fermentation

MDPI

Article

Characteristics and Anaerobic Co-Digestion of Press Water from Wood Fuel Preparation and Digested Sewage Sludge

Gregor Sailer [1,*,†], Florian Empl [1,†], Daniel Kuptz [2,†], Martin Silberhorn [1], Darwin Ludewig [1], Simon Lesche [2], Stefan Pelz [1] and Joachim Müller [3]

[1] Department of Bioenergy, University of Applied Forest Sciences Rottenburg, Schadenweilerhof, 72108 Rottenburg, Germany; empl@hs-rottenburg.de (F.E.); silberhorn@hs-rottenburg.de (M.S.); ludewig@hs-rottenburg.de (D.L.); pelz@hs-rottenburg.de (S.P.)

[2] Technology and Support Centre in the Centre of Excellence for Renewable Resources (TFZ), Department of Solid Biofuels, Schulgasse 18, 94315 Straubing, Germany; daniel.kuptz@tfz.bayern.de (D.K.); simon.lesche@tfz.bayern.de (S.L.)

[3] Tropics and Subtropics Group, Institute of Agricultural Engineering, University of Hohenheim, Garbenstrasse 9, 70599 Stuttgart, Germany; joachim.mueller@uni-hohenheim.de

* Correspondence: sailer@hs-rottenburg.de

† These authors contributed equally to this work.

Abstract: Technical drying of harvested wood fuels is heat and energy consuming, while natural pre-drying in the forest, e.g., in stacks or storage piles, is accompanied by energy losses through natural degradation processes. Dewatering of energy wood by mechanical pressing is an innovative method to reduce the moisture content prior to thermal drying while producing press waters (PW, also referred to as wood juice) as a by-product. To date, the characteristics and utilization potentials of PW are largely unknown. In this study, three different spruce- and poplar-based PW were analyzed for their characteristics such as dry matter (DM), organic dry matter (oDM) concentration, pH-value, element concentration or chemical compounds. Additionally, they were used for anaerobic digestion (AD) experiments with digested sewage sludge (DSS) serving as inoculum. The fresh matter-based DM concentrations of the PW were between 0.4 and 3.2%, while oDM concentrations were between 87 and 89%$_{DM}$. The spruce-based PW were characterized by lower pH-values of approx. 4.4, while the poplar-based PW was measured at pH 8. In the AD experiments, DSS alone (blank variant) achieved a specific methane yield of 95 ± 26 mL/g$_{oDM}$, while the mixture of spruce-based PW and DSS achieved up to 160 ± 12 mL/g$_{oDM}$, respectively. With further research, PW from wood fuel preparation offer the potential to be a suitable co-substrate or supplement for AD processes.

Keywords: wood juice; by-products from wood processing; mechanical dewatering of wood; spruce and poplar-based liquids; anaerobic digestion; forestry; bio-economy; circular economy

Citation: Sailer, G.; Empl, F.; Kuptz, D.; Silberhorn, M.; Ludewig, D.; Lesche, S.; Pelz, S.; Müller, J. Characteristics and Anaerobic Co-Digestion of Press Water from Wood Fuel Preparation and Digested Sewage Sludge. *Fermentation* **2022**, *8*, 37. https://doi.org/10.3390/fermentation8010037

Academic Editors: Eldon R. Rene, Maria Carmen Veiga and Christian Kennes

Received: 30 December 2021
Accepted: 14 January 2022
Published: 17 January 2022

1. Introduction

Wood fuels in their raw state such as log wood, wood chips, saw dust or wood shavings as well as upgraded wood products such as pellets with standardized physico-chemical fuel properties are important feedstocks for the transformation of current energy systems from fossil fuels towards climate-neutral, renewable energies. Thereby, wood fuels have relevant advantages compared to fluctuating renewables such as solar or wind including, e.g., high energy densities, flexible utilization and easy storage. In Germany, wood fuels are especially relevant for heat production. In 2020, approx. 65% of the total renewable and final energy consumption for heating derived from solid biofuels (i.e., 117 TWh, mainly from wood fuels) [1].

