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Coupled Biogas and Fiber Production from Agricultural Residues and Energy Crops with Steam Explosion Treatment

Benedikt Hülsemann [1,*], Marian Baumgart [1], Leonhard Lenz [1], Elviliana [2], Marie Föllmer [3], Gregor Sailer [1], Konstantin Dinkler [1] and Hans Oechsner [1]

[1] State Institute of Agricultural Engineering and Bioenergy, University of Hohenheim, Garbenstraße 9, 70599 Stuttgart, Germany; leonhard.lenz@uni-hohenheim.de (L.L.)
[2] Department of Agro-Industrial Technology, Faculty of Agricultural Technology, Universitas Brawijaya, Malang 65145, Indonesia
[3] Hochschule der Medien, University of Applied Sciences, Nobelstraße 10, 70569 Stuttgart, Germany
* Correspondence: benedikt.huelsemann@uni-hohenheim.de

Abstract: The global demand for packaging materials and energy is constantly increasing, requiring the exploration of new concepts. In this work, we presented a bioeconomic concept that uses steam explosion and phase separation to simultaneously generate fibers for the packaging industry and biogas substrate for the energy sector. The concept focused on fiber-rich residues and fiber-rich ecological energy crops from agriculture. Feasibility of the concept in the laboratory using feedstocks, including Sylvatic silphia silage, Nettle silage, Miscanthus, Apple pomace, Alfalfa stalks, and Flax shives was confirmed. Our results showed that we were able to separate up to 26.2% of the methane potential while always extracting a smaller percentage of up to 17.3% of organic dry matter (ODM). Specific methane yields of 297–486 L_{CH4} $kg_{ODM}{}^{-1}$ in the liquid and 100–286 L_{CH4} $kg_{ODM}{}^{-1}$ in the solid phase were obtained. The solid phases had high water absorption capacities of 216–504% due to the steam explosion, while the particle size was not significantly affected. The concept showed high potential, especially for undried feedstock.

Keywords: renewable energy; bioeconomy; biowaste; residuals; silage; liquid–solid separation

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1. Introduction

The United Nations Development Programme identified 17 Sustainable Development Goals (SDGs) that outline a blueprint for a sustainable future. Five of these SDGs are particularly relevant to agriculture and biogas production, namely Zero Hunger, Affordable and Clean Energy, Industry, Innovation and Infrastructure and Climate Action [1]. Biogas is a renewable energy carrier produced through the anaerobic digestion of organic matter. The biogas technology was proven to be relevant for the reduction in greenhouse gases while simultaneously producing clean energy. Despite its potential, fossil fuels still account to approximately 79% of the global energy consumption [2].

Germany is one of the worldwide market leader for biogas and the sector grew over the last twenty years to roughly 9600 plants in Germany today [3]. The number of biogas plants was growing mainly because the Renewable Energy Sources Act (EEG), in its 2004 and 2009 versions, guaranteed high remunerations for electricity for twenty years [4]. Biogas plants in Germany generated an average electricity revenue of 23.4 EUR -ct/kWhel [5]. However, operation without high legal remuneration is not economically viable. The current version of the EEG (2023) limits the guaranteed remuneration for electricity in a tender procedure to a maximum of 18.03 EUR -ct/kWh for existing biogas plants and 16.07 EUR -ct/kWh for new biogas plants [6]. At the same time, the production costs are 18.9 EUR -ct/kWhel in Germany [5] and the cost increased according to higher costs expected due to stricter regulations, higher feedstock prices, and the advanced age of existing plants. Therefore, operation modes based on only continuous energy generation is not economically feasible for future business models of biogas plants [5].

Alternative products such as the electricity generation on demand, heat sale, production of biomethane or chemicals as well as the generation of fibers must be considered.

Another approach is the reduction in production costs through the use of other energy crops and residues that are cheaper and more ecological than the commonly used maize silage (70% of the energy crops used in German biogas plants [7]).

The use of alternative energy crops that can grow on marginal land also has a positive impact on public acceptance of the biogas technology because these energy crops are not in direct conflict with food production and are characterized with lower CO_2 emission [4,8,9]. Nevertheless, most alternative feedstocks have lower methane potentials and slower anaerobic digestion rates compared to maize silage due to their high fiber content, which has high resistance to microbes during anaerobic digestion [10]. For this reason, the use of these feedstocks is still more expensive than the use of maize silage.

To solve this problem of expensive biogas plant feedstocks, a bioeconomy concept was developed in which energy crops and residual materials are thermo-chemically pretreated via steam explosion (SE) followed by a solid–liquid separation. The solid fraction mainly consists of fibers (hardly digestible in the biogas process), while the liquid fraction contains components that are rapidly and easily degradable in an anaerobic digestion process. Thus, the liquid will be used in a biogas plant for electricity and heat production, while the solid fraction will be used as fiber material in various processes, e.g., for packaging material production.

