (litre) were measured under laboratory room conditions, converted to standard conditions (273.15 K, 101.325 kPa) and expressed as normal litre (L<sub>N</sub>) or normal cubic meter (m<sup>3</sup><sub>N</sub>). These methane volumes were referred to as methane yields when they were related both to the amount of volatile solids (VS) in the substrate (L<sub>N</sub> kg<sup>-1</sup> VS) and to the area harvested (m<sup>3</sup><sub>N</sub> ha<sup>-1</sup>).

### 7.2.4 Energy balance

Energy balances were calculated for conversion of the silage on eight different sampling dates through WCD and through IFBB, with six different treatments of hydrothermal conditioning. The energy balance was calculated both with an area-specific functional unit (1 ha) and a biomass-specific functional unit (1 kg DM of the herbage harvested). The system boundaries included the cultivation, harvest and transport of the grassland biomass, the conversion of the biomass into useful energy and the return of the residual material (digestate, coarse ash) to

agricultural land. It also included indirect energy input for provision of the technical infrastructure (agricultural machinery, energy conversion plants, buildings). The cultivation and harvest of the biomass comprised grass harrowing, mowing, swath, recovering the biomass with a field-chopper, transporting and ensiling the biomass in a bunker silo once per year. No ploughing or re-seeding was assumed, as the grassland is a permanent grassland under extensive management. The distance from the farm to the field was assumed to be 4 km and from field to field 2 km. DM losses due to microbial activities during the ensiling process were estimated to be 0.12 (KTBL, 2006).

Net energy yield was calculated as the difference between energy input and energy output. Energy input parameters used for the calculations are shown in Table 7.1. As the design layout of the conditioning unit, press, digesters and other technical components and infrastructures in a commercial IFBB plant is yet not known, implementation of exact figures for the indirect energy input is not possible at this stage. Several studies have shown that the indirect energy input for bioenergy plants is less than 0.1 of the direct energy input (GEMIS, 2009; Lootsma, 2006; Bachmaier et al., 2008). Therefore 0.1 of the direct energy input, divided evenly over electrical and heat energy, was assumed in this study. The heat energy demand ( $Q$ ) for hydrothermal conditioning of a certain quantity of silage ( $m = 1 \text{ g}$ ) was calculated based on the specific heat capacity of the silage ( $c = 3.6 \text{ J } ^\circ\text{C}^{-1} \text{ g}^{-1}$ ) and the change in temperature ( $\Delta T$ ) between the silage in the bunker silo (mean temperature of  $10^\circ\text{C}$  throughout the year) and during hydrothermal conditioning according to:

$$\frac{Q}{m} = c * \Delta T$$

In a commercial IFBB plant the water used as conditioning liquid will be captured after conditioning in an insulated sedimentation tank, recycled and used again as conditioning liquid under the assumption that the loss in temperature is no more than  $2^\circ\text{C}$ , so that an additional energy demand for heating the water ( $c = 4.1826 \text{ J } ^\circ\text{C}^{-1} \text{ g}^{-1}$ ) might be necessary. Additional losses as heat transmission of 0.1 of the calculated heat demand were assumed and the heat required by the fermenter was calculated accordingly. The fermentation temperature was assumed to be  $37^\circ\text{C}$ . In the IFBB process the fermenter for the digestion of press fluids needs only to be heated for the treatments without hydrothermal conditioning and with hydrothermal conditioning at  $10^\circ\text{C}$  and  $30^\circ\text{C}$ , as the continuous inflow of warm press fluid in the  $50^\circ\text{C}$ ,  $70^\circ\text{C}$  and  $90^\circ\text{C}$  treatments provides enough heat for fermentation in the mesophilic

temperature range. If necessary, the press fluids can be cooled down in a full-scale IFBB plant, without additional energy input by heat exchangers.

**Table 7.1** Energy input parameters for LCA calculation of the anaerobic digestion of whole-crop silage (WCD) and the IFBB procedure.

Energy input parameters	WCD	IFBB	Source
Diesel:			
Cultivation, harvest, transport of biomass	49.02 kWh t <sup>-1</sup> FM herbage	46.79 kWh t <sup>-1</sup> FM herbage	KTBL (2006)
Transport of pellets (30 km)	---	6.45 kWh t <sup>-1</sup> FM pellets	Bühle (2008)
Electricity:			
Screw press	---	14 kWh t <sup>-1</sup> FM silage	Bühle (2008)
Biogas plant operation	15.16 kWh t <sup>-1</sup> FM silage	0.45 kWh t <sup>-1</sup> FM press fluid	Bühle (2008)
Drying blower for press cakes	---	6.94 kWh t <sup>-1</sup> FM	Bühle (2008)
Pelleting of press cakes	---	113 kWh t <sup>-1</sup> FM	Sokhansanj and Fenton (2006)
Heat:			
Hydrothermal conditioning, 30°C	---	25 kWh t <sup>-1</sup> FM silage	Own Calculations
Hydrothermal conditioning, 50°C	---	47 kWh t <sup>-1</sup> FM silage	Own Calculations
Hydrothermal conditioning, 70°C	---	69 kWh t <sup>-1</sup> FM silage	Own Calculations
Hydrothermal conditioning, 90°C	---	91 kWh t <sup>-1</sup> FM silage	Own Calculations
Heating of fermenter	27 kWh t <sup>-1</sup> FM silage	8-31 kWh t <sup>-1</sup> FM press fluid	Own Calculations
Drying of press cakes <sup>†</sup>	---	0.45-0.63 kWh kg <sup>-1</sup>	Lootsma and Raussen (2008)

<sup>†</sup>DM concentration of fresh press cake after mechanical dehydration: 366-500 g kg<sup>-1</sup> FM, target DM concentration of dry press cake: 850 g kg<sup>-1</sup> FM

Calculation of the energy output implies the combustion of the biogas from the press fluid in a CHP with the generation of heat and electricity. Methane yields used for the calculation were based on the batch experiments. Electrical efficiency of the CHP was estimated to be 0.37, while thermal efficiency was estimated to be 0.48, based on manufacturer information for biogas CHPs (Scholwin et al., 2006). Thermal energy from the combustion of biogas in the

IFBB procedure is completely used as a heat source for drying the press cake. The degree of utilization of the thermal energy from the combined heat and power plant (CHP) in WCD was 0.2, which is a reference value for commercial biogas plants in Germany (FNR, 2009). The third output parameter is the amount of energy produced as heat from the combustion of the press cakes, which was calculated based on the HHV with a thermal efficiency during combustion of 0.9. Energy output from PF (electricity plus heat) and PC (heat) were calculated as proportions of gross energy of the herbage at harvest and were used for calculation of the energy balance. The energy conversion efficiency was determined as the ratio of the net energy yield and the gross energy yield of the herbage at harvest, which is the HHV of the herbage ( $\text{MJ kg}^{-1} \text{DM}$ ).

### 7.2.5 Statistical analysis

Analysis of variance (ANOVA) was performed to test the effect of hydrothermal conditioning before mechanical dehydration on the concentrations of organic fractions in the PF. This was done by the comparison of two treatments, mechanical dehydration without conditioning and mechanical dehydration after conditioning at  $10^{\circ}\text{C}$ , which was also the ambient temperature at which the mechanical dehydration was conducted. As there was only one observation for each combination of treatment and sampling date, the sampling dates were allocated to four periods with two consecutive samplings each, allowing an ANOVA with the two samplings used as replications. The procedure MIXED in SAS (SAS Institute, 1996) was used with treatment (T), sampling period (SP) and T x SP as fixed effects and SP as repeated subject. Due to the lack of replications, the ANOVA results are only tentative and should be interpreted cautiously.

Multiple regression analysis was performed to test the effect of chemical constituents in the parent silage on concentrations of organic compounds in the PF as well as on energy output from PF and PC as a proportion of gross energy and energy conversion efficiency, using the procedure GLM in SAS (SAS Institute, 1996). As the sampling date is a year-specific variable without much biological information and due to some insight from previous calculations (Richter et al., 2010b), the concentration of NDF was included in the starting model for describing the maturity of grassland swards, as well as the DM concentration of the silage and the conditioning temperature. Pearson's correlation test between NDF and DM in the silage showed a low correlation coefficient ( $r = 0.37$ ), so both variables were used in the analysis

(Richter et al., 2010b). Further information on the selection of terms for inclusion in the model was given by Richter et al. (2010b).

## 7.3 Results and Discussion

### 7.3.1 Organic compounds in the silage, in press cakes and press fluids

A continuous decrease of concentration (amount of decrease in  $\text{g kg}^{-1}$  DM) in the silage with progressive sampling date was measured for CP (87.5), EE (19.7) and NFC (125.9) (Table 7.2). In contrast, NDF and ADF increased with progressive sampling date by 237.0 and 135.3  $\text{g kg}^{-1}$  DM, respectively. ADL showed no directional variation over time.

**Table 7.2** Concentration of organic compounds ( $\text{g kg}^{-1}$  DM) of the silage, the press cake (PC) and press fluid (PF) with (+hc) and without (-hc) prior hydrothermal conditioning at eight different sampling dates.

