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Aqueous Leaching Prior to Dewatering Improves the Quality of Solid Fuels from Grasslands

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Abstract: Renewable energies are necessary to reduce greenhouse gas emissions in energy production, and biomass plays a dominant role in the renewable energy sector. Combustion of biomass constitutes an efficient conversion technique, but is hindered by harmful elements which are frequently contained in residual grassland biomass. We investigated the effect of leaching on solid fuel quality with three independent experiments including the following treatments: (1) with or without leaching, (2) with leaching at various water to silage ratios and (3) with the use of press liquid versus fresh water. Biomass was mechanically dehydrated and press cakes for combustion were produced and analyzed for their concentrations of ash, N and minerals harmful for combustion. Solid fuel quality was improved by leaching prior to dewatering, and the application of higher proportions of fresh water enabled even higher quality to be attained.

Keywords: IFBB; biomass; fuel; combustion; leaching

1. Introduction

Renewable energies have been proposed as a solution to the huge challenges of the 21st century since they trigger economic growth, provide new jobs, help to mitigate climate change [1] and are perceived as a clean energy source [2]. Therefore, political measures have been put in place to subsidize and foster the development of renewable energies, resulting in a rapidly growing contribution of renewable energies to total energy consumption [3]. Biomass is one of the major sources of renewable energy production worldwide [4]. However, there are also some negative side effects to the increased production of renewable energy from biomass which threaten its social acceptance [5,6]. Studies have shown that the carbon neutrality of bioenergy is debatable, depending on the choice of feedstock, land use-changes, processing methods applied and other indirect effects of removal of biomass for energy production [7,8]. In order to produce sustainable renewable energy, biomass should be used that is not suitable for human or animal consumption in adapted energy recovery systems. Such a system was developed by the University of Kassel with the Integrated Generation of Solid fuel and Biogas from Biomass (IFBB) [9]. IFBB consists of a leaching step followed by mechanical dehydration. The leaching is carried out by putting the biomass into a modified concrete mixer with addition of heated water in a defined ratio for a defined time under permanent mechanical stirring. This step is identical to the step referred to as hydrothermal conditioning in previous publications about the IFBB system. It results in a press liquid (PL) containing the water-soluble parts of the biomass [10] and a press cake (PC) which has reduced concentrations of mineral elements harmful for combustion and can thus be used as a solid fuel in state-of-the-art combustion units [11]. IFBB has proven its ability to work with a range of residual biomass types that are currently not used for human consumption or animal forage, but rather are left to decay on the field and roadsides or are composted. The system was tested with biomass from roadside verges [12], leaves from parks [13,14] and streets [15], horse manure [16], cuttings from sports fields [17] and late cuts of semi-natural grassland invaded by the alien plant species *Lupinus polyphyllus* [18]. A life cycle assessment was carried out and proved the sustainability of the IFBB system [19]. The first step in IFBB is a leaching step, whereby the biomass is submersed in and mixed with warm water, typically at 40 °C and in a ratio of 1 part biomass to 4 parts water, and stirred for 15 min. Hardly any systematic research exists regarding this critical step of IFBB: only Richter et al. [20] investigated the effects of water temperature. Little is known about the effect of other influential parameters, such as the silage to water ratio or the type of mashing liquid used. Improving the leaching step is of interest for economic reasons, considering the costs of using fresh water, and ecological reasons, e.g., considering the amount of fresh water a commercial IFBB plant would need to produce renewable energy. The present paper aims to close these knowledge gaps by summarizing results of three independent experiments which systematically investigated the leaching step to answer the following three research questions:

1. How does leaching affect fuel quality?
2. What is the effect of the silage to water ratio applied in leaching on fuel quality parameters?
3. What is the effect of using recycled press liquid for leaching instead of fresh water?

2. Results

2.1. Experiment 1: Effect of the Leaching Step within the IFBB System

IFBB reduced total ash and mineral concentrations both with (PC+L) and without (PC-L) applying the leaching step (L) (Figure 1), but PC+L showed a higher overall mean reduction rate (37.5%, 23.0%, 38.3%, 83.5%, 32.0% and 92.0% for ash, N, S, K, Mg and Cl, respectively) compared to PC-L (24.2%, 15.3%, 26.8%, 52.5%, 25.2% and 63.9% for ash, N, S, K, Mg and Cl, respectively).