Lignocellulose is the most abundant organic biomass in the world and an important feedstock for bioenergy technologies, biodegradable materials, and bio-based chemicals in biorefineries [11]. Its complex structure comprises hemicellulose linked cellulose microfibrils embedded in a matrix of lignin, cross-linked polysaccharide networks, and glycosylated proteins [12]. However, the resistance of lignocellulosic substrates to biological degradation in anaerobic digestion, known as biomass recalcitrance, hinders the conversion of the structural polysaccharides of the cell wall into fermentable sugars for use as fuel or chemicals [13]. To tackle this issue, various pretreatment methods were developed, including physical, physicochemical, chemical, and biological methods [14]. Among them, SE is a promising physicochemical approach that simultaneously modifies the biomass chemically, fractures the cell wall, removes hemicellulose, and increases the accessible surface area of cellulose without significant cellulose degradation [15]. Additionally, SE is effective in autocatalytically removing acetic- and uronic-groups forming their respective acids and depolymerizing hemicellulose, making it an attractive method for pretreating lignocellulosic materials [16,17]. SE is usually operated at a temperature of 160–220 °C with a pressure of 0.6–1.0 MPa. Boiling and rapid depressurization break down the lignin structure and degrade the hemicellulose to oligomers and sugars. Pressure around 0.5 MPa depolymerize the hemicellulose but not the cellulose [15,18,19]. Furthermore, after SE, a separation of a liquid and solid phase is easily possible. The pretreatment of fiber-rich substrate to increase biogas production was already investigated in several studies [20–22].

The fibers produced as part of the bioeconomic concept can be used to produce several different products such as paper, packaging, or flower pots. Because of this, the fibers can close the production cycle for these products locally and significantly reduce transport distances and, thus, CO_2 emissions, as they replace wood from *Eucalyptus* spp. or *Pinus* spp. in Germany, which is mainly produced in South America and especially in Brazil [23]. In the case of flower or plant pots, conventional plastic- or peat-based products can be substituted [24]. The production of paper and packaging also holds great potential. The growing popularity of e-commerce led to an increased demand for packaging materials, exacerbating the environmental impact of the packaging industry in terms of CO_2 emissions and energy consumption [25–27]. Kim et al. (2022) reported that due to increasing online trade, the amount of packaging in Germany is 4.8 times higher compared to offline trade and Järvinen et al. (2020) predicted that the demand for paper will almost be doubled by 2050 [28,29].

This study investigates the suitability of various residual materials and ecological energy crops with variable properties (e.g., dry matter (DM)) for the use in the bioeconomy concept with coupled fiber and biogas production by processing the fiber-rich biomass in a SE followed by solid–liquid separation (Figure 1). To investigate the potential of the concept, a wide range of residual materials and ecological energy crops were investigated. The following crops were considered: the silage of the whole plant of *Silphium perfoliatum* L. (sylvatic silphia silage), *Urtica dioica* (nettle silage), and *Miscanthus sinensis* (miscanthus whole plant), as well as the hop grubbing chaff of *Humulus lupulus* (hop bine chaff), the straw of *Miscanthus sinensis* (miscanthus straw), the stalks of *Medicago sativa* (alfalfa stalks), the shives of *Linum usitatissimum* (flax shives), and the pomace of *Malus* (apple pomace). The aim was to evaluate the developed bioeconomy concept and to check what kind of substrate can be used for this concept.

Figure 1. The investigated feedstocks sylvatic silphia silage (**a**), nettle silage (**b**), hop grubbing chaff (**c**), miscanthus whole plant (**d**), miscanthus straw (**e**), apple pomace (**f**), alfalfa stalk (**g**), flax shives (**h**).

2. Materials and Methods

2.1. Substrates and Sampling

Silvatic silphy silage was taken from stored silage (Ostrach, Germany) on 3 September 2021 (see Figure 1). Nettle silage (Ostrach, Germany) was harvested, pressed, and stored as silo bales on 6 September 2021. The silo was open on the day of further treatment steps. Hop grubbing chaff was taken from stored silage (Hallertau, Germany) in January 2021. Miscanthus was harvested as whole plant on 20 October 2021, and as straw on 2 February 2021 (Unterer Lindenhof, Eningen unter Achalm, Germany). Both were chopped before being treated with SE. Apple pomace was taken from juice extractor (Kelterei Widemann, Bermatingen, Germany). Alfalfa stalks were harvested in September 2021 (Futtertrocknung Lamerdingen eG, Lamerdingen, Germany). Flax was harvested at different locations in France. The flax shives were sorted during the production process of flax and were then sent to the authors (Terre de Lin, Saint-Pierre-le-Viger, France). All fresh samples were immediately after harvesting stored in compressed condition in 20 L barrels and at 4 °C for several days prior to testing. Compression was performed by hand.

2.2. Steam Explosion (SE)

All substrates were pretreated using the SE unit at the Department of Yeast Genetics and Fermentation Technology of Hohenheim University (Stuttgart, Germany). The treatment was carried out in a gastight and double-walled laboratory reactor with a volume of 20 L (H & K GmbH Behälter und Edelstahltechnik, Kehl, Germany). The steam was supplied indirectly by heating the substrate–water mixture.