		27/04	02/05	09/05	16/05	24/05	31/05	11/06	21/06
Crude protein (CP)	Silage	165.3	139.6	118.7	108.3	93.7	81.2	81.5	77.8
	PC <sub>-hc</sub>	124.1	108.1	83.9	73.2	75.0	60.6	68.4	58.6
	PC <sub>+hc</sub> †	95.7±5.2	79.9±1.7	69.6±0.5	62.5±0.7	59.1±0.9	47.9±1.2	56.5±0.6	48.8±0.6
	PF <sub>-hc</sub>	321.8	274.7	232.1	227.0	218.1	210.3	219.7	180.1
	PF <sub>+hc</sub> †	245.4±2.1	214.4±2.6	153.3±1.5	147.5±1.8	154.2±1.9	133.1±1.6	143.5±2.1	126.2±1.4
Ether extract (EE)	Silage	41.2	37.4	40.6	33.0	30.5	26.7	29.0	21.5
	PC <sub>-hc</sub>	49.6	43.4	45.2	35.2	29.9	25.7	26.5	19.8
	PC <sub>+hc</sub>	53.3±0.5	47.8±0.5	46.0±0.3	35.1±1.6	28.5±0.7	25.1±0.4	26.9±0.4	18.2±0.2
	PF <sub>-hc</sub>	9.4	11.6	25.6	25.6	34.5	33.0	55.4	30.6
	PF <sub>+hc</sub>	27.2±0.4	24.3±0.7	36.8±0.2	31.0±1.2	34.1±1.2	29.2±0.6	34.5±1.2	27.0±0.4
Neutral detergent fibre (NDF)	Silage	402.7	436.0	481.3	517.0	567.5	601.3	618.3	639.7
	PC <sub>-hc</sub>	457.0	503.7	574.3	615.3	628.0	667.0	662.5	696.5
	PC <sub>+hc</sub>	559.7±15	624.0±13	686.5±8.8	709.3±6.2	708.7±8.4	754.8±3.6	737.9±3.3	775.2±2.2
	PF <sub>-hc</sub>	196.9	145.1	177.5	185.1	164.8	190.6	152.5	336.6
	PF <sub>+hc</sub>	223.2±9.0	201.9±8.7	337.2±3.2	353.2±6.8	321.2±12	361.7±6.7	321.0±12	414.0±4.4
Acid detergent fibre (ADF)	Silage	326.7	332.0	373.7	376.3	421.0	434.0	443.7	462.0
	PC <sub>-hc</sub>	374.0	382.7	438.7	439.0	462.7	478.5	475.0	498.0
	PC <sub>+hc</sub>	429.3±9.2	446.5±6.4	473.5±3.5	478.6±5.5	497.8±4.8	518.7±3.3	510.3±2.6	533.0±1.6
	PF <sub>-hc</sub>	147.4	114.1	161.3	164.6	143.5	155.8	113.9	269.9
	PF <sub>+hc</sub>	209.1±4.4	189.1±4.7	303.6±1.7	289.6±2.6	287.4±5.1	302.2±2.4	278.7±5.1	343.7±2.7
Acid detergent lignin (ADL)	Silage	51.2	47.7	44.3	43.2	53.2	50.9	52.8	56.7
	PC <sub>-hc</sub>	48.0	44.5	46.4	39.9	44.6	47.1	49.3	54.1
	PC <sub>+hc</sub>	35.8±2.7	32.2±1.9	31.8±2.2	36.1±2.4	41.3±1.7	40.8±2.4	46.0±1.5	46.9±1.4
	PF <sub>-hc</sub>	63.3	61.5	37.4	54.3	110.4	74.7	89.7	70.6
	PF <sub>+hc</sub>	68.5±2.3	66.9±1.7	52.8±1.1	49.4±2.0	73.9±2.6	66.1±3.2	69.2±3.1	72.8±1.8
Non-fibre carbohydrates (NFC)	Silage	301.5	304.5	264.0	240.5	223.4	204.9	194.8	175.6
	PC <sub>-hc</sub>	286.5	263.4	210.6	180.5	189.2	167.6	170.7	142.4
	PC <sub>+hc</sub>	232.2±8.1	194.9±11	139.0±8.9	126.6±6.0	148.2±7.0	119.5±2.3	124.8±2.9	96.9±2.7
	PF <sub>-hc</sub>	358.5	480.8	438.3	443.2	451.1	437.9	448.5	352.5
	PF <sub>+hc</sub>	380.5±5.7	440.0±8.3	351.3±3.0	337.5±5.7	354.1±9.6	338.2±3.5	368.6±8.2	306.3±0.8

† Mean value ± s.e. of mean of five PC/PF after hydrothermal conditioning at 10, 30, 50, 70, and 90°C



The increasing maturity of the grassland sward follows a typical pattern for grasslands, with a decline in CP and an increase in fibre (Collins and Casler, 1990; Čop et al., 2009). The high ADL concentration at the early growth stages is possibly connected with lignified plant tissue remaining from the winter period, which is gradually overgrown by new young plant tissue with advancing spring growth. From mid-May onwards, however, the new plant tissue also undergoes the process of lignification and the ADL concentration increases. Similar dynamics of ADL levels in forage crops were reported by Mupangwa et al. (2006), who found a decline in ADL in three forage legumes between 8 and 14 weeks after germination and an increase afterwards with increasing maturity.

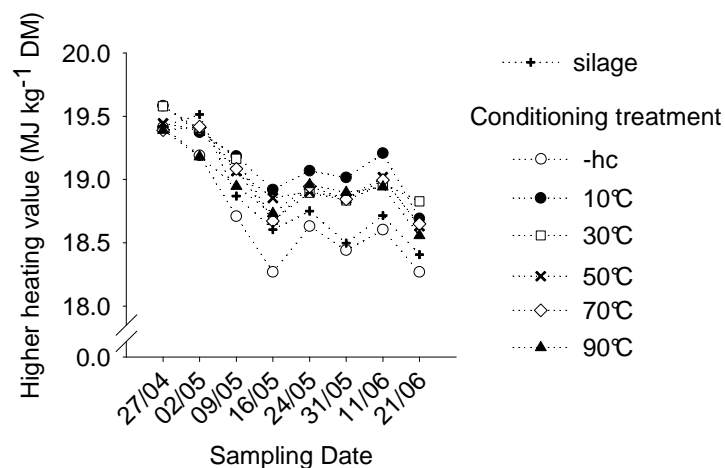
The concentrations of organic compounds in the PC obtained through mechanical dehydration without prior hydrothermal conditioning (PC<sub>-hc</sub>) and with hydrothermal conditioning (PC<sub>+hc</sub>) showed the same pattern over time, with a decrease for CP, EE and NFC and an increase for NDF and ADF. CP levels of PC<sub>+hc</sub> (48.8-95.7 g kg<sup>-1</sup> DM) were lower compared to the silage and to PC<sub>-hc</sub> (58.6-124.1 g kg<sup>-1</sup> DM), while EE and ADL in the PC were in the same magnitude as in the silage. NFC concentrations were also lowest in PC<sub>+hc</sub> (96.9-232.2 g kg<sup>-1</sup> DM) and somewhat higher in PC<sub>-hc</sub> (142.4-286.5 g kg<sup>-1</sup> DM). PC<sub>+hc</sub> achieved higher concentrations of NDF (559.7-775.2 g kg<sup>-1</sup> DM) and ADF (429.3-533.0 g kg<sup>-1</sup> DM) compared to PC<sub>-hc</sub> (457.0-696.5 and 374.0-498.0 g kg<sup>-1</sup> DM for NDF and ADF, respectively) and compared to the silage.

In the PF, concentrations of CP (180.1-321.8 and 126.2-245.4 g kg<sup>-1</sup> DM in PF<sub>-hc</sub> and PF<sub>+hc</sub>, respectively) and NFC (352.5-480.8 and 306.3-440.0 g kg<sup>-1</sup> DM in PF<sub>-hc</sub> and PF<sub>+hc</sub>, respectively) were higher compared to the silage. NDF (145.1-336.6 and 201.9-414.0 g kg<sup>-1</sup> DM in PF<sub>-hc</sub> and PF<sub>+hc</sub>, respectively) and ADF (113.9-269.9 and 189.1-343.7 g kg<sup>-1</sup> DM in PF<sub>-hc</sub> and PF<sub>+hc</sub>, respectively) were lower compared to the silage. EE levels were lower in the first half, but higher in the second half of the sampling period, at 9.4-55.4 g kg<sup>-1</sup> DM for PF<sub>-hc</sub> and 24.3-36.8 g kg<sup>-1</sup> DM for PF<sub>+hc</sub>. ADL concentrations of the PF were higher compared to the silage, apart from PF<sub>-hc</sub> at the third sampling date and achieved values between 37.4 and 110.4 g kg<sup>-1</sup> DM for PF<sub>-hc</sub> and between 49.4 and 73.9 g kg<sup>-1</sup> DM for PF<sub>+hc</sub>.

Fibre concentrations (NDF and ADF) were lower in all PF compared to the silage, but ADL concentrations were higher. Richter et al. (2009b) found a proportional reduction of crude fibre levels in IFBB-PF of 0.34-0.42 compared to the parent grassland silage, which is in the same range as for the PF<sub>+hc</sub> regarding ADF or NDF. However, the authors are not aware of any studies in which ADL was investigated in PF.

### 7.3.2 Energy in the press cake and the press fluid

The HHV decreased from the first to the fourth sampling date both in the silage and the PC and levelled off until the eighth sampling date (Figure 7.1). The PC and the silage ranged in a comparable magnitude, with highest HHV at the first sampling date (19.39 - 19.58 MJ kg<sup>-1</sup> DM) and lowest at the eighth sampling date (18.27 to 18.83 MJ kg<sup>-1</sup> DM).



**Figure 7.1** Higher heating value of the herbage and of press cakes generated without hydrothermal conditioning (-hc) and with hydrothermal conditioning at five different temperatures (10, 30, 50, 70, 90°C) at eight consecutive sampling dates.