Efficient and low emission combustion of wood requires fuels with defined physical and chemical fuel properties that fit to the requirements of the respective combustion units. These requirements differ largely between small-scale appliances such as wood stoves

or private boilers and larger heating plants or combined heat and power (CHP) plants. Recent international standards such as ISO 17225 (Part 1 to 9) [2] give recommendations for defined classifications of solid biofuels including wood pellets, wood briquettes, wood chips and log wood for both small- and industrial-scale use.

The moisture content (M) in wt% on wet basis (w.b.), i.e., the mass fraction of water in fuels, is one of the most relevant fuel parameters determining the quality of wood fuels as it directly affects their storage and combustion behavior [3]. High gaseous and particulate matter emissions can be expected when fuels with unsuitable $M_{w.b.}$ are combusted, especially in small-scale boilers. This is mainly due to incomplete combustion, caused by cooling of the combustion chamber at high $M_{w.b.}$ or by an uncontrolled, fast combustion when the $M_{w.b.}$ is not suitable for the respective stove or boiler [4].

These effects have often been reported in recent studies [5,6]. For instance, Nord-Larsen et al. [7] and Shen et al. [8] observed an increase of particulate matter, organic carbon emissions and of aromatic hydrocarbons while decreasing the overall efficiency during combustion of wood fuels with $M_{w.b.}$ of 27 wt% compared to 5 wt% in small-scale wood burning stoves. Thus, especially for small-scale combustion units, a defined $M_{w.b.}$ is often recommended [3,4].

Freshly harvested wood usually has a high $M_{w.b.}$ of 40 to 55 wt%, exceeding recommendations for $M_{w.b.}$ of most medium- and small-scale combustion units [9]. Thus, drying processes of wood fuels are often relevant processing steps during fuel production. For upgrading wood and other biomasses by drying, current industrial process chains usually rely on thermal drying or storage methods [3,10]. Both approaches entail disadvantages and risks. For conventional drying, including techniques with moving feedstock such as thermal belt dryer, walking floor dryer and drum dryer as well as stationary drying systems such as drying containers, heat (for drying) and electrical energy (for ventilation) are required [11,12]. These processes might somewhat be optimized by using excess heat, e.g., from biogas plants, but still, a relevant input of energy, time and labor is required for fuel production [13,14]. In contrast, drying during storage of the feedstock in piles utilizes heat that is produced through degradation of the biomass by bacteria and fungi [3,15]. This can lead to energy losses in terms of dry matter (DM). For instance, Hofmann et al. [16] reported a decrease of 11.3% for the usable energy content in a spruce wood chip pile with no protective layer during five months of storage [16] due to DM degradation caused by decomposing bacteria strains and fungi [17]. A survey on the forest and wood energy sector in Bavaria revealed an average storage period of 6.4 months, which can therefore lead to severe energy losses [18]. Other negative effects or hazards of microbial activity during storage include the following: (a) possible heating up of the piles sometimes leading to spontaneous self- combustion, (b) damage to health of workers or nearby residents due to released fungal spores in the air, (c) formation of methane in the anaerobic pile center due to insufficient ventilation and (d) space requirements or delays in a time- and cost-critical logistics chain [15,19–22].

To optimize conventional drying processes in terms of throughput rate and energy efficiency as well as drying of comminuted biomass during storage in piles, pre-drying of the feedstock before processing might be an interesting approach [23]. With lowered $M_{w.b.}$ in the initial wood, a faster and less energy-consuming drying to the desired or targeted $M_{w.b.}$ could be performed. Pre-drying can be achieved during storage of uncomminuted wood in the forest before further processing [24,25] or it occurs naturally during delayed processing of wood that accumulates after forest calamities. However, pre-drying might also be achieved by applying innovative and energy efficient process steps. Recently, a mechanical dewatering press at industrial scale was developed by Bohnert Technik GmbH (Seebach, Germany) to actively decrease $M_{w.b.}$ in wood fuels while aiming at the optimization of subsequent technical drying steps with special regard to the thermal energy demand. The press is especially designed to pre-dry wood chips and sawmill by-products. Thereby, this dewatering press produces wood fuels with lowered $M_{w.b.}$ while also generating press waters (PW) as by-products.