Before SE, the samples were crushed by blades in a Thermomix (Vorwerk, Wuppertal, Germany) for 30 s and mixed with water to obtain a similar water content (Table 1). SE was performed in a gastight and double-walled laboratory reactor with a volume of 20 L (H & K GmbH Behälter und Edelstahltechnik, Kehl, Germany) at 160 °C and 0.5 MPa (see Figure 2). The reactor was heated by steam. A change in temperature and pressure was not possible. All reaction times were set to 10 min, resulting in a severity factor of 2.77, excluding the alfalfa stalks (30 min with a severity factor of 3.54) [30–32].

Table 1. Substrate-to-water ratio for steam explosion and overall water content of different trails.

Sample	Substrate: Water Ratio Substrate: Fresh Water	Water Content %
Sylvatic silpiha silage	3:2	85.8
Nettle silage	2:1	79.7
Hop grubbing chaff	3:2	84.9
Miscanthus whole plant	3:2	74.5
Miscanthus straw	1:2	75.1
Apple pomace	3:1	85.3
Alfalfa stalks	2:1	65.8
Flax shives	1:2	71.1

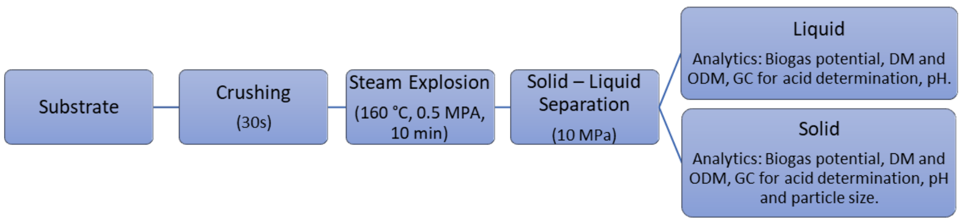

Figure 2. Overall flow chart of methodology used.

2.3. Solid–Liquid Separation

Solid–liquid separation was carried out using a DPH2/5 hydraulic tincture press (Doninger, Achern, Germany) with a sample volume of 2 L under a pressure of 10 MPa. The separated solid and liquid phases were measured using a Kern PCB precision scale (Kern & Sohn GmbH, Balingen, Germany). The separation process was performed in triplicate for all tests. Untreated flax shives were soaked in water for 24 h prior to the separation process, to ensure sufficient moisture content for successful separation, as low water content would hinder the separation process.

2.4. Biogas Potential Determination

The biogas potential of the liquid and solid phase was determined by Hohenheim biogas yield test (HBT). The HBT is a batch-test performed in 100 mL syringes. These syringes are closed gastight through a hose clamp and silicone. Each syringe was stored in a motor-driven plate to ensure mixing. This plate was stored in a heating cabinet (Memmert, Schwabach, Germany) at 37 °C for 35 days.

The test was carried out with an organic dry matter (ODM)-inoculum-to-substrate ratio of 2.25 and the whole procedure was executed according to VDI 4630 [33]. The substrate was utilized without undergoing additional crushing. The inoculum was taken from a 400 L laboratory reactor that was fed continuously with a broad spectrum of nutrients with a low organic loading rate, to obtain a low specific methane yield of the inoculum. The inoculum was also fermented alone and its methane yield subtracted from the total methane formation. A positive control was performed with concentrated feed and hay as standard feedstock. Further information on the method can be found in the literature [34–36].

2.5. Analytic Parameters Determined for Solid and Liquid Fractions

DM and ODM were measured according to DIN EN 12880 and DIN EN 12879. A DM/ODM correction (with regard to results of the HBT experiments) was performed for all samples according to Weißbach et al. [37,38]. The organic acids were determined by a gas chromatograph GC 2010plus with an AOC-20i autoinjector (Shmiadzu, Jyoto, Japan). The alcohols were determined by Rio detector, the column BioRad Aminex (Hercules, CA, USA), HPLC column HPX-87H (7.8 × 300 mm; part. Size: 5.0 µm) (Sigma-Aldrich, St. Louis, MO, USA), and BioRad–pre-column HPX-87H (Hercules, CA, USA). The concentration of the volatile fatty acids, acetic acid, propionic acid, iso-butyric acid, n-butyric acid, iso-valeric acid, n-valeric acid, and caproic acid was summed up as one cumulative parameter (SUM-VFA). The pH value was determined using a pH meter Type 211 (HANNA Instruments, Woonsocket, RI, USA). All chemical measurements were carried out at least in duplicate.

Due to the low number of replicates, no statistical analysis was performed.

2.6. Characterization of Solid-Phase Specific Properties

The particle size of the solid fraction after SE was determined in triplicate with Analysette 3 SPARTAN (Fritsch GmbH, Idar-Oberstein, Germany). Eight sieves according to ISO 3310-1 (diameter 200 mm, height 50 mm) were used. The mesh sizes used were 0.063 mm, 0.125 mm, 0.25 mm, 0.5 mm, 1 mm, 2 mm, 4 mm, and 8 mm.