The HHV was higher for the PC with hydrothermal conditioning (18.56 to 19.21 MJ kg<sup>-1</sup> from mid-May until the end of June) compared to the PC without hydrothermal conditioning. The HHV of other common biomass fuels range between 18.5 (wheat straw), 19.1 (Miscanthus), 19.8 (poplar from short rotation coppice) and 20.2 (spruce wood) MJ kg<sup>-1</sup> DM (Hartmann, 2001). With regard to their energy properties, IFBB PC from grassland was between straw and wood from short rotation coppice. In earlier studies with PC from semi-natural grasslands with hydrothermal conditioning at 60°C, an HHV of 18.4 MJ kg<sup>-1</sup> DM was determined (Richter et al., 2010a).

The ANOVA showed significant treatment (T) effects for concentrations of all compounds in the PF, apart from EE and ADL and significant sampling period (SP) effects for the concentrations of VS, ash, CP and EE (Table 7.3). Significant T x SP interactions indicate variable conditioning effects for VS and EE depending on the sampling date. For ash and NFC, the repeated statement was removed from the model as the null model likelihood ratio test was not significant.

**Table 7.3** Levels of significance of ANOVA for the effects of treatment (T), sampling period (SP) and T x SP, and for the estimate of the repeated measurement effect (LRT) on concentrations of volatile solids (VS, g kg<sup>-1</sup> FM), ash and various organic compounds (g kg<sup>-1</sup> DM) in the press fluid with (10°C) and without (-hc) prior hydrothermal conditioning of grassland silage.

Variable	Treatment†		Level of significance (P-value)			
	-hc	10°C	LRT‡	Treatment (T)	Sampling period (SP)	T x SP
VS	87.9 ± 11.3	20.6 ± 0.9	***	**	***	***
ash	116.4 ± 5.4	130.7 ± 2.9	ns	*	*	ns
CP	235.5 ± 9.4	163.6 ± 15.0	**	*	**	ns
EE	28.2 ± 4.4	30.9 ± 1.5	**	ns	**	*
NDF	193.6 ± 22.9	327.6 ± 23.0	*	*	ns	ns
ADF	158.8 ± 18.9	282.5 ± 17.1	**	*	ns	ns
ADL	70.2 ± 7.9	58.6 ± 2.9	**	ns	ns	ns
NFC	426.3.0 ± 16.1	347.2 ± 12.0	ns	**	ns	ns

†Values represent least square means (± s.e. of mean) per treatment

‡LRT, null model likelihood ratio test; the null model, which excludes the specified covariance structure of repeated measurements, was used if not significant ( $P > 0.05$ )

ns, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$

The multiple regression models including the NDF concentration of the silage, the temperature of hydrothermal conditioning and the DM concentration of the silage, as well as their quadratic terms and twofold interactions explained the concentrations of organic compounds in the PF with high accuracy (Table 7.4). Coefficients of determination ranged between  $R^2 = 0.76$  for EE and  $R^2 = 0.99$  for CP. The model for the ash concentration included only linear effects of the coefficients NDF and T, without any interaction and had a comparably low  $R^2$  of 0.58 (data not shown).

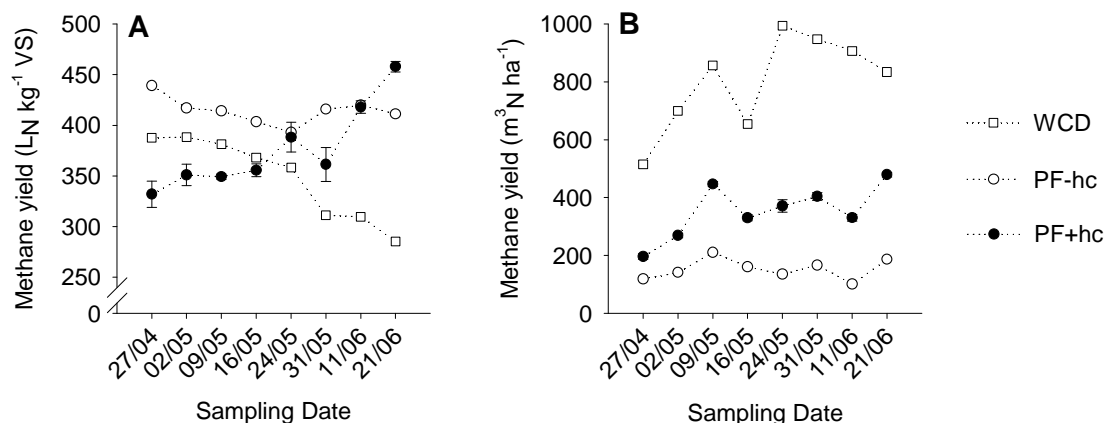
Specific methane yields of WCD decreased constantly from 388 to 285 L<sub>N</sub> kg<sup>-1</sup> VS (Figure 7.2A). In contrast, the specific methane yields of the PF with hydrothermal conditioning (PF<sub>+hc</sub>) achieved the lowest yield at the first sampling (332 L<sub>N</sub> kg<sup>-1</sup> VS) and the highest yield at the eighth sampling (458 L<sub>N</sub> kg<sup>-1</sup> VS). The PF without hydrothermal conditioning (PF<sub>-hc</sub>) achieved constantly high specific methane yields between 393 and 439 L<sub>N</sub> kg<sup>-1</sup> VS. The area-related methane yield was highest for WCD, with a strong increase from the first (515 m<sup>3</sup><sub>N</sub> ha<sup>-1</sup>) to the fifth sampling date (994 m<sup>3</sup><sub>N</sub> ha<sup>-1</sup>) and a decline afterwards (Figure 7.2B). Area-related methane yields of PF<sub>+hc</sub> were lower than those of silage, but higher than those of PF<sub>-hc</sub> and ranged between 196 m<sup>3</sup><sub>N</sub> ha<sup>-1</sup> at the first sampling date and 479 m<sup>3</sup><sub>N</sub> ha<sup>-1</sup> at the last sampling date.

**Table 7.4** Coefficient of determination and parameter estimates for the models of concentration of plant compounds in the press fluid.

	VS	CP	EE	NDF	ADF	ADL	NFC
R <sup>2</sup>	0.96***	0.99***	0.76***	0.94***	0.94***	0.77***	0.81***
F value	138.57	536.66	27.89	105.31	176.94	22.19	36.80
Estimates:							
Intercept	-44.1226***	838.852***	75.0626*	132.868**	357.144***	89.1960ns	-249.260ns
NDF†	0.17868***	-2.55074***	0.07523ns	0.69224***	0.08333ns	-0.43897*	1.75479**
NDF <sup>2</sup>	-0.00014***	0.00199***	-0.00028***	ns	ns	0.00062**	ns
T‡	-0.00155ns	0.27984*	ns	-2.52177**	ns	0.12111***	ns
T <sup>2</sup>	0.00041**	-0.00330**	ns	0.00987*	ns	ns	ns
DM¶	0.14179***	0.58602***	-0.45842***	-0.60999***	-1.43522***	0.46605**	1.32474**
DM <sup>2</sup>	ns	-0.0072***	ns	ns	ns	ns	0.00610***
NDF x T	ns	ns	ns	0.00292*	ns	ns	ns
NDF x DM	-0.00019***	ns	-0.00083***	ns	0.00182**	-0.00071**	-0.00794***

†NDF in the silage in g kg<sup>-1</sup> DM

‡T in °C

¶DM in the silage in g kg<sup>-1</sup> FMns, not significant; \*,  $P < 0.05$ ; \*\*,  $P < 0.01$ ; \*\*\*,  $P < 0.001$ **Figure 7.2** (A) Specific methane yield and (B) area-related methane yield of the grassland silage in WCD and of press fluids generated without (PF<sub>-hc</sub>) and with (PF<sub>+hc</sub>, mean value with s.e of mean) hydrothermal conditioning at eight consecutive sampling dates.

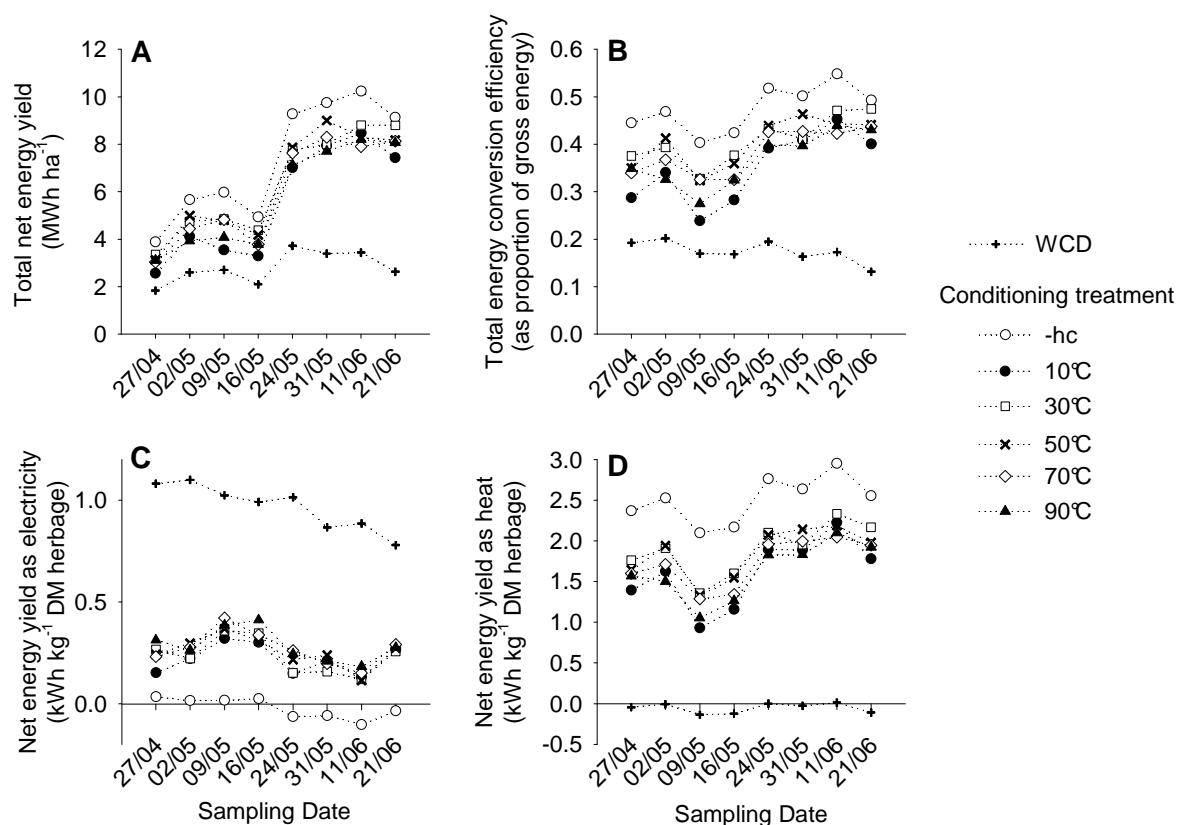
The ANOVA showed that hydrothermal conditioning resulted in significantly ( $P < 0.05$ ) higher NDF and ADF concentrations and significantly lower NFC concentrations in PF<sub>+hc</sub> compared to the treatment without conditioning. It is well known that methane yield in anaerobic digestion is primarily dependent on organic compounds, and that easily soluble fractions (protein, fat, non-fibre carbohydrates) achieve higher specific methane yields than