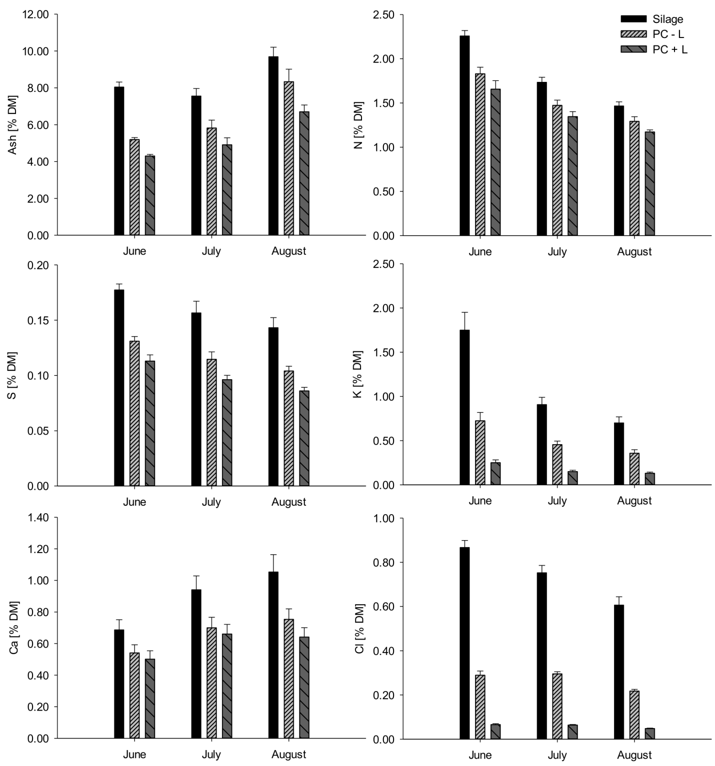

Figure 1. Experiment 1: Mean concentrations of ash, N, S, K, Ca and Cl in silage and press cake (PC), with (PC+L) and without (PC-L) leaching step (L) at different sampling dates (June = 17 June 2013, July = 15 July 2013, August = 13 August 2013). Error bars show the standard errors of the means. DM: dry matter.

The IFBB treatment reduced the total ash and mineral concentrations regardless of the harvest date (Figure 1, Table A1); thus, the nitrogen and mineral concentrations were lower in the solid fuel of the PC+L treatment. These effects were statistically significant for ash, S, K, and Cl (Table 1). Additionally, a reduction of N, S, K and Cl in the PC of biomass harvested later was also observed (Figure 1). The highest values were measured in June and lowest values in August, with values in July falling in between. While Ca concentrations increased with time, total ash decreased from June to July and then increased in August.

Table 1. Experiment 1: *p*-values of the Wilcoxon Test for differences in ash, N, S, K, Ca and Cl concentrations in press cakes due to the application of leaching step. Significant values ($p < 0.05$) are in bold letters.

Parameter	*p*-Value
Ash	0.041
N	**0.149**
S	0.004
K	<0.001
Ca	**0.191**
Cl	<0.001

2.2. Experiment 2: Effect of the Water to Silage Ratio in the Leaching Step within the IFBB System

Concentrations of ash, N, S, K Ca and Cl were reduced through the IFBB treatment for both silage types at all ratios of water to silage (Figure 2, Table A2). While the *Nardus stricta* silage had lower ash and mineral element concentrations than the *Lupinus* silage (Figure 2), the *Lupinus* silage showed higher reduction rates when treated with IFBB.