Furthermore, water adsorption capacity of the solid fraction after separation was analyzed according to DIN 53923. The samples were first dried at 105 °C for 24 h. Afterwards, 5 g_{DM} of the samples were distributed on a mesh size 0.063 mm sieve (Retsch, Haan, Germany) and placed in a water bath for 6 s at a depth of 20 mm with a temperature of 20 ± 1 °C. To avoid air bubbles, the sieve was inserted at an angle of around 20°. Finally, the sieve was submerged for 120 s and weighed.

3. Results and Discussion

3.1. Mass Balance

The origin and nature of the feedstocks used in this study varied, which resulted in a significant difference in the DM content of the substrates before solid–liquid separation, ranging between 19.6 and 86.8%, as shown in Table 2. However, the DM content of the solid phases after SE and tincture press showed only minor variations, with a value range between 42.6 and 50.0% for most of the substrates investigated. Only the solid phase of apple pomace exhibited a DM content of 37.1%. Zhao et al. (2022) showed that water holding capacity of apple pomace with SE pretreatment is high due to its stable hydrogen bonds [39]. This could explain the lower DM content observed in the solid fraction of apple pomace. Additionally, apple pomace contains a high amount of dietary fiber, e.g., lignocellulosic compounds, which complicate solid/liquid separation [40].

In contrast to the solid phase, the liquid phase of the different biomasses after SE and tincture press showed a huge variation between DM contents (3.5–10.6%), which was due to both the DM variation of the substrate and the changing water to substrate ratio before SE. The DM contents of liquid phases were in the range of manure, and, from a technical perspective, the liquid phases can be considered pumpable with auxiliaries present at biogas plants [41,42]. Nevertheless, low DM concentration of the liquid phase would require a large reactor volume compared to an energy crop fed biogas plant with identic hydraulic retention time and methane production, which increases investment and operating costs. Therefore, dilution should be as low as possible to ensure the function of SE [43] and reduce the water input. Further research to optimize this parameter would be useful.

Table 2. Distribution of ODM in the respective phases after SE, dry matter (DM) and organic dry matter (ODM) of input substrates prior and solid and liquid fractions after steam explosion with solid–liquid separation.

Substrate	Phase	Distribution of ODM after Phase Separation	DM	ODM
		%	%	$\%_{DM}$
Sylvatic silphia silage	Substrate		23.7 ± 0.1	88.4 ± 0.1
	Solid	86.2	47.1 ± 0.9	92.4 ±0.0
	Liquid	13.8	6.0 ± 0.1	58.9 ± 0.1
Settle silage	Substrate		30.5 ± 0.2	79.2 ± 0.0
	Solid	86.9	46.6 ± 1.2	90.3 ±0.4
	Liquid	13.1	7.4 ± 0.5	74.2 ± 0.5
Hop grubbing chaff	Substrate		25.2 ± 1.1	80.1 ± 5.5
	Solid	82.7	48.0 ± 2.6	89.8 ± 1.3
	Liquid	17.3	10.1 ± 0.5	81.8 ± 0.2
Miscanthus whole plant	Substrate		42.6 ± 0.1	96.3 ± 0.1
	Solid	93.9	42.6 ± 1.6	96.1 ± 0.2
	Liquid	6.1	4.5 ± 0.3	77.6 ± 1.3
Miscanthus straw	Substrate		74.6 ± 0.4	96.1 ± 0.1
	Solid	97.3	47.6 ± 0.0	97.1 ± 0.1
	Liquid	2.7	3.5 ± 0.0	77.7 ± 0.5
Apple pomace	Substrate		19.6 ± 0.1	97.8 ± 0.0
	Solid	92.1	37.1 ± 0.4	99.1 ± 0.1
	Liquid	7.9	7.7 ± 0.0	87.9 ± 1.1
aAfalfa stalk	Substrate		51.3 ± 0.8	92.5 ± 0.7
	Solid	88.8	49.8 ± 1.0	95.0 ± 0.2
	Liquid	11.2	11.0 ± 0.1	76.8 ± 0.4
Flax shives	Substrate		86.8 ± 0.7	84.5 ± 1.9
	Solid	95.3	50.0 ± 2.8	96.7 ± 0.9
	Liquid	4.7	5.4 ± 0.0	90.0 ± 0.4

Regarding the distribution of ODM, the majority remained in the solid phase after SE and separation, with a range of 82.7% to 97.2%. Only a small proportion of ODM migrated into the liquid phase, ranging from 2.7% to 17.3%. (Table 2, Figure 3). This proportion was independent from the added water in the observed range of substrate/water ratios. The bioeconomic concept generates new revenue through the recovery of fibers. However, substantial potential is lost for biogas production or as fertilizer if the products are not returned to the field at the end of the lifecycle. In terms of nutrients, it is advantageous to cycle them back to the fields in the form of digestate. Other compounds such as sand or heavy metals could also be accumulated in the liquid phase [44,45]. This may cause problems such as induced phytogenic and/or genotoxic effects in crops and potential health risks for humans and should be further investigated [44,45].