fibre fractions (Amon et al., 2007b). Hence, it was expected that methane yields would be highest for PF<sub>-hc</sub>, followed by PF<sub>+hc</sub> and by the silage. However, methane yields of PF<sub>+hc</sub> were lower at the first three sampling dates (332-351 L<sub>N</sub> kg<sup>-1</sup> VS) compared to the silage, but increased strongly up to 458 L<sub>N</sub> kg<sup>-1</sup> VS at the last sampling date. This development could not be predicted from the organic composition of the PF<sub>+hc</sub>, where NDF in the PF was the only significantly influencing parameter in a regression analysis (Methane yield = 270.91 + 0.3399\*NDF, R<sup>2</sup>=0.23, P < 0.01). However, the methane yield of PF<sub>-hc</sub> proved to be highly correlated with the fibre fractions, with a positive influence of NDF and a negative influence of ADF and ADL (methane yield = 491.91 + 1.5452\*NDF - 1.9367\*ADF - 1.0757\*ADL, R<sup>2</sup>=0.92, P < 0.05). It is well known that the hemicellulose in NDF is digestible by anaerobic microorganisms and contributes to methane production, while ADF and ADL comprise lignocellulose compounds, which are resistant to anaerobic digestion (Kirchgeßner, 2004). However, fibre fractions were not able to predict methane yields of PF in general and the influencing factors on methane production in PF remain somewhat unclear.

### 7.3.3 Energy balance

The total area-related net energy yield of the IFBB procedure increased over time and was highest for the treatment without hydrothermal conditioning (-hc), with a maximum of 10.23 MWh ha<sup>-1</sup> at the seventh sampling date (Figure 7.3A). The highest total net energy yields among the IFBB treatments with hydrothermal conditioning were achieved by the 30°C treatment (maximum of 8.81 MWh ha<sup>-1</sup> at the seventh sampling date) and by the 50°C treatment (maximum of 9.00 MWh ha<sup>-1</sup> at the sixth sampling date). Over the whole sampling season, the 10°C treatment performed worst among the IFBB treatments. WCD achieved lower total net energy yields than IFBB, with a maximum of 3.72 MWh ha<sup>-1</sup> at the fifth sampling date, and showed only a minor increase over time.

Total energy conversion efficiencies of all IFBB treatments showed a general increase over time and were between 0.38 and 0.54 for -hc and between 0.32 and 0.46 for the 50°C treatment (Figure 7.3B). The energy conversion efficiency of WCD decreased over time and was between 0.13 and 0.20. Energy output as a proportion of gross energy of the IFBB treatments with hydrothermal conditioning ranged from 0.14 to 0.27 for PF and from 0.31 to 0.59 for PC (data not shown).



**Figure 7.3** (A) Total net energy yield, (B) total energy conversion efficiency, (C) net electric energy yield and (D) net thermal energy yield of the silage in WCD and of the IFBB system without hydrothermal conditioning (-hc) and with hydrothermal conditioning at different temperatures (10, 30, 50, 70 and 90°C) at eight consecutive sampling dates.

The area-related net energy yield of WCD was determined by the DM yield of the herbage and the specific methane yield. While DM yield continuously increased (Richter et al., 2010b), specific methane yield continuously declined. These two effects added up to a drop in the net energy yield at the fourth sampling date, which otherwise increased until the fifth sampling date, due to increasing DM yields, and levelled off afterwards, due to a balancing of increasing DM yields and declining methane yields. Hence, the conversion efficiency of WCD slightly declined over time as a result of a steadily increasing gross energy yield. Richter et al. (2010a) calculated an average net energy yield of 2.35 MWh ha<sup>-1</sup> for the anaerobic digestion of five semi-natural grassland silages harvested in July and August, which is in a comparable range to the values of this study. Comparable studies in the literature are rare. In some studies, the energy calculations are based on high-input grasslands with gross energy yields of 21-34 MWh ha<sup>-1</sup> (Rösch et al., 2009; Smyth et al., 2009), compared to 9-19 MWh ha<sup>-1</sup> in this study obtained on an extensively managed grassland. Other studies were based on different conversion techniques, such as gasification, ethanol production or combustion (Tilman et al., 2006; Prochnow et al., 2009b).

The herbage-related total net energy yield ( $\text{kWh kg}^{-1}$  DM herbage) showed the same curve shapes as the total energy conversion efficiency and was therefore not shown, but separated into its two components net electric and thermal energy yield. The net electric energy yield was highest for WCD, but decreased over time from a maximum of  $1.10 \text{ kWh kg}^{-1}$  DM herbage at the second sampling date to  $0.78 \text{ kWh kg}^{-1}$  DM herbage at the eighth sampling date (Figure 7.3C). All IFBB treatments with hydrothermal conditioning were of a comparable magnitude over the whole sampling period from  $0.11$  ( $50^\circ\text{C}$ , seventh sampling date) to  $0.42 \text{ kWh kg}^{-1}$  DM herbage ( $70^\circ\text{C}$ , third sampling date).

The -hc treatment achieved a positive net electric energy yield only at the first four sampling dates, while there was a net electric energy demand at the fifth to eighth sampling date. The net thermal energy yield of the IFBB treatments was much higher than the net electric energy yield and accounted for the major part of the total net energy yield, which is why it showed a comparable pattern over time (Figure 7.3D). The -hc treatment achieved the highest net thermal energy yield, which ranged from  $2.10$  to  $2.95 \text{ kWh kg}^{-1}$  DM herbage. The maximum net thermal energy yield of the IFBB treatments with hydrothermal conditioning was  $2.33 \text{ kWh kg}^{-1}$  DM herbage ( $30^\circ\text{C}$ , seventh sampling date), the minimum was  $0.93 \text{ kWh kg}^{-1}$  DM herbage ( $10^\circ\text{C}$ , third sampling date). WCD had slightly negative net thermal energy yields, and thus thermal energy demands, except for the fifth and the seventh sampling date.

Net energy yields of the IFBB treatments were on a higher level compared to WCD, due to a combined production of biogas and solid fuel. The increase in DM yield in the second half of May as well as an increase in herbage-related solid fuel production, which accounts for about  $0.87$  of the total energy production, resulted in a sharp rise in the area-related net energy yield of the IFBB treatments up to a maximum of  $8\text{-}10 \text{ MWh ha}^{-1}$  at 11 June. The conversion of five different semi-natural grasslands with the IFBB technology resulted in slightly higher net energy yields of  $12\text{-}14 \text{ MWh ha}^{-1}$ , mainly due to higher DM yields (Richter et al., 2010a).

Conversion efficiencies from mid-May until the end of June ranged between  $0.39$  and  $0.54$ , which was slightly lower compared to the production of biomethane from intensive grassland for direct injection into the gas grid ( $0.56$ ) as reported by Smyth et al. (2009), but higher than conventional anaerobic digestion of whole-crop silage ( $0.10\text{-}0.15$ ; Faaij, 2006). However, it is noteworthy that in contrast to direct injection of biomethane into the gas grid, the energy balance of this study is based on effective energy (electricity and heat). While the IFBB treatments with hydrothermal conditioning produced sufficient amounts of both electricity and heat, net energy production of WCD was deficient in terms of heat, meaning that the heat

demand for heating the fermenter could not be accomplished. Conversely, in the case of the IFBB treatment without conditioning, there was a lack of electricity to support the technical facilities.

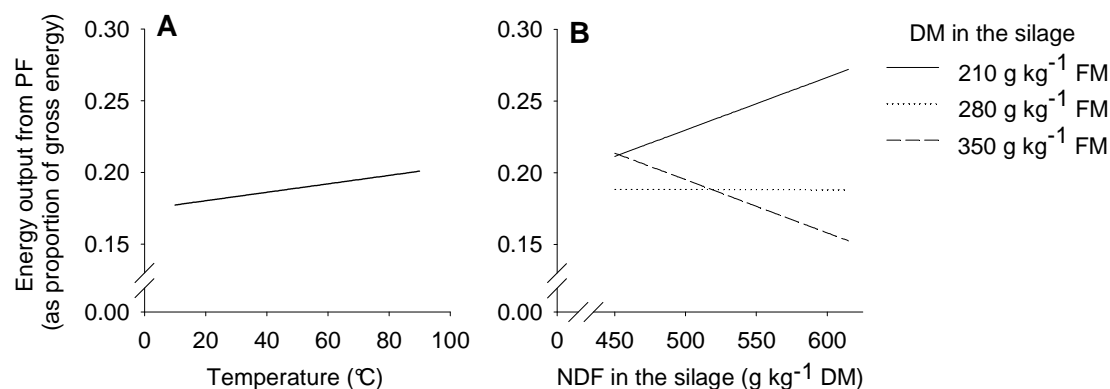
Variation of energy output from PF and PC as a proportion of gross energy as well as total energy conversion efficiency could be explained by multiple regression analysis, with high coefficients of determination of 0.90, 0.99 and 0.90, respectively (Table 7.5).