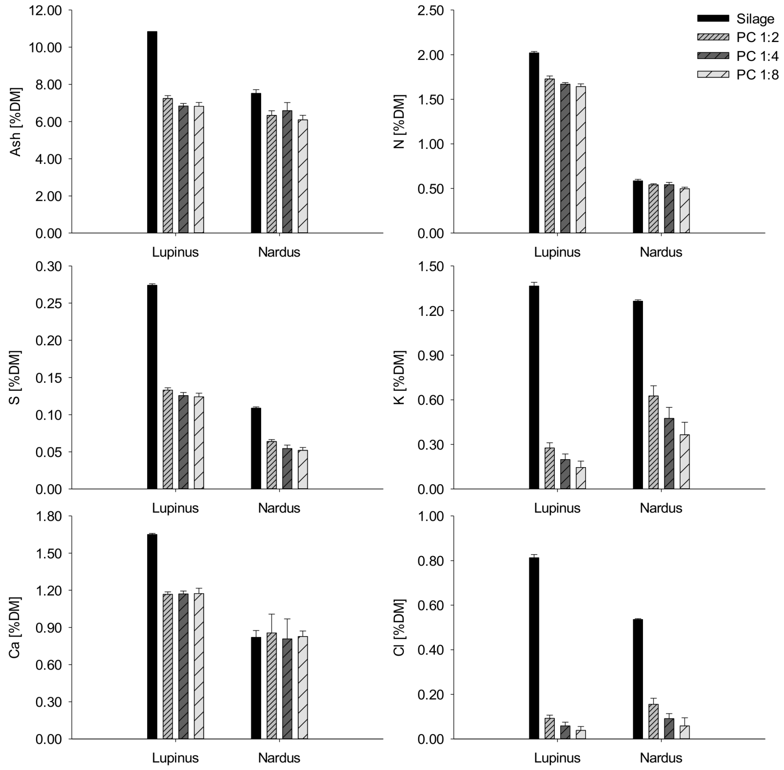

Figure 2. Experiment 2: Mean concentrations of ash, N, S, K, Ca and Cl in silage and PC from grassland dominated by *Lupinus polyphyllus* (Lupinus) or *Nardus stricta* (Nardus). The PC was leached with a water to biomass ratio of 1:2, 1:4 or 1:8, respectively. Whiskers display standard errors of the means.

Reduction rates increased with increasing shares of water. Differences in nutrient concentrations in the PCs between ratio treatments were statistically significant for the *Lupinus* material in the case of N, K and Cl (Table 2). In all cases, the 1:8 ratio resulted in the lowest concentrations of elements, but only for K and Cl were concentrations significantly different across all three ratios, whereas for N, the concentrations differed significantly only between ratios of 1:8 and 1:2. For the *Nardus* biomass, S, K and Cl concentrations were significantly lower for the 1:4 and 1:8 treatments than for 1:2, but a significant further reduction with a ratio of 1:8 was only achieved for K and Cl.

Table 2. Experiment 2: Statistical results for ash, N, S, K, Ca and Cl concentrations in press cakes of grassland dominated by *Lupinus polyphyllus* or *Nardus stricta* created by leaching with water to biomass ratios of 1:2, 1:4 or 1:8, respectively.

Parameter	Shapiro-Wilk-Test	Levene-Test	ANOVA	Tukey-HSD-Test
	***p*-Value**	***p*-Value**	***p*-Value**	**1:2, 1:4, 1:8**
Lupinus polyphyllus dominated grassland				
Ash	0.744	0.554	0.087	
N	0.378	0.824	0.025	a, ab, b
S	0.138	0.579	0.579	
K	0.132	0.492	<0.001	a, b, c
Ca	0.831	0.774	0.988	
Cl	0.008	0.630	<0.001	a, b, c
Nardus stricta dominated grassland				
Ash	0.520	0.923	0.786	
N	0.715	0.845	0.082	
S	0.830	0.630	0.011	a, b, b
K	0.163	0.813	<0.001	a, b, c
Ca	0.287	0.707	0.975	
Cl	0.208	0.273	<0.001	a, b, c

ANOVA: Analysis of variance; HSD: Honest significant difference, different letters (a,b,c) indicate significant differences between groups

2.3. Experiment 3: Effect of Using Recycled Press Liquid as a Leaching Liquid instead of Fresh Water

Press liquids obtained from dewatering biomass from semi-natural (SNG) and intensive (ING) grasslands without addition of water during leaching showed higher elemental concentrations in comparison to fresh water (Table 3).

Table 3. Experiment 3: Mineral concentrations in press liquids (PL), obtained from dewatering biomass from semi-natural (SNG) and intensive (ING) grasslands, and tap water.

Parameter	SNG PL	ING PL	Water
	(mg kg^{-1})	**(mg kg^{-1})**	**(mg L^{-1})**
DM	17	8	0
Ca	3040	2050	26
K	11,400	2230	1
Mg	1190	1270	9
P	1330	443	n.d.
S	456	387	2
Cl	2694	649	5

n.d. = not detectable.

The IFBB treatment did reduce all element concentrations, irrespective of the type of biomass and leaching liquid (Figure 3, Table A3). Semi-natural grassland silage contained lower concentrations of ash, N, K and Cl, but higher concentrations of S and Ca than ING silage. The use of pure water delivered superior fuel quality for both biomass types compared to the use of press liquid (Table 4). While the fluid type significantly affected all measured concentrations for the SNG biomass, there was no significant effect of the fluid type on Ca and ash concentration for the ING biomass.