Comparing the ODM ratio of the liquid phase with the fiber (acid detergent lignin (ADL) and neutral detergent fiber (NDF)) content of the feedstock, it was noticeable that a high content of these substances seemed to be associated with a low ODM ratio of the liquid (Figure 4). High fiber content as well as high ADL and NDF content are expected in straw and other late-harvested materials.

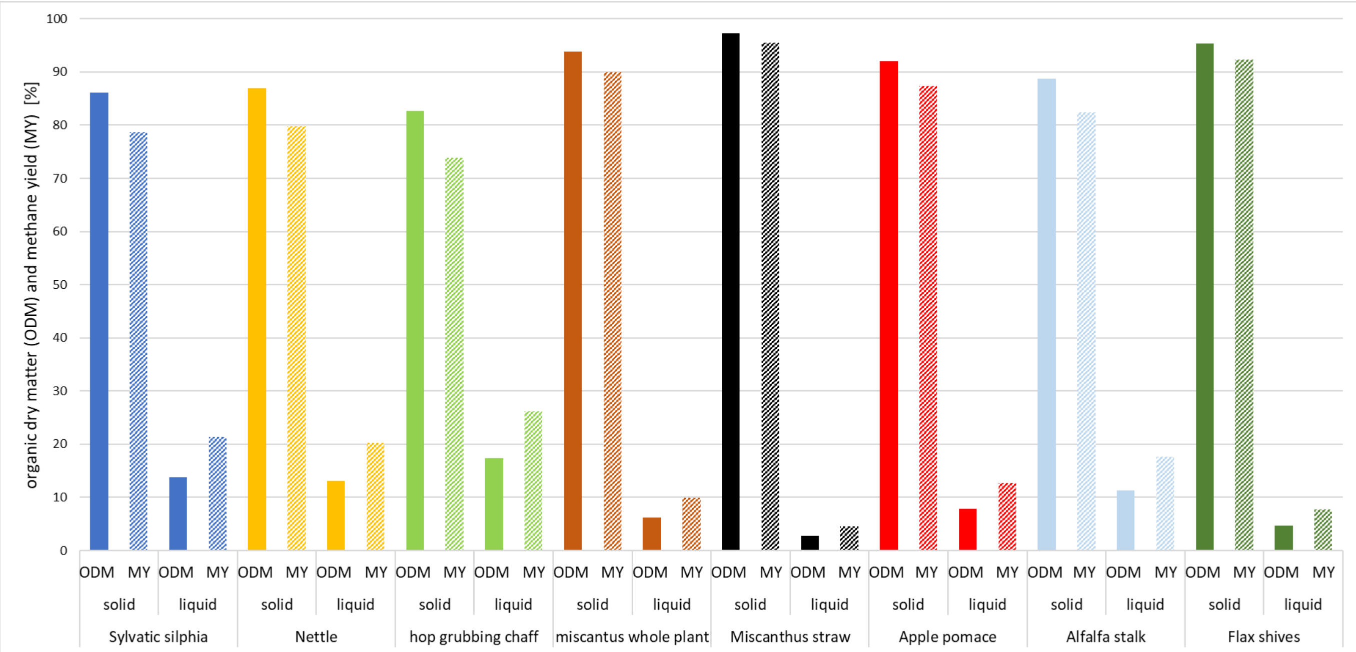

Figure 3. Proportion of organic dry matter (ODM) and methane yield (MY) portion after SE in solid and liquid phase for all investigated substrates.

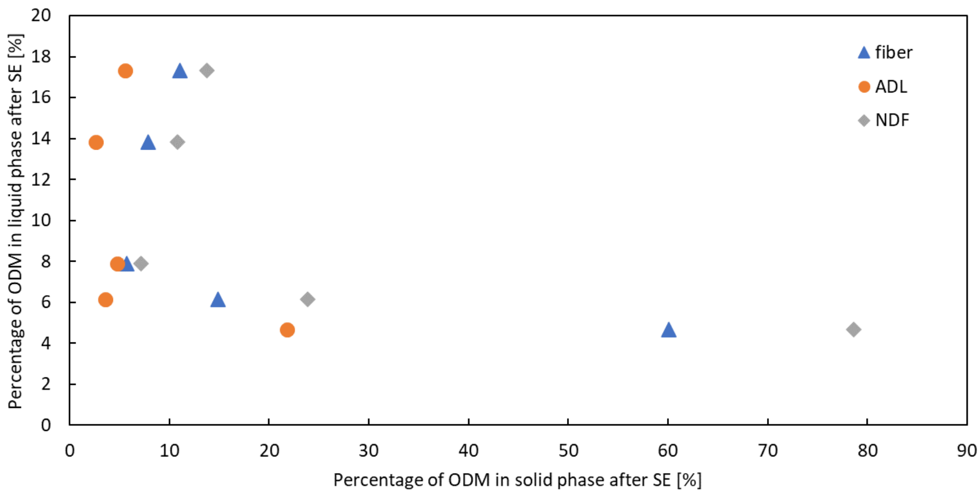

Figure 4. Percentage of organic dry matter (ODM) in liquid phase respective to ODM in the solid fraction (acid detergent lignin (ADL) and neutral detergent fiber (NDF)) after SE.