**Table 7.5** Coefficient of determination and parameter estimates for the models of energy output from PF and PC as well as energy conversion efficiency of the IFBB system.

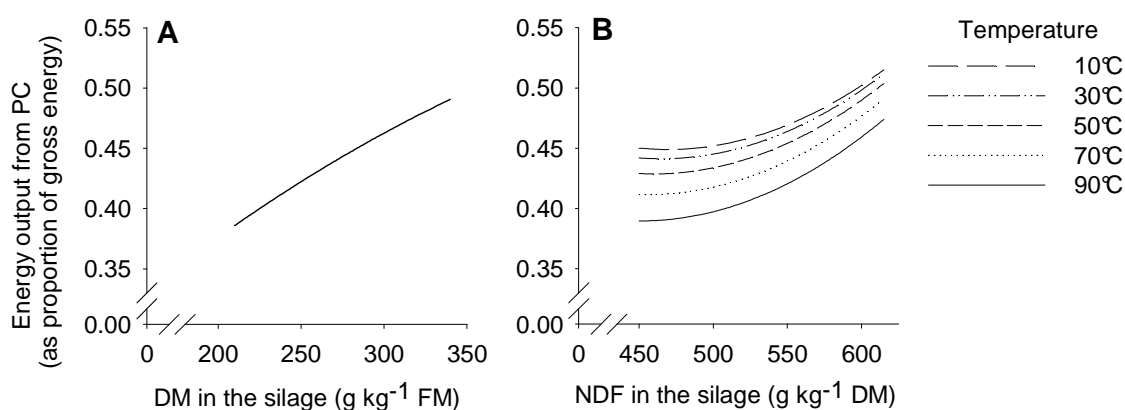
	Energy output from PF (as proportion of gross energy)	Energy output from PC (as proportion of gross energy)	Total energy conversion efficiency
R <sup>2</sup>	0.90***	0.99***	0.90***
F value	64.75	521.69	47.12
Coefficients:			
Intercept	-0.10843ns	0.82992***	-0.05938ns
NDF	0.00147***	-0.00293***	-0.00032ns
NDF <sup>2</sup>	ns	0.000003***	0.000002*
T	0.00029***	-0.00085*	0.00299***
T <sup>2</sup>	ns	-0.000006**	-0.00003***
DM	-0.00037ns	0.00144***	0.00211***
DM <sup>2</sup>	0.000005***	-0.000001***	ns
NDF x T	ns	0.000001**	ns
NDF x DM	-0.000005***	ns	-0.000003**

Energy output from PF showed a slight increase with increasing temperature of hydrothermal conditioning (Figure 7.4A). The significant NDF x DM interaction suggests that an increase in NDF resulted in an increase in energy output from PF at low DM levels in the silage and to a decline in energy output from PF at high DM levels in the silage (Figure 7.4B). Energy output from PC increased with increasing DM in the silage (Figure 7.5A) and increasing NDF in the silage (Figure 7.5B). The temperature had a negative influence on energy output from PC, which was less pronounced at high NDF levels in the silage (Figure 7.5B). Total energy conversion efficiency showed a maximum with a temperature of 50°C during hydrothermal conditioning (Figure 7.6A) and increased with increasing NDF in the silage (Figure 7.6B). Conversion efficiency was higher at high levels of DM, but DM effects decreased with increasing NDF concentrations.

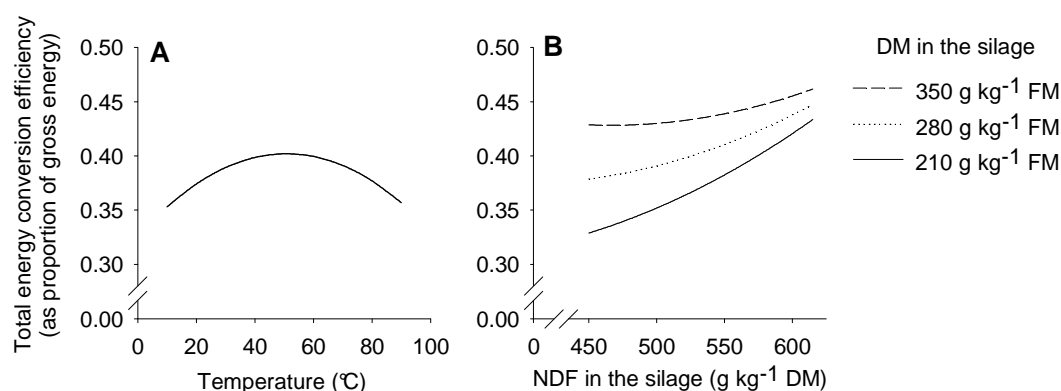




**Figure 7.4** Predictions of energy output from press fluid: (A) Temperature of hydrothermal conditioning ( $^{\circ}\text{C}$ ) at a mean DM concentration in the silage of  $280\text{ g kg}^{-1}\text{ FM}$  and a mean NDF concentration in the silage of  $533\text{ g kg}^{-1}\text{ DM}$ . (B) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}\text{ DM}$ ) with DM concentration in the silage ( $\text{g kg}^{-1}\text{ FM}$ ) at a mean temperature of  $50^{\circ}\text{C}$ .



**Figure 7.5** Predictions of energy output from press cake: (A) DM concentration in the silage ( $\text{g kg}^{-1}\text{ FM}$ ) at a mean temperature of hydrothermal conditioning of  $50^{\circ}\text{C}$  and a mean NDF concentration in the silage of  $533\text{ g kg}^{-1}\text{ DM}$ . (B) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}\text{ DM}$ ) with temperature of hydrothermal conditioning ( $^{\circ}\text{C}$ ) at a mean DM concentration in the silage of  $280\text{ g kg}^{-1}\text{ FM}$ .



**Figure 7.6** Predictions of total energy conversion efficiency of the IFBB system: (A) Temperature of hydrothermal conditioning ( $^{\circ}\text{C}$ ) at a mean DM concentration in the silage of  $280\text{ g kg}^{-1}\text{ FM}$  and a mean NDF concentration in the silage of  $533\text{ g kg}^{-1}\text{ DM}$ . (B) The interaction of NDF concentration in the silage ( $\text{g kg}^{-1}\text{ DM}$ ) with DM concentration in the silage ( $\text{g kg}^{-1}\text{ FM}$ ) at a mean temperature of  $50^{\circ}\text{C}$ .

Multiple regression analysis showed that the main parameters influencing energy output from PF and PC and energy conversion efficiency of the IFBB system were the temperature of hydrothermal conditioning as well as the NDF and DM concentrations of the silage, which also influenced mass flows of various plant compounds into the PF and concentrations of elements in the press cake (Richter et al., 2010b). Energy output from PF was positively correlated with temperature of hydrothermal conditioning, which is consistent with the finding of higher mass flows of DM into the PF at higher temperatures, as reported by Richter et al. (2010b), providing more digestible organic matter for methane production.

However, effects of NDF in the silage on the energy output from PF were complex: While NDF led to a decline in DM mass flow into the PF (Richter et al., 2010b), it increased specific methane yield at the same time. Obviously, the second effect was more important when DM mass flow into the PF was on a high level (i.e. at low DM concentration in the silage), resulting in an increasing energy output from PF, whereas at a lower level of DM mass flow into the PF (i.e. at high DM concentration in the silage), energy output from PF declined with increasing NDF in the silage. The energy output from PC was predicted to increase with increasing DM and NDF in the silage and decreasing temperature of hydrothermal conditioning, as less DM is transferred into the PF and a higher proportion of DM goes into the PC (Richter et al., 2010b).

The regression model suggests a maximum total energy conversion efficiency at a conditioning temperature of 50°C (Figure 7.6A). At lower temperatures, more DM is directed into the PC (Richter et al., 2010b), increasing the heat demand for drying it, which eventually leads to a reduction of total energy conversion efficiency, as the internal heat supply from biogas combustion for drying becomes deficient at the same time. By contrast, at temperatures above 50°C, much more energy is needed for heating the biomass during conditioning than is being produced from PF, as indicated by the minor slope in Figure 7.4A. As for PC, an increase in temperature even results in a decline in energy production (Figure 7.5B).

The positive effect of increasing DM concentration of the silage on energy conversion efficiency is more pronounced at low compared to high NDF concentrations in the silage. This is due to the ambivalent effect of increasing NDF concentration in the silage on the energy output of PF. However, as energy output from PC, which is up to 4 times as high as energy output from PF, generally increases with increasing NDF concentration in the silage,

the total energy output always increases, but less significantly at high NDF concentration in the silage, when energy output from PF decreases.

Richter et al. (2010b) found that the highest fuel quality, i.e. defined as the press cake with the lowest concentrations of elements detrimental for combustion in IFBB-PC, was obtained with a combination of high temperatures of hydrothermal conditioning, low levels of DM and high levels of NDF in the silage. Since temperatures above 50°C only showed minor effects on element concentration, which particularly applied at high levels of NDF in the silage (Richter et al. 2010b), this temperature may be an appropriate compromise, as it also provided the highest total conversion efficiency. Concerning DM in the silage, a compromise between solid fuel quality and total conversion efficiency may be possible at high NDF concentrations in the silage (late harvest dates), where the effect of DM was less pronounced and even treatments with low DM concentration in the silage achieved high conversion efficiencies.