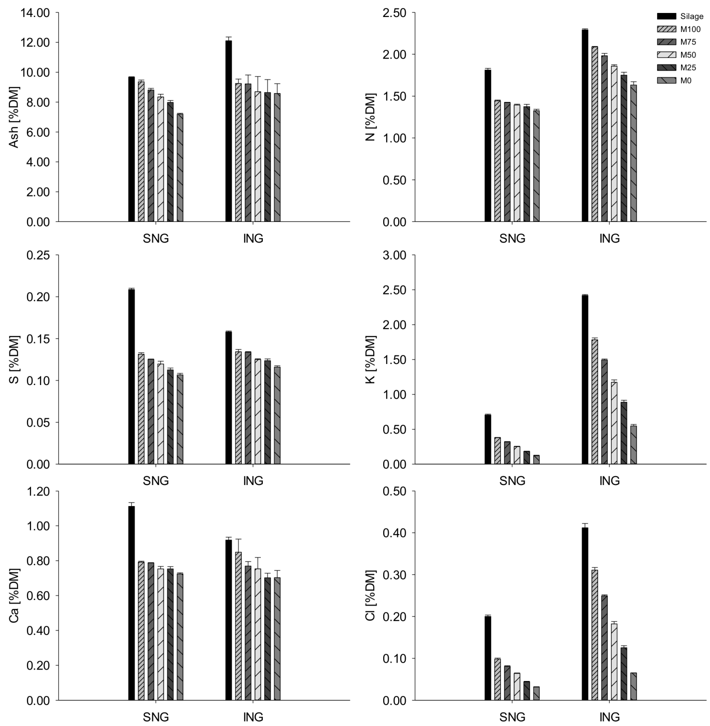

Figure 3. Experiment 3: Mean concentrations of ash, N, S, K, Ca and Cl in silage and press cakes obtained from dewatering after leaching with liquids with varying shares of recycled press liquid (M100/75/50/25/0 = 100%/75%/50%/25%/0% press liquid). Elemental concentrations are shown for biomass from semi-natural (SNG) and intensive (ING) grassland biomass. Error bars depict the standard errors of the means.

Table 4. Experiment 3: Statistical results for ash, N, S, K, Ca and Cl concentrations in press cakes from semi-natural and intensive grassland biomass, as obtained after leaching with liquids with varying shares of recycled press liquid (M100/75/50/25/0 = 100%/75%/50%/25%/0% press liquid).

Variable	Shapiro-Wilk-Test	Levene-Test	ANOVA	Tukey-HSD-Test
	p-Value	*p*-Value	*p*-Value	M100, M75, M50, M25, M0
Semi-natural grassland biomass				
Ash	0.833	0.786	<0.001	a, ab, bc, c, d
N	0.200	0.796	0.004	a, a, ab, ab, b
S	0.922	0.699	<0.001	a, ab, bc, cd, d
K	0.228	0.914	<0.001	a, b, c, d, e
Ca	0.215	0.723	0.003	a, a, ab, ab, b
Cl	0.728	0.859	<0.001	a, b, c, d, e
Intensive grassland biomass				
Ash	0.765	0.751	0.931	
N	0.455	0.776	<0.001	a, ab, bc, cd, d
S	0.627	0.776	<0.001	a, a, b, bc, c
K	0.331	0.959	<0.001	a, b, c, d, e
Ca	0.358	0.769	0.303	
Cl	0.795	0.609	<0.001	a, b, c, d, e

a,b,c,d,e,: different letters indicate that differences between groups (M100, M75,M50, M25, M0) are statistically significant.

2.4. 2S/Cl Ratio

Values for the 2S/Cl ratio found in silages of all experiments were very low (<1) (Table 5), indicating a high risk of corrosion when combusted without any pre-treatment [21]. Mechanical separation with a screw press without prior leaching (PC-L) resulted in a small increase in the 2S/Cl ratio, whereas additional leaching (PC+L) led to a substantial increase, resulting in values up to about 4.00. High ratios of fresh water (PC 1:4, PC 1:8) led to higher values of the 2S/Cl index (up to a mean value of 7.20) whereas low shares of fresh water (PC 1:2) showed only a minor effect. Leaching with fresh water (M0) resulted in the highest index values of up to 7.48 and 3.96 for SNG and ING, respectively, whereas the use of pure press liquid (M100) showed minor increases for both silage types.

Table 5. Arithmetic means of 2S/Cl index in silages and press cakes (PC); ± indicates the standard errors of the means.

		2S/Cl				
Experiment 1	Silage	PC+L [1]	PC-L [2]			
June	0.46 ± 0.02	3.85 ± 0.15	1.02 ± 0.05			
July	0.47 ± 0.04	3.38 ± 0.16	0.86 ± 0.03			
August	0.54 ± 0.06	4.00 ± 0.09	1.07 ± 0.08			
Experiment 2	Silage	PC 1:2 [3]	PC 1:4 [4]	PC 1:8 [5]		
Lupinus	0.75 ± 0.01	3.19 ± 0.12	4.78 ± 0.14	7.20 ± 0.32		
Nardus	0.45 ± 0.01	0.93 ± 0.08	1.32 ± 0.05	1.97 ± 0.09		
Experiment 3	Silage	M100 [6]	M75 [7]	M50 [8]	M25 [9]	M0 [10]
SNG [11]	0.66 ± 0.01	2.96 ± 0.03	3.45 ± 0.07	4.15 ± 0.04	5.74 ± 0.22	7.48 ± 0.13
ING [12]	0.86 ± 0.02	0.96 ± 0.03	1.19 ± 0.01	1.52 ± 0.04	2.20 ± 0.12	3.96 ± 0.02

[1,2] with and without leaching, respectively; [3–5] leached with a water to biomass ratio of 1:2, 1:4 or 1:8, respectively; [6–10] leached with liquids with varying shares of recycled press liquid (M100/75/50/25/0 = 100%/75%/50%/25%/0% press liquid, respectively); [11,12] semi-natural and intensive grassland silage, respectively.