3.2. Energy Balance

Specific methane yield (SMY) based on ODM and FM were calculated both for solid and liquid phases, as presented in Table 3. The SMY of the solid phases were in the range of 100–287 L_{CH4} $kg_{ODM}{}^{-1}$ and the SMY of the liquid phases were in the range of 297–486 L_{CH4} $kg_{ODM}{}^{-1}$. Based on the results and the kinetics, no inhibition in anaerobic digestion was to be expected.

The SMY of the liquid phases after SE and tincture press were in range or even higher than the SMY of maize silage (355 L $kg_{oTS}{}^{-1}$ [46]). This suggests that there were less anaerobically hard-to-digest or non-digestible components such as fibers in the liquid. The aim of the bioeconomic concept, which is to separate easily degradable cell contents for the biogas process and upcycle poorly degradable fiber constituents, was thus achieved. Furthermore, SE leads to hydrolysis of the substrate, which results in acid generation and, therefore, a reduction in the pH value especially in the liquid phase (Table 4) [17].

High concentrations of SUM-VFAs found in the liquid phase of this study supported this observation (Table 4). Taking into account the mass dependent methane yield, it is observable that the methane yield of the liquid fraction (10–33 L $kg_{FM}{}^{-1}$; Table 3) was much lower than that of maize silage (118 L $kg_{FM}{}^{-1}$) but in the range of cow manure (17 L $kg_{FM}{}^{-1}$) [46]. This suggests an economic use of the liquid phase in a biogas plant, but transport distances should be kept short due to the highwater content. Recirculating the liquid phase instead of adding fresh water could reduce production costs and increase methane yield by enriching the liquid with organics in each circulation step. It is also useful to reduce the required tank volume of the biogas plant through an optimized plant design. Nevertheless, the addition of water is necessary and needs to be adjusted optimally because the moisture content ratio increases the efficiency of SE pretreatment by increasing the mechanical force produced by the expanding gas (water vapor) [31,47].

Table 3. Specific methane yield (SMY) in solid–liquid fractions of the substrates examined in this study. SMY based on organic dry matter (ODM) and fresh matter (FM). Proportion of Methane yield in solid and liquid phase.

Substrate	Phase	SMY	Methane Yield	Proportion of Methane Yield
		L_{CH4} $kg_{ODM}{}^{-1}$	L_{CH4} $kg_{FM}{}^{-1}$	%
Sylvatic silphia silage	Solid	208 ± 6	91 ± 3	78.6
	Liquid	340 ± 10	12 ± 0	21.4
Nettle silage	Solid	217 ± 20	91 ± 8	79.7
	Liquid	334 ± 70	18 ± 4	20.3
Hop grubbing chaff	Solid	179 ± 24	77 ± 10	73.8
	Liquid	336 ± 2	28 ± 0	26.2
Miscanthus whole plant	Solid	280 ± 25	115 ± 10	90.0
	Liquid	391 ± 64	14 ± 2	10.0
Miscanthus straw	Solid	192 ± 10	89 ± 5	95.5
	Liquid	354 ± 4	10 ± 0	4.5
Apple pomace	Solid	287 ± 17	106 ± 6	87.3
	Liquid	486 ± 53	33 ± 4	12.7
Alfalfa stalk	Solid	184 ± 3	85 ± 1	82.3
	Liquid	344 ± 23	28 ± 2	17.7
Flax shives	Solid	100 ± 8	48 ± 4	92.3
	Liquid	297 ± 5	14 ± 0	7.7

As expected, the SMY of the solid phases were lower than that of the liquid phases and, e.g., for sylvatic silphia silage, also lower than the expected methane yield of the substrates according to KTBL (2021) [46]. These results highlight the effective separation of the fiber into the solid phase. Nonetheless, the methane yield of the solid phase (100–287 L $kg_{FM}{}^{-1}$; Table 3) was still high and, for example, in the range of stored solid cow manure (180 L $kg_{oTS}{}^{-1}$ [46]). Based on fresh matter, the SMY of the solid phases were even higher than the literature value of grass silage, a commonly used substrate in German biogas plants (81 L $kg_{FM}{}^{-1}$ [46]). It can be assumed that the solid phase contained other components besides the fibers, which have to be washed out at great expense and are of no benefit to the industry. This assumption is supported by the results of the acid concentrations and the Weender-van-Soest analysis, revealing the presence of SUM-VFA (Table 4), raw protein, and raw fat (1.0–1.9% and 0.5–1.2%) in all samples.