Levels of NDF and DM in the silage as well as temperature during hydrothermal conditioning were strong predictors for most technical parameters of the IFBB process, which may provide a basis for the development of tools for process control in practice. However, the models presented in this study are based on a single experimental sward and need to be validated with others, in order to determine the impact of botanical sward composition.

## 7.4 Conclusions

1. Methane yield of PF after hydrothermal conditioning increased with increasing sward maturity. HHV of PC first decreased, but remained constant from mid-May onwards. Temperature effects were inconsistent for both parameters.
2. Energy conversion efficiency increased with increasing sward maturity up to 0.47 (IFBB with hydrothermal conditioning). It was higher than in the WCD reference system (0.20).
3. NDF and DM in the silage and temperature during hydrothermal conditioning were strong predictors for energy conversion efficiency ( $R^2=0.90$ ) of the IFBB process. Maximum conversion efficiency was predicted at a combination of medium temperature (50°C) and high levels of DM and NDF.

## 8 General discussion

This study has helped to improve the general understanding of the IFBB process and identify some crucial cause-and-effect-chains. Furthermore it has raised new questions, so that the most important issues regarding the conversion of biomass from semi-natural grasslands through the IFBB process can be outlined as follows:

- (i) The hydrothermal conditioning and subsequent mechanical dehydration, which are supposed to minimize the water content and the concentration of elements detrimental for combustion in the PC as well as to optimise the separation of DM into PF and PC.
- (ii) The net energy yield, the energy conversion efficiency and the GHG mitigation of the IFBB process, which are supposed to be maximised and which are affected by technical and biomass-related parameters.
- (iii) The predictability of mass and energy flows within the IFBB process from the maturity and the concentration of DM of the grassland sward at harvest time.

### 8.1 Hydrothermal conditioning and mechanical dehydration

Hydrothermal treatments have been widely investigated as methods for the solubilisation of hemicellulose, cellulose and lignin from woody biomass through hydrolysis reactions and conversion to biofuel (Bonn et al., 1983; Avellar and Glasser, 1998; Sasaki et al., 2003). Temperatures in this process are usually between 150°C and 230°C, since the kinetics of hydrolysis are low at temperatures below 100°C (Garotte et al., 1999). In the IFBB process, however, low temperature hydrothermal conditioning is primarily aimed at enhancing the separation of elements detrimental for combustion (K, Mg, Cl, N, S) from plant cells, in order to facilitate their transfer into the PF during the subsequent mechanical dehydration.

K, Mg and Cl occur predominantly as ions in solution of the phloem, in the cytoplasm or the vacuole, while to a minor extent Mg is bound in cell wall pectins of low solubility (Marschner, 1995). In contrast, N and S are mainly bound in amino acids and proteins with a low mobility, especially in case of structure proteins of cell walls (Marschner, 1995). At high levels of N nutrition, a large amount of N is stored as soluble nitrate in the vacuole of the plant, but since the concentration of nitrate-N is low in mature herbage (Weissbach et al., 1993), most of the N is bound in proteins. Due to these different occurrences of elements in

the plant cell, the mass flows after hydrothermal conditioning of K and Cl (>0.8) were higher than those of Mg (0.65-0.75), S (0.5-0.7) and N (0.4-0.6) (Figure 3.2 and Figure 6.3).

In previous experiments using the IFBB process as conversion technique for silage from cereals, a positive influence of higher temperatures during hydrothermal conditioning on mass flows was observed (Graß et al., 2009). In the first experiment of this study, significantly higher mass flows were observed for ash, K and Mg by increasing the temperature of hydrothermal conditioning from 5°C to 60°C (Figure 3.2). A further increase to 80°C did not result in significant changes. In the second experiment of this study, the influence of increasing temperatures from 10°C to 90°C, with three intermediate steps, was positive regarding the increase of mass flows, however, to a rather low extent (Figure 6.3).

The water content of the PC after mechanical dehydration is an important factor for the determination of the internal heat demand of the IFBB process. The lower the water content, i.e. the higher the DM concentration, the less heat is needed for thermal drying of the PC to a DM concentration of 850 g kg<sup>-1</sup> FM in order to make it suitable for pelleting and storage. The DM concentration of the PC is primarily dependent on the technical adjustment of the screw press, which was different in the two experiments of this study.

In the first experiment, with a pitch of the screw of 1:7.5 and a rotational speed of 12 revolutions min<sup>-1</sup>, DM concentrations of the PC after mechanical dehydration were between 464 and 543 g kg<sup>-1</sup> FM. In contrast, in the second experiment, mechanical dehydration with a screw press pitch of 1:6 and a rotational speed of 6 revolutions min<sup>-1</sup>, obtained PC with lower DM concentrations of 388 to 452 g kg<sup>-1</sup> FM. In a comparison of different pitches and rotational speeds of screw presses, highest DM concentrations were obtained with the highest pitch and low rotational speeds of <6 revolutions min<sup>-1</sup> (Seckler, 2008). However, if the rotational speed decreases, the capacity of the screw press decreases, which might be a problem in a commercial IFBB plant. Thus, for further optimisation, a screw with a pitch of 1:7.5 and a rotational speed of 6 revolutions min<sup>-1</sup> should be investigated.

In a green biorefinery project, where grass silage was separated with a screw press, DM concentrations of PC were about 500 g kg<sup>-1</sup> FM (Mandl et al., 2006). Apart from screw presses, there are other possible press techniques for the dehydration of biomass available. Turn et al. (1997) subjected banagrass (*Pennisetum purpureum*) to mechanical dehydration with a hydraulic plunger press and obtained DM concentrations in PC of 480 g kg<sup>-1</sup> FM in a single pressing and 550 g kg<sup>-1</sup> FM in a double pressing. In a mechanical dewatering of swine

manure with a belt press DM concentrations were increased from 8 to 177g kg<sup>-1</sup> FM (Xiu et al., 2009). Further research on different press technologies within the IFBB process would be recommended.

## **8.2 Net energy yield, energy conversion efficiency and GHG mitigation**

Producing energy from semi-natural grassland or other agriculturally abandoned land is of increasing interest as a sustainable attempt to generate bioenergy without food-fuel competition and has worldwide a large potential (Campbell, 2008). This potential, however, will only be exploited if the biomass can be converted with high net energy yields, high conversion efficiencies and high GHG mitigation potential. Zhou et al. (2007) showed that the energy output of LIHD grassland biomass on degraded soil in China is nearly equal to that of ethanol from conventional corn grain on fertile soil and far more economical. Nine out of sixteen sustainability indicators for the energy production from grassland biomass, identified by Rösch et al. (2009), were related to energy efficiency and emissions, highlighting the importance of these issues. Another four indicators were connected to biodiversity, soil and water protection and conservation of landscapes, which are also target objectives of the energy production from semi-natural grasslands with the IFBB process.

It has been shown for the conversion of semi-natural grassland, that IFBB is superior to conventional techniques, either in regard of energy and GHG balances (WCD, Figure 5.4 and Figure 5.5), or in regard of fuel quality (CH, Figure 5.2 and Figure 5.3). Area-related net energy yield and GHG mitigation of the IFBB process are mainly dependent on the DM yield of the biomass, since variations in mass flows between different grassland vegetations turned out to be of minor importance. In contrast, specific net energy yield and conversion efficiency are rather dependent on the conversion technology. There is, however, rarely one solution for the optimisation of all target parameters, so that different benefits have to be balanced (Botha and Von Blottnitz, 2006). Regarding the IFBB process, different technology optimisation steps could be considered.

For example, an increase of DM mass flow, which could be realised by a bigger perforation of the screen around the screw press, would increase the amount of DM in the PF and thus the methane production. More electricity could be fed into the grid and more waste heat would be available for the drying of the PC. GHG mitigation would increase, since the substitution of fossil electricity production saves more GHG than the substitution of fossil thermal energy production (GEMIS, 2009). However, less PC would be available and the conversion

efficiency would decline, since the conversion of PC into thermal energy is more efficient than the conversion of methane into electric and thermal energy. Less solid fuel would be available for heating, even though thermal energy accounts for more than 50% of the total energy consumption in Germany, whereas electric energy only accounts for about 15-20% (BMW, 2010). This example shows, that in further research projects different scenarios have to be considered in terms of a complete life-cycle assessment and an economic analysis, in order to optimise the energy production and GHG mitigation with the IFBB process.

### **8.3 Predictability of mass and energy flows from biomass related parameters**

Regression models can be a useful tool to understand the roles of specific variables in a system in determining the performance of the system as a whole. Applications in agriculture are numerous and can, for example, comprise the estimation of clover performance in swards dependent on biotic and climatic factors (Wachendorf et al., 2001). A new approach in this study is the prediction of the performance of an energy conversion process (IFBB) based on biotic and process variables. A good predictability of mass and energy flows of the IFBB process from easily determinable biomass parameters, such as DM or fibre concentration, is important for a quick estimation of possible energy production and quality of energy carriers.

NDF showed a linear increase over time with increasing sward maturation, which is in line with observations by Čop et al. (2009), and was therefore chosen as chemical parameter in the silage representing the maturity of the sward. Together with DM concentration of the silage and temperature of hydrothermal conditioning it was a good predictor of mass and energy flows in multiple regression models (Table 6.4, Table 6.6 and Table 7.5). By using these models only two parameters, DM and NDF concentration of the silage (temperature of hydrothermal conditioning is a technical adjustment and is always known when analyzing IFBB data), have to be determined. It is, however, important to validate and potentially modify these models on other data sets of the conversion of grassland biomass through the IFBB process. Further research is needed for optimisation.