3. Discussion

Richter et al. [22] also investigated the reduction of minerals in press cakes with and without leaching. They found that the concentrations of elements in the press liquid were significantly higher if the biomass was leached before dewatering, corresponding to higher mass flows of elements into the PL [22]. Thus, the higher reduction rates of elements in the PC with leaching found in the current study confirm these findings. Concerning the date of harvest, the present results are not fully comparable with those of Richter et al. [22], as the harvest dates in that study were earlier (April–June). Nevertheless, they also found that material from the earlier sampling dates showed a higher mass flow of minerals and N into the press liquid, resulting in higher reduction rates for those elements in the press cake. This higher reduction may be attributed to plant tissue being softer at earlier stages of crop maturity, which may particularly apply to N, as it predominantly occurs in the form of structurally insoluble proteins at later stages of maturity [23]. However, Corton et al. [24] also investigated the IFBB system with and without leaching and found no difference in the reduction of ash, K, Mg, Na, and P. This might be explained by the fact that Corton et al. [24] used a different leaching set-up (i.e., cold water added directly into the screw press) compared to most other IFBB studies. Furthermore, they used a special plant biomass, from *Juncus effusus* dominated grasslands. *Juncus effusus* has a water-repellent outer layer which may explain the lower reduction rates found by Corton et al. [24].

According to Obernberger et al. [25], guiding concentrations of N, Cl and S for unproblematic combustion are <0.6%, <0.1% and <0.1% DM, respectively. The guiding value could not be reached for N, irrespective of the type of treatment or date of harvest. However, this does not necessarily lead to problems with NO_x emissions if appropriate biomass combustion units equipped with air or fuel staging are used [26,27]. Considering Cl, values only stayed below the guiding value if a leaching step was applied. In case of S, concentrations below the guiding value were achieved for the later harvest dates (July and August) with leaching. The results show that leaching plays a crucial role within the IFBB system to produce a press cake with acceptable mineral concentrations: without leaching, the resulting press cake will not have the adequate quality for use as a solid fuel. These findings also have relevance for other thermo-chemical processes, such as pyrolysis, as they have similar demands considering the feedstock quality [28,29]. However, the chemical composition analysis can only be the first step of an investigation of the combustion quality of a fuel. Combustion tests should be carried out and emissions measured. This was done for the IFBB PC for example by Bühle et al. [30], showing that the treatment of semi-natural grassland biomass led to increased performance in terms of increased ash deforming temperatures. Khalsa et al. [14] also performed combustion tests with pellets made from foliage and grass samples that were treated with a leaching and subsequent dewatering treatment comparable to the IFBB treatment. The resulting emissions were in accordance with German legal thresholds for CO emissions, but slightly above threshold values for NO_x emissions occurred for the grass based fuels, but not for the foliage based fuels. The emission tests highlighted the need for a filter to reduce particulate matter emissions.

Furthermore, the results showed higher mineral reduction rates with increased shares of fresh water used in leaching. Tonn et al. [31] investigated the effect of leaching on semi-natural grassland biomass and found significant concentration reductions for Cl, K, S, Mg and ash. These reductions increased with the amount of water used in the experiments, as well as with the time the biomass was exposed to the leaching treatment. K and Cl were the elements that showed the greatest reduction through leaching, which is due to the fact that these elements are present in plants in their ionic forms [32] and, thus, can be easily leached. In the present experiment, results below the guiding values for combustion proposed by Obernberger et al. [25] could not be achieved for every biomass and silage to water ratio. The N concentration in the silage dominated by *Lupinus* was high and the IFBB treatment did not lead to a high enough reduction to reach the target value of 0.6, whereas the *Nardus*-dominated silage and the resulting PCs produced at all silage to water ratios were below the critical value. For the Cl concentration, the ratio of silage to water made a crucial difference: for the *Nardus*-dominated biomass, only the PCs at the 1:4 and 1:8 ratio achieved concentrations below the guiding value. Thus, the concentrations of elements in the original biomass has a strong influence on whether an adequate fuel quality can be achieved, but using larger shares of fresh water helped to reduce the mineral concentrations. However, the achievement of relatively small improvements in fuel quality with higher amounts of fresh water used have to be weighed against the higher costs and ecological impacts of an increased water consumption. There is a need for further research on the question of the optimal silage to water ratio from the economic and ecological point of view.