Table 4. pH, volatile fatty acids (SUM-VFA), acetic acid, and butyric acid concentration of solid and liquid phase after SE and separation; standard deviation of all values are <0.0 g kg^{-1}.

Substrate	Phase	pH	SUM-VFA g $kg_{FM}{}^{-1}$	Acetic Acid g $kg_{FM}{}^{-1}$	Butyric Acid g $kg_{FM}{}^{-1}$
Sylvatic silphia silage	solid	5.7	10.6	5.4	0.2
	liquid	5.3	25.0	13.9	0.5
Nettle silage	solid	5.2	7.7	5.7	2.1
	liquid	5.0	16.0	12.2	4.1
Hop grubbing chaff	solid	5.2	16.4	8.6	3.0
	liquid	5.2	29.9	17.4	5.0
Miscanthus whole plant	solid	4.8	1.1	1.0	0.1
	liquid	4.3	4.8	4.8	0.1
Miscanthus straw	solid	6.0	0.4	0.4	0.0
	liquid	5.2	3.2	3.1	0.1
Apple pomace	solid	3.7	1.2	1.1	0.2
	liquid	3.5	1.8	1.6	0.2
Alfalfa stalk	solid	6.0	1.6	1.3	0.3
	liquid	5.0	6.2	5.8	0.4
Flax shives	solid	4.3	3.1	2.9	0.2
	liquid	4.2	7.7	7.4	0.4

The comparison of the methane yield ratio of solid–liquid phase underlines the fact that a high methane yield is not fully exploited, if the solids are not digested (Figure 3). In any case investigated in this study, more than 70% of the methane yield was contained in the solid phase and was, therefore, lost for energy production in the bioeconomy concept. For each kg of substrate, only a maximum of 4.8 L_{CH4} $kg_{FM,substrate}{}^{-1}$ was produced, revealing that biogas and energy production were only by-products and the main income must be generated by the fiber utilization. Alternatively, further optimization of the bioeconomy concept may be necessary.

It is important to note that butyric acid has an unpleasant odor for humans and could decrease the quality of the fiber by limiting its potential applications, such as in packaging materials, especially for food, as butyric acid is already detectable at concentrations of 0.06 mg m^{-3}. Even the low concentration of up to 3.0 g $kg_{FM}{}^{-1}$ can, therefore, produce a bad smell, as observed for sylvatic silphia silage, nettle silage, and hop grubbing chaff in this study (Table 4). Based on the data, it was not possible to observe when the butyric acid is generated. Therefore, further research is needed to investigate the inhibition of butyric acid production or the reduction in its concentration especially in the solid phase through optimization of storage, such as in silage, SE parameters, and fiber washing.

The methane yield ratio of the liquid phase was found to be particularly low for flax shives and miscanthus straw, which were dry substrates, with values below 10%, resulting in a maximum production of only 0.7–0.8 L_{CH4} per kg of feedstock. Based on this, it makes sense to harvest miscanthus earlier with higher content of separable cell contents, to get a better separation and to achieve a higher SMY of the liquid phase. According to Tarabanko et al., (2022), flax shives have a similar structure to lignin-rich soft woods [48], in which lignin accounting for up to 25% of the total lignocellulose compound [49,50].

In contrast, sylvatic silphia silage, nettle silage, hop rubbing chaff, and alfalfa stalks (wet substrates) exhibited the best separation of the biogas potential observed in this study, resulting in the highest methane yields in the liquid phases per each kg of feedstock (2–5 L_{CH4} $kg_{FM,substrate}{}^{-1}$). This observation is consistent with the finding that these substrates also had the highest SUM-VFA concentrations (Table 4). Despite the possibility of inhibition due to the high SUM-VFA concentration, it was not observed during the experiments because the inoculum used had a high buffer capacity [51–55].

By comparing the combined methane yield per kg of fresh matter of both liquid and solid fractions after SE and separation with the methane yield reported in the literature for the substrates, it was found that the methane yields for sylvatic silphia silage (80 to 59 L_{CH4} $kg_{FM}{}^{-1}$ [46]) and hop grubbing chaff (69 to 46 L_{CH4} $kg_{FM}{}^{-1}$ [56]) increased by 36–50% after SE. In a recent study, the utilization of lignin-rich macrophytes by semi-continuous anaerobic digestion with SE pretreatment at a severity factor of 4.4, and, therefore, a bit higher value than used in this study, showed an increase in methane yield of up to 90% [57]. Overall, it can be observed that the proportion of methane yield in the solid fraction was always lower than the proportion of ODM and, therefore, vice versa in the liquid fraction (Figure 3).

In order to achieve a higher methane yield and fiber quality additional pretreatment, separation or anaerobic digestion seems to be reasonable when applying the bioeconomy concept. Furthermore, attention should be given to SE operation conditions, as they were not modified or optimized in this work.