## 9 Conclusions

Based on two experiments, one with five-semi natural grassland swards (*Arrhenaterion* I and II, *Caricion fuscae*, *Filipendulion ulmariae*, *Polygono-Trisetion*) and a single sampling date, the second with one semi-natural grassland sward (*Arrhenaterion*) and eight consecutive sampling dates, in which the conversion of biomass through the IFBB process was comprehensively evaluated, the following conclusions can be drawn:

- (i) Mass flows of plant compounds in treatments with hydrothermal conditioning varied in a large range (0.16-0.95), with generally lower values in the first experiment and higher values in the second experiment. Apart from fibre fractions, all plant compounds showed higher mass flows than DM, which equates to an accumulation of these compounds in the PF. Mass flows of organic compounds were highest for CP and lowest for fibre fractions. Element mass flows were highest for Cl and lowest for N. Increasing temperature of hydrothermal conditioning resulted in increasing mass flows of all compounds, however, not always significantly. Different botanical composition of the grassland biomass in the first experiment had only minor effects on mass flows. Increasing sward maturity in the second experiment had an indefinite or negative effect on mass flows. Overall, a separation of easily digestible compounds as well as elements detrimental for combustion on the one hand and fibre fractions on the other hand was possible with the IFBB process, but there is still need for optimisation concerning certain compounds, such as N.
- (ii) Through the digestate of the PF after anaerobic digestion, a considerable part of essential plant nutrients (32-76% of N, 61-91% of P, 64-95% of K, 52-85% of Mg, 44-74% of Ca and 48-80% of S) contained in the harvested grassland biomass could be relocated to the grassland. Thus, the IFBB process meets the demands of sustainability to a large extent regarding the recycling of nutrients.
- (iii) Chemical composition of PF compared to the PM was characterized by an increase of ash, CP and ADL as well as a decline of CF and NDF. All elements detrimental for combustion were significantly reduced in the PC compared to the PM. In general, increasing temperatures of hydrothermal conditioning resulted in a higher reduction of elements in the PC, but no directional effect was determined on the compounds in the PF. With increasing sward maturity, CP and NFC in the PF declined, while NDF in the PF increased, and the influence of other organic compounds was not strongly directed.



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- Concentration of all elements in the PC declined with increasing sward maturity. IFBB-PC was of superior fuel quality compared to untreated grassland biomass (silage, hay).
- (iv) Specific methane yields and degrees of degradation of OM of PF were significantly higher compared to grassland silage in WCD. Area-specific methane yields of WCD were between 1 and 2.5 times as high as those of PF. There was a trend for PF towards higher yields with increasing sward maturity, but no influence of temperature of hydrothermal conditioning. However, PF without hydrothermal conditioning obtained higher specific methane yields at early stages of sward maturity. In contrast, sward maturity had a strong negative influence on specific methane yields in WCD.
  - (v) HHV of PC and untreated grassland biomass were in a comparable range. AST of PC was increased up to 1156-1254°C compared to untreated grassland biomass (1016-1111°C), indicating a high fuel quality of PC.
  - (vi) In the first experiment, net energy yield of the IFBB process was 11.9-14.1 MWh ha<sup>-1</sup>, conversion efficiency was 0.43-0.51 and GHG mitigation was 3.6-4.4 t CO<sub>2</sub>eq ha<sup>-1</sup>. Regarding these parameters, the IFBB process was inferior compared to CH, but superior compared to WCD. Performance of CH was strongly dependent on good weather conditions during curing of hay in the field, performance of WCD was strongly dependent on the utilization of waste heat from the CHP. CH produced only thermal energy, while IFBB produced thermal and electric energy with an internal and year-round concept of heat utilization.
  - (vii) In multiple regression models with main effects, quadratic terms and twofold interactions, three parameters, DM and NDF concentration in the silage as well as temperature of hydrothermal conditioning, were identified for the prediction of mass flows, elemental concentrations in the PC, organic compounds in the PF and energy parameters with high accuracy ( $R^2=0.70-0.99$ ).

## 10 Summary

Energy production from biomass and the conservation of ecologically valuable grassland habitats are two important issues of agriculture today. The combination of a bioenergy production, which minimises environmental impacts and competition with food production for land with a conversion of semi-natural grasslands through new utilization alternatives for the biomass, led to the development of the IFBB process. Its basic principle is the separation of biomass into a liquid fraction (press fluid, PF) for the production of electric and thermal energy after anaerobic digestion to biogas and a solid fraction (press cake, PC) for the production of thermal energy through combustion.

This study was undertaken to explore mass and energy flows as well as quality aspects of energy carriers within the IFBB process and determine their dependency on biomass-related and technical parameters. Two experiments were conducted, in which biomass from semi-natural grassland was conserved as silage and subjected to a hydrothermal conditioning and a subsequent mechanical dehydration with a screw press. Methane yield of the PF and the untreated silage was determined in anaerobic digestion experiments in batch fermenters at 37°C with a fermentation time of 13-15 and 27-35 days for the PF and the silage, respectively. Concentrations of dry matter (DM), ash, crude protein (CP), crude fibre (CF), ether extract (EE), neutral detergent fibre (NDF), acid detergent fibre (ADF), acid detergent ligning (ADL) and elements (K, Mg, Ca, Cl, N, S, P, C, H, N) were determined in the untreated biomass and the PC. Higher heating value (HHV) and ash softening temperature (AST) were calculated based on elemental concentration. Chemical composition of the PF and mass flows of all plant compounds into the PF were calculated.

In the first experiment, biomass from five different semi-natural grassland swards (*Arrhenaterion* I and II, *Caricion fuscae*, *Filipendulion ulmariae*, *Polygono-Trisetion*) was harvested at one late sampling (19 July or 31 August) and ensiled. Each silage was subjected to three different temperature treatments (5°C, 60°C, 80°C) during hydrothermal conditioning. Based on observed methane yields and HHV as energy output parameters as well as literature-based and observed energy input parameters, energy and green house gas (GHG) balances were calculated for IFBB and two reference conversion processes, whole-crop digestion of untreated silage (WCD) and combustion of hay (CH). In the second experiment, biomass from one single semi-natural grassland sward (*Arrhenaterion*) was harvested at eight consecutive dates (27/04, 02/05, 09/05, 16/05, 24/05, 31/05, 11/06, 21/06)

and ensiled. Each silage was subjected to six different treatments (no hydrothermal conditioning and hydrothermal conditioning at 10°C, 30°C, 50°C, 70°C, 90°C). Energy balance was calculated for IFBB and WCD. Multiple regression models were developed to predict mass flows, concentrations of elements in the PC, concentration of organic compounds in the PF and energy conversion efficiency of the IFBB process from temperature of hydrothermal conditioning as well as NDF and DM concentration in the silage.

Results showed a relative reduction of ash and all elements detrimental for combustion in the PC compared to the untreated biomass of 20-90%. Reduction was highest for K and Cl and lowest for N. HHV of PC and untreated biomass were in a comparable range (17.8-19.5 MJ kg<sup>-1</sup> DM), but AST of PC was higher (1156-1254°C). Methane yields of PF were higher compared to those of WCD when the biomass was harvested late (end of May and later) and in a comparable range when the biomass was harvested early and ranged from 332 to 458 L<sub>N</sub> kg<sup>-1</sup> VS. Regarding energy and GHG balances, IFBB, with a net energy yield of 11.9-14.1 MWh ha<sup>-1</sup>, a conversion efficiency of 0.43-0.51, and GHG mitigation of 3.6-4.4 t CO<sub>2</sub>eq ha<sup>-1</sup>, performed better than WCD, but worse than CH. WCD produces thermal and electric energy with low efficiency, CH produces only thermal energy with a low quality solid fuel with high efficiency, IFBB produces thermal and electric energy with a solid fuel of high quality with medium efficiency.

Regression models were able to predict target parameters with high accuracy ( $R^2=0.70-0.99$ ). The influence of increasing temperature of hydrothermal conditioning was an increase of mass flows, a decrease of element concentrations in the PC and a differing effect on energy conversion efficiency. The influence of increasing NDF concentration of the silage was a differing effect on mass flows, a decrease of element concentrations in the PC and an increase of energy conversion efficiency. The influence of increasing DM concentration of the silage was a decrease of mass flows, an increase of element concentrations in the PC and an increase of energy conversion efficiency. Based on the models an optimised IFBB process would be obtained with a medium temperature of hydrothermal conditioning (50°C), high NDF concentrations in the silage and medium DM concentrations of the silage.

## 11 Zusammenfassung

Die Energieerzeugung aus Biomasse und der Erhalt von ökologisch wertvollen Grünlandhabitaten sind zwei wichtige Aufgaben der heutigen Landwirtschaft. Die Kombination einer Bioenergieproduktion, die Umweltbelastungen und Flächenkonkurrenzen mit der Nahrungsmittelproduktion minimiert, und einem Erhalt von extensiv bewirtschaftetem Grünland durch neue Nutzungsalternativen führte zur Entwicklung des IFBB-Verfahrens. Dessen Grundprinzip ist die Separierung von Biomasse in eine flüssige Fraktion (Presssaft, PS) zur Strom- und Wärmeerzeugung nach einer Vergärung zu Biogas und eine feste Fraktion (Presskuchen, PK) zur Wärmeerzeugung durch Verbrennung.