The liquids used for leaching (i.e., press liquids of ING or SNG silages and fresh water) were very different in chemical composition, which is due to the fact that the press liquids contained minerals that were extracted from the initial biomass. This resulted in lower reduction rates through leaching when press fluids were used instead of fresh water, as the solubility of minerals in a solution depends, among other factors, on the concentration of minerals already dissolved in the liquid. Comparable experiments using the press liquid as a leaching solution were carried out by Graß et al. [33] for wheat silage. They also found lower reduction rates for ash and N, but no significance difference for K if press liquid was used instead of fresh water. The concentrations of minerals in the press cakes produced by Graß et al. [33] with fresh water were tentatively lower than those produced using press liquid. Regarding the present study and concerning the suitability of the produced press cake as a solid fuel, it has to be stated that although a reduction in mineral concentrations was achieved with IFBB for both biomass types and all leaching liquids, the values for N and S were above the guiding values for unproblematic combustion [25]. In the case of Cl, the concentrations in the SNG PC were below the guiding value for all leaching liquids except the pure PL. For the ING PC, only mashing with pure water achieved Cl concentrations below the guiding value. Thus, it can be concluded that a pure water leaching is more likely to achieve adequate solid fuel qualities with a wider range of input materials than a press fluid leaching.

The 2S/Cl ratio is described in detail by Sommersacher et al. [21]. It was shown that the molar 2S/Cl ratio is an indicator for high-temperature corrosion risk. Sommersacher et al. [21] stated that if fuels have a 2S/Cl ratio >4, only minor risk of corrosion is to be expected, and if the ratio is >8, corrosion risk is negligible. The values found in the silages were below 1, indicating a high risk of corrosion due to high Cl concentrations. As Cl is easily soluble in water, it was washed out to a greater extent than S, which led to increased 2S/CL ratios in the PC. Experiment 1 showed that the reduction of Cl achieved with the leaching step was much higher than without such a treatment. Thus, the PC+L had a higher ratio and lower corrosion risk, while the risk for the PC-L was still high. When more water was added in Experiment 2, the increase to the wash-out rate of Cl was higher than the increase to the wash-out rate of S, leading to an even higher 2S/Cl ratio. This means a decreased risk of corrosion with increased water addition to the silage, resulting in PC with low risk of corrosion for water to silage ratios of 1:4 and 1:8, but not for 1:2. Experiment 3 showed that using pure water (M0) was superior in reducing S and Cl in comparison with partial or pure PL (M25-M100). Again, the wash-out rate of Cl was higher than that of S, leading to a high increase in the 2S/Cl ratio for the pure water treatment (M0) with low risk of corrosion and a small increase in the 2S/Cl ratio but still high risk of corrosion for the pure PL (M100). The results of the 3 experiments with regard to risk of corrosion can be summarized as follows: The risk of corrosion is lower with leaching, and lower still if pure water is used in a ratio of silage to water of 1:4 or 1:8.

4. Materials and Methods

4.1. Experiment 1: Effect of the Leaching Step within the IFBB System

4.1.1. Biomass Sampling

Biomass samples were taken in the Rhön UNESCO Biosphere reserve, which is located in the German states of Hesse, Bavaria and Thuringia. The samples were collected in the Bavarian part. Five typical lower mountain grassland sites were chosen which were infested with the invasive alien plant species *Lupinus polyphyllus* (Lindl.). Each grassland site contained one plot of 50 m^2, which was equally divided into 3 sub-plots for 3 different harvest dates. The sub-plots were harvested on the 17th of June, 15th of July and 13th of August in 2013. Harvest took place with a finger bar mower at 5-cm cutting height. Biomass from each subplot was compressed in a separate airtight 60-L polyethylene barrel without any headspace and stored for ensiling until October 2013. A total of 15 samples were taken (3 dates $\times$ 5 replications).

4.1.2. Processing of the Biomass

After ensiling and storage, the samples were processed. Each barrel was opened, mixed and silage samples for chemical analysis and dry matter determination were taken. Ten kg of silage from each sample (n = 5) was weighed and directly placed in the screw press (type AV, Anhydro GmbH, Kassel, Germany). The screw had a pitch of 1:6 and a rotational speed of 6 rpm. The cylindrical screen encapsulating the screw had a perforation of 1.5 mm. The resulting press liquid was discarded, while the press cake (without prior leaching (PC-L)) was weighed and collected for analysis. Another 10 kg of silage from each sample (n = 5) was weighed and put into a modified concrete mixer by adding warm water (40 °C) to create a silage to water ratio of 1:4 for 15 min. The mash (water + silage) was then put into the screw press and processed. The press cake (with leaching (PC+L)) was weighed and sampled for analysis.