Due to the high SUM-VFA concentration in the liquid fraction, it could be interesting to use this fraction to obtain other value-added products before the energy production [58].

3.3. Characterization of Solid Fraction

The range of water absorption capacities observed for the substrates varied from 216% to 504%. In addition, the calculated average particle sizes for the substrates were found to range from 388 µm to 1857 µm (Table 5).

Table 5. Water absorption capacity and average particle size of investigated substrates.

Substrate	Water Absorption Capacity $\%_{FM}$	Average Particle Size µm
Sylvatic silphia silage	340	1575
Nettle silage	440	1857
Hop grubbing chaff	409	1540
Miscanthus whole plant	401	748
Miscanthus straw	417	756
Apple pomace	216	388
Alfalfa stalk	318	1207
Flax shives	504	520

Both the water capacity and particle size were the lowest for apple pomace. The highest water absorption capacity was observed for flax shives. When the fibers are used in the paper or packaging industry, they are ground by mills to cut the fibers. Low fiber length, as apple pomace fibers in this study, can reduce the additional energy required. Similarly, a low water absorption capacity can reduce the cost of subsequent drying of the paper/packaging material [59]. However, for apple pomace, the dry matter content after separation was the lowest like explained before. The high values of flax shives probably occurred by a multi-porous structure and the high fiber content [60].

The average particle size and water adsorption capacity did not exhibit a dependence. The same behavior was observable on a study on hammer milled palm lignocellulosic by-products [61]. In contrast to the referenced particles (water absorption between 100 and 300%), that were not pretreated with SE, it is observable that the SE pretreated particles showed a higher water absorption captivity with 216–504%. No reason could be found so far.

Particle size was not significantly affected by SE pretreatment for all substrates, as shown in Figure 5 in case of miscanthus (whole plant), because of missing mechanical treatment during the SE pretreatment. According to this, the particle size depends only on the cutting length during harvesting. Particle size should be chosen to optimize storage and SE treatment. The impact on both needs to be considered in future experiments. In the case of SE, several studies suggest large particles for high glucose and xylose concentration. Small particles do not optimize the degradation according to their research [62,63].

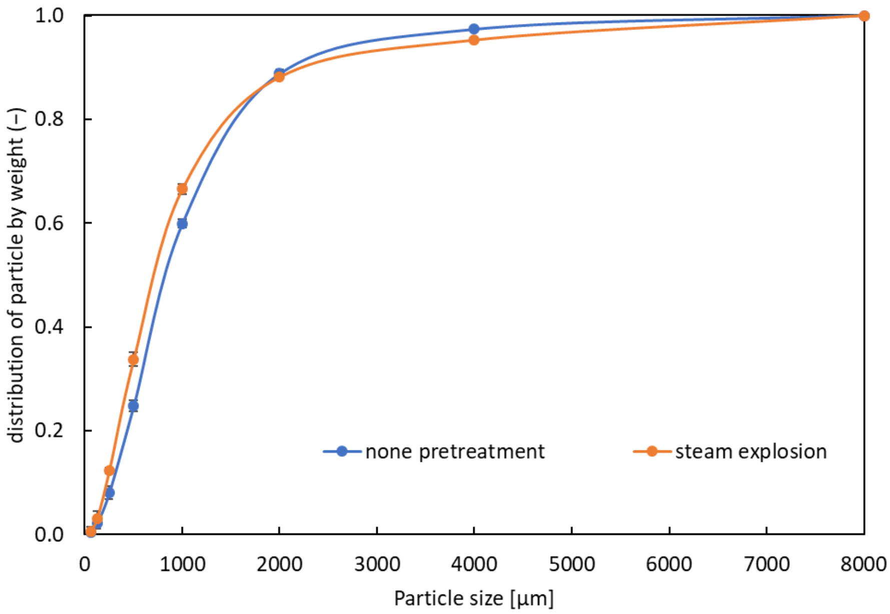

Figure 5. Sum curve of particle size distribution by weight of none pretreated and SE pretreated miscanthus whole plant.

4. Conclusions

In this study, we presented a bioeconomic concept for lignocellulosic feedstock that combined SE, solid–liquid separation, and anaerobic digestion of the liquid fraction. Our results demonstrated that this process effectively separated the easily anaerobically digestible fractions of ODM into a liquid phase while leaving behind fibers with high water adsorption capacity, resulting in a significant separation of SMY. The concept showed particularly high potential for undried feedstocks, with more methane yield being recovered in the liquid phase of undried substrates than for dried ones. However, butyric acid was detected in some of the samples, with levels of up to 3.0 g/kg in the solid phase. This is a major problem for the bioeconomic concept, as it reduces the application range of the fibers. Further investigation into the silage conditions is needed to address this issue. To further develop the bioeconomic concept, it is necessary to optimize the SE operating parameters, the anaerobic digestion process, and search for other biomass types such as biowaste to evaluate the influences on the separation of methane yield and fiber quality, as well as the economic aspects.

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