Die vorliegende Untersuchung wurde durchgeführt, um Massen- und Energieflüsse sowie Qualitätsaspekte von Energieträgern im IFBB-Verfahren und deren Abhängigkeit von biomassespezifischen und technischen Parametern zu untersuchen. In zwei Versuchen wurde Biomasse von extensiv bewirtschafteten Grünlandbeständen siliert und nach einer hydrothermalen Konditionierung mit einer Schneckenpresse mechanisch entwässert. Die Methanausbeute des PS sowie der unbehandelten Silage wurde in Gärversuchen im Batch-Verfahren bei 37°C und einer Gärdauer von 13-15 Tagen (PS) beziehungsweise 27-35 Tagen (Silage) ermittelt. Die Gehalte an Trockenmasse (TM), Asche, Rohprotein (XP), Rohfaser (XF), Rohfett (XL), neutraler Detergentienfaser (NDF), saurer Detergentienfaser (ADF), saurem Detergentienlignin (ADL) und Elementen (K, Mg, Ca, Cl, N, S, P, C, H, N) wurden im PK und der unbehandelten Biomasse bestimmt. Der Brennwert ( $H_o$ ) und die Ascheerweichungstemperatur (ET) wurden auf Grundlage der Elementkonzentrationen berechnet. Die chemische Zusammensetzung des PS und die Massenflüsse aller Pflanzenbestandteile in den PS wurden berechnet.

Im ersten Versuch wurde Biomasse von fünf verschiedenen Grünlandbeständen (*Arrhenaterion* I and II, *Caricion fuscae*, *Filipendulion ulmariae*, *Polygono-Trisetion*) an einem späten Schnitttermin (19. Juli oder 31. August) geerntet und siliert. Jede Silage wurde in drei Temperaturvarianten (5°C, 60°C, 80°C) hydrothermal konditioniert. Mit den ermittelten Methanausbeuten und  $H_o$  als Energieoutput sowie gemessenen und der Literatur entnommenen Werten für den Energieinput wurden Energie- und Treibhausgas (THG)-Bilanzen des IFBB-Verfahrens sowie zweier Vergleichsverfahren, Ganzpflanzenvergärung der unbehandelten Silage (GPV) und Heuverbrennung (HV), errechnet. Im zweiten Versuch wurde Biomasse eines einzelnen Grünlandbestandes (*Arrhenaterion*) an acht aufeinander

folgenden Terminen (27.04, 02.05, 09.05, 16.05, 24.05, 31.05, 11.06, 21.06) geerntet und siliert. Jede Silage wurde in sechs Varianten (ohne hydrothermale Konditionierung und mit hydrothormaler Konditionierung bei 10°C, 30°C, 50°C, 70°C, 90°C) behandelt. Für IFBB und GPV wurde eine Energiebilanz gerechnet. Multiple Regressionsmodelle wurden erstellt, um Massenflüsse, Elementgehalte im PK, Gehalte an organischen Fraktionen im PS und die Konversionseffizienz des IFBB-Verfahrens anhand der Konditionierungstemperatur sowie des NDF- und des TM-Gehaltes der Silage abzuschätzen.

Die Ergebnisse zeigen eine relative Gehaltsminderung der Asche und aller für die Verbrennung schädlichen Elemente im PK im Vergleich zur unbehandelten Biomasse um 20-90%. Die Minderung war am stärksten bei K und Cl und am schwächsten bei N. Der  $H_o$  des PK und der unbehandelten Biomasse waren vergleichbar (17.8-19.5 MJ kg<sup>-1</sup> TM), die ET des PK war jedoch höher (1156-1254°C). Die Methanausbeute des PS war höher als die der GPV, wenn die Biomasse spät geerntet wurde (Ende Mai und später), aber vergleichbar, wenn die Biomasse früh geerntet wurde, und betrug 332-458 L<sub>N</sub> kg<sup>-1</sup> oTS. In Bezug auf Energie- und THG-Bilanzen war das IFBB-Verfahren mit einem Nettoenergieertrag von 11.9-14.1 MWh ha<sup>-1</sup>, einer Konversionseffizienz von 0.43-0.51 und einer THG-Vermeidung von 3.6-4.4 t CO<sub>2</sub>eq ha<sup>-1</sup> besser als GPV, aber schlechter als HV. GPV erzeugt Wärme und Strom bei geringer Effizienz, HV erzeugt nur Wärme bei geringer Brennstoffqualität und hoher Effizienz, IFBB erzeugt Wärme und Strom bei hoher Brennstoffqualität und mittlerer Effizienz.

Die Regressionsmodelle konnten die Zielparameter mit hoher Genauigkeit ( $R^2=0.70-0.99$ ) abschätzen. Der Einfluss einer steigenden Konditionierungstemperatur war ein Anstieg der Massenflüsse, eine Abnahme der Elementgehalte im PK und ein uneindeutiger Effekt auf die Konversionseffizienz. Der Einfluss eines steigenden NDF-Gehaltes in der Silage war ein uneindeutiger Effekt auf die Massenflüsse, eine Abnahme der Elementgehalte im PK und ein Anstieg der Konversionseffizienz. Der Einfluss eines steigenden TM-Gehaltes in der Silage war eine Abnahme der Massenflüsse, ein Anstieg der Elementgehalte im PK und ein Anstieg der Konversionseffizienz. Auf Grundlage der Modelle kann das IFBB-Verfahren durch die Kombination von mittlerer Konditionierungstemperatur (50°C), hohem NDF-Gehalt in der Silage und mittlerem TM-Gehalt in der Silage optimiert werden.

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## Appendix

It is assumed that (i) the DM content of PMC ( $DM_{PMC}$ ) depends on the DM content of PM ( $DM_{PM}$ ), the mass of PM ( $m_{PM}$ ) and the mass of PMC ( $m_{PMC}$ ), (ii) the mass of PMC ( $m_{PMC}$ ) is equal to the mass of PC ( $m_{PC}$ ) plus the mass of PF ( $m_{PF}$ ) and (iii) the mass of DM of PMC is equal to the mass of DM of PC plus the mass of DM of PF:

$$(i) \quad DM_{PMC} = DM_{PM} * \frac{m_{PM}}{m_{PMC}}$$

$$(ii) \quad m_{PMC} = m_{PC} + m_{PF}$$

$$(iii) \quad m_{PMC} * DM_{PMC} = m_{PC} * DM_{PC} + m_{PF} * DM_{PF}$$

The combination and rearrangement of equations (ii) and (iii) leads to:

$$(iv) \quad \frac{m_{PC}}{m_{PF}} * DM_{PMC} + DM_{PMC} = \frac{m_{PC}}{m_{PF}} * DM_{PC} + DM_{PF}$$

The quantity of press fluid as a proportion of the parent material after hydrothermal conditioning (X) is:

$$(v) \quad X = \frac{m_{PF}}{m_{PMC}} = \frac{m_{PF}}{m_{PF} + m_{PC}} = \frac{1}{1 + \frac{m_{PC}}{m_{PF}}} \Rightarrow \frac{m_{PC}}{m_{PF}} = \frac{1}{X} - 1$$

The combination of equations (iv) and (v) leads to:

$$(vi) \quad \frac{1}{X} * DM_{PMC} - DM_{PMC} + DM_{PMC} = \frac{1}{X} * DM_{PC} - DM_{PC} + DM_{PF}$$

which can be rearranged to:

$$(vii) \quad X = \frac{DM_{PC} - DM_{PMC}}{DM_{PC} - DM_{PF}}$$

The corresponding quantity of press cake as a proportion of the parent material after hydrothermal conditioning (Y) is:

$$(viii) \quad Y = 1 - X$$

The term mass flow (MF) signifies the proportion of DM or any other compound (Z) that is transferred from PMC to PC and PF, respectively. The concentration of Z (g kg<sup>-1</sup> DM) in PMC and in PM can be assumed equal, as the mash water generally contains negligible concentrations of nutrients. DM mass flow for PF (MF\_DM<sub>PF</sub>) was determined by:

$$(ix) \quad MF\_DM_{PF} = \frac{m_{PF} * DM_{PF}}{m_{PMC} * DM_{PMC}} = \frac{X * m_{PMC} * DM_{PF}}{m_{PMC} * DM_{PMC}} = \frac{X * DM_{PF}}{DM_{PMC}}$$

and DM mass flow for PC (MF\_DM<sub>PC</sub>) was determined by:

$$(x) \quad MF\_DM_{PC} = \frac{Y * DM_{PC}}{DM_{PMC}}$$

The combination of (x), (viii), (iii) and (v) leads to:

$$(xi) \quad MF\_DM_{PC} = \frac{(1-X) * \frac{m_{PMC} * DM_{PMC} - m_{PF} * DM_{PF}}{m_{PMC} - m_{PF}}}{DM_{PMC}} = \frac{(1-X) * \frac{DM_{PMC} - \frac{m_{PF}}{m_{PMC}} * DM_{PF}}{1 - \frac{m_{PF}}{m_{PMC}}}}{DM_{PMC}}$$

$$= \frac{(1-X) * \frac{DM_{PMC} - X * DM_{PF}}{1-X}}{DM_{PMC}} = 1 - \frac{X * DM_{PF}}{DM_{PMC}} = 1 - MF\_DM_{PF}$$

Mass flows of Z in PF (MF\_Z<sub>PF</sub>) and PC (MF\_Z<sub>PC</sub>) were calculated according to:

$$(xii) \quad MF\_Z_{PF} = \frac{m_{PF} * DM_{PF} * Z_{PF}}{m_{PMC} * DM_{PMC} * Z_{PMC}} = \frac{X * DM_{PF} * Z_{PF}}{DM_{PMC} * Z_{PMC}}$$

$$(xiii) \quad MF\_Z_{PC} = \frac{Y * DM_{PC} * Z_{PC}}{DM_{PMC} * Z_{PMC}} = 1 - MF\_Z_{PF}$$

In conclusion, the mass flow of each compound (represented by Z) in the PF can be calculated (by the combination of (xii) and (xiii)) from the concentration of Z in PM and PMC according to:

$$(xiv) \quad Z_{PF} = \frac{DM_{PMC} * Z_{PM} - Y * DM_{PC} * Z_{PC}}{X * DM_{PF}}$$