4.1.3. Analysis

Dry matter (DM) was determined by drying the samples at 105 °C in a drying oven for 48 h. Ash concentration was measured by incineration of a dried subsample at 550 °C in a muffle furnace. Volatile solids were defined as DM minus the ash concentration. Silage and PCs were dried for 48 h at 65 °C for elemental analysis. Samples for elemental analysis were ground with a cutting mill (SM 1; Retsch GmbH, Haan, Germany) to pass a 4 mm sieve and, subsequently, with a sample mill (1093 Cyclotec; Foss GmbH, Hamburg, Germany) to 1 mm. X-ray fluorescence analysis was conducted to determine concentrations of Cl, K, Ca, P, S, and Mg. C, H and N concentrations were determined with an elemental analyzer (Vario Max CHN; Elementar Analysesysteme GmbH, Hanau, Germany).

4.1.4. Statistics

Statistical tests were conducted by utilizing the software R [34]. A non-parametric test was chosen to determine if the leaching treatment had an effect on the chemical composition of PCs. The Wilcoxon-Test was used to test for differences in the concentrations of ash, N, S, K, Ca and Cl in PC+L and PC-L samples.

4.2. Experiment 2: Effect of the Water to Silage Ratio in the Leaching Step of the IFBB System

4.2.1. Biomass Sampling

Silage samples were taken from the Rhön biosphere reserve (Germany) by purchasing silage bales wrapped in plastic foil from local farmers. Four silage bales with a weight of approximately 400 kg each were purchased, two of them containing *Lupinus polyphyllus* (Lindl.) infested biomass from a wet area of the biosphere reserve and two of them containing *Lupinus polyphyllus* free biomass from a dry *Nardus stricta* (L.) dominated grassland in the biosphere reserve. The bales were harvested and conserved in July (*Nardus*) and August (lupine) 2015 and stored.

4.2.2. Processing of the Biomass

Silage bales were opened and processed in April 2016. The two lupine bales were opened and material infested with mold was removed. The clean material from the two bales was mixed and cut with a maize chopper to approximately 4-cm fiber length. Approximately 15 kg of silage was then filled into each of 9 60-L polyethylene barrels. The material from the two *Nardus* bales was treated accordingly, but due to the higher DM content, each 60-L polyethylene barrel was only filled with 10 kg of silage. Two samples of *Lupinus* silage and 3 samples of *Nardus* silage were taken for analysis. 15-kg or 10-kg samples of silage (*Lupinus* or *Nardus*, respectively) were weighed and were subjected to leaching. Three ratios of silage to water (1:2, 1:4 and 1:8) were tested, with 3 replicates per ratio and silage type. The leaching was followed by mechanical separation with the same machinery and settings as described in experiment 1 (Section 4.1.2). The resulting press cakes were weighed and collected for analysis.

4.2.3. Analysis

Dry matter, volatile solids, ash content, mineral content (Cl, K, Ca, P, S, and Mg) and C, H and N content of silage and PC were determined as described in detail for experiment 1 (Section 4.1.3).

4.2.4. Statistics

For each biomass type, a separate one-way analysis of variance (ANOVA) was carried out to test for differences between the ash, N, S, K, Ca and Cl concentrations of the PCs with different silage to water ratios. The assumptions of ANOVA were tested using the Shapiro-Wilk-Test to test for the normality of the distribution of residuals and the Levene's-Test to test for the equality of variances. All assumptions were met, except for the case of the Cl concentration in lupine PC, for which the Shapiro-Wilk-Test indicated a violation of the normality assumption. As a post-hoc test, the Tukey-honest significant difference (HSD)-test was carried out.

4.3. Experiment 3: Effect of Using Recycled Press Liquid Ainstead of Fresh Water

4.3.1. Biomass and Press Liquid Sampling

Two different kinds of silages were used. The extensive grassland biomass was harvested and ensiled in the lower mountain region of the Vogelsberg (Hesse, Germany) from semi-natural grassland under Natura 2000 protection. The intensive grassland silage was produced in close proximity to the University of Kassel in Witzenhausen, Germany. Both silage types were chopped with a modified maize chopper to a fiber length of about 4 cm. Material from both biomass types was separately introduced into the screw press without leaching and with the same machinery and settings described in Experiment 1 (Section 4.1.2) to produce 600 L of pure press liquid from each biomass type.

4.3.2. Processing of the Biomass

Five samples of each silage type were taken for analysis. 20-kg samples of silage were mixed with a leaching liquid and processed according to the IFBB system. For leaching, five different liquids were tested with three replicates. The treatments consisted of different mixtures of fresh water and PL as follows: M100 = 100% PL, M75 = 75% PL and 25% water, M50 = 50% PL and 50% water, M25 = 25% PL and 75% water and M0 = 100% water. The press liquid for leaching was taken from the corresponding silage type so that the extensive grassland samples were leached with press liquid from extensive silage. The following mechanical separation was done as described in Experiment 1 (Section 4.1.2).

4.3.3. Analysis

Dry matter, volatile solids, ash content, mineral content (Cl, K, Ca, P, S, and Mg) and C, H and N content of silage and PCs were determined as described in detail for experiment 1 (Section 4.1.3).

4.3.4. Statistics

For each biomass type, a separate one-way ANOVA was carried out to test for differences between the ash, N, S, K, Ca and Cl concentrations of the PCs with different water to PL ratios. The assumptions of ANOVA were tested using the Shapiro-Wilk-Test to test for the normality of the distribution of residuals and the Levene's-Test to test for the equality of variances. All assumptions were met. As a post-hoc test, the Tukey-HSD-Test was carried out.

5. Conclusions

The present study reports findings from three experiments which deal with various aspects of leaching prior to mechanical separation for solid fuel production from biomass. Leaching prior to mechanical separation leads to lower concentrations of harmful elements in the produced press cake. The silage to water ratio in leaching does have an effect on the fuel quality parameters, with higher amounts of fresh water leading to higher reductions of harmful elements and, thus, higher fuel quality. Pure fresh water as a leaching liquid reduces harmful elements to a much greater extent than the use of pure press liquid or a mixture of press liquid and water. Thus, the present study fills knowledge gaps about the treatment of biomass for improved solid fuel production from biomass and demonstrates the role of an adopted pre-treatment within the integrated generation of solid fuel and biogas from biomass (IFBB).

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Appendix A

Table A1. Rate of reduction (%) of concentrations of Ash, N, S, K, Ca and Cl from biomass DM to press cake DM achieved in Experiment 1.

	June		July		August	
Parameter	PC+L [1]	PC-L [2]	PC+L [1]	PC-L [2]	PC+L [1]	PC-L [2]
Ash	46.53	35.45	35.05	22.99	30.82	14.01
N	26.65	18.94	22.41	15.07	20.07	11.89
S	36.30	26.16	38.57	26.82	39.94	27.37
K	85.67	58.61	83.46	49.87	81.26	48.94
Ca	27.06	21.33	29.83	25.65	39.14	28.48
Cl	92.43	66.65	91.58	60.79	92.12	64.18

[1,2] with and without leaching, respectively. DM: dry matter.

Table A2. Rate of reduction (%) of concentrations of Ash, N, S, K, Ca and Cl from biomass DM to press cake DM achieved in Experiment 2.

	Lupinus			*Nardus*		
Parameter	PC 1:2 [1]	PC 1:4 [2]	PC 1:8 [3]	PC 1:2 [1]	PC 1:4 [2]	PC 1:8 [3]
Ash	33.20	36.94	37.07	15.73	12.35	18.96
N	14.47	17.35	18.71	7.71	7.34	15.26
S	51.46	54.12	54.74	41.12	50.05	52.16
K	79.78	85.47	89.38	50.50	62.40	71.13
Ca	29.29	29.09	28.89	−4.35	1.59	−0.82
Cl	88.60	92.83	95.29	70.99	82.94	89.03

[1–3] leached with a water to biomass ratio of 1:2, 1:4 or 1:8, respectively.

Table A3. Rate of reduction (%) of concentrations of Ash, N, S, K, Ca and Cl from biomass DM to press cake DM achieved in Experiment 3.

	SNG [1]					ING [2]				
Parameter	M100 [3]	M75 [4]	M50 [5]	M25 [6]	M0 [7]	M100 [3]	M75 [4]	M50 [5]	M25 [6]	M0 [7]
Ash	3.22	8.91	13.69	17.49	25.60	23.59	23.80	28.12	28.59	29.11
N	20.28	21.42	23.07	24.09	26.80	8.92	13.50	18.90	23.59	28.72
S	37.06	39.93	42.62	45.97	48.85	15.00	15.38	20.89	21.71	26.58
K	46.06	54.85	64.25	74.21	82.76	26.25	38.24	51.48	63.25	77.37
Ca	28.78	29.20	32.28	32.31	34.87	7.57	16.25	17.96	23.55	23.47
Cl	50.85	59.65	68.00	78.15	84.15	24.57	39.48	55.74	69.58	84.22

[1,2] semi-natural and intensive grassland silage, respectively; [3–7] leached with liquids with varying shares of recycled press liquid (M100/75/50/25/0 = 100%/75%/50%/25%/0% press liquid, respectively).

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