In the literature, only few publications were found that deal with mechanical compression drying of biomass, especially with regard to wood fuels [26–30]. Especially, the lower energy demand of the mechanical compression process compared to conventional thermal drying is often mentioned. However, none of the studies dealt with the PW, although it is always generated as a by-product.

Frodeson et al. [30] introduced the possibility of PW utilization as biogas feedstock or, due to the hemicellulose content, as raw material for bioplastics or bio-hydrogen production. The idea of integrating PW into a bio-based circular economy was taken up by Ciesielski et al. [31] and Wilke et al. [32].

In addition to laboratory tests and theoretical application considerations, the available literature particularly agrees with one point, namely that exploitation options for PW must be found [30–32]. Disposal or, if possible, utilization of the by-products from this wood processing technology is an important aspect for its industrial-scale application, as large quantities of PW are produced, which then must be treated or recycled. Therefore, this study aims at a basic characterization of PW from mechanical drying of wood fuels to identify potential areas of application. Different PW produced by a dewatering press were analyzed in detail, focusing on parameters with relevance for disposal or utilization of wastewater such as chemical oxygen demand (COD) or the chemical composition. All samples were analyzed for chemical compounds by using gas chromatography–mass spectrometry (GC–MS). Furthermore, the utilization of wood-based PW in anaerobic digestion (AD), as one potential area of application, was investigated in this study. Therefore, batch experiments with a co-digestion of PW and digested sewage sludge (DSS) were executed. The physico-chemical PW characteristics were then combined with the results of the batch experiments to discuss the application potential in AD.

2. Materials and Methods

2.1. Samples and Sampling Procedure

For mechanical dewatering of wood chips, a specially designed 30 kW roller press (Wood Chip Squeezer, Bohnert Technik GmbH, Seebach, Germany) recently introduced to the market was used. The press breaks up the wood structure of the wood chips by means of pressure exerted on the material by a conveyor chain [33]. The water trapped in the capillaries of the wood is then squeezed out by pressures of up to 40 MPa, and the $M_{w.b.}$ approaches the fiber saturation point, which is about 21–25 wt% depending on the type of wood [3]. After the PW has been pressed out of the wood, it flows through the special geometry of the chain together with fine fiber material via a channel into a transversely mounted vibrating screen. There, the water with the finest solid residues is separated from the coarser particles and fed into a container. The coarser solid residues are manually fed back into the compression process of the dewatering press.

For this study, PW from three different raw materials was obtained from a sawmill, which uses the press on an industrial scale. As raw material, Norway spruce (*Picea abies*) was chosen as it represents the most common tree species in Germany in terms of forest area, which accounted for 25.4% in 2017 [34]. In total, two spruce wood batches consisting of round wood from the Black Forest in Baden-Wuerttemberg (one with low bark content and the other with slightly higher bark content, which was determined by visual inspection) were used to produce PW. In addition, poplar wood from a short rotation coppice in Rhineland-Palatinate (*Populus* ssp., young age with high bark content) was chosen as raw material due to its fast-growing character while being well suited for utilization as energy wood [10]. The plant operator did not provide the exact values with regard to the bark contents of the raw material.

Over the course of the respective compression process, partial amounts of 1 L each were taken from the PW container 20 times per wood assortment, resulting in a composite sample of 20 L for each of the three samples. The PW was then autoclaved in a laboratory autoclave (Systec DX-45, Systec GmbH, Linden, Germany) at 121 °C with a holding time of 15 min and sealed inside the autoclave at a residual temperature of 88 °C. The PW was

then stored in closed glass bottles (Figure 1) in an air-conditioned room (19 °C) for several days until further analyzes and experiments were carried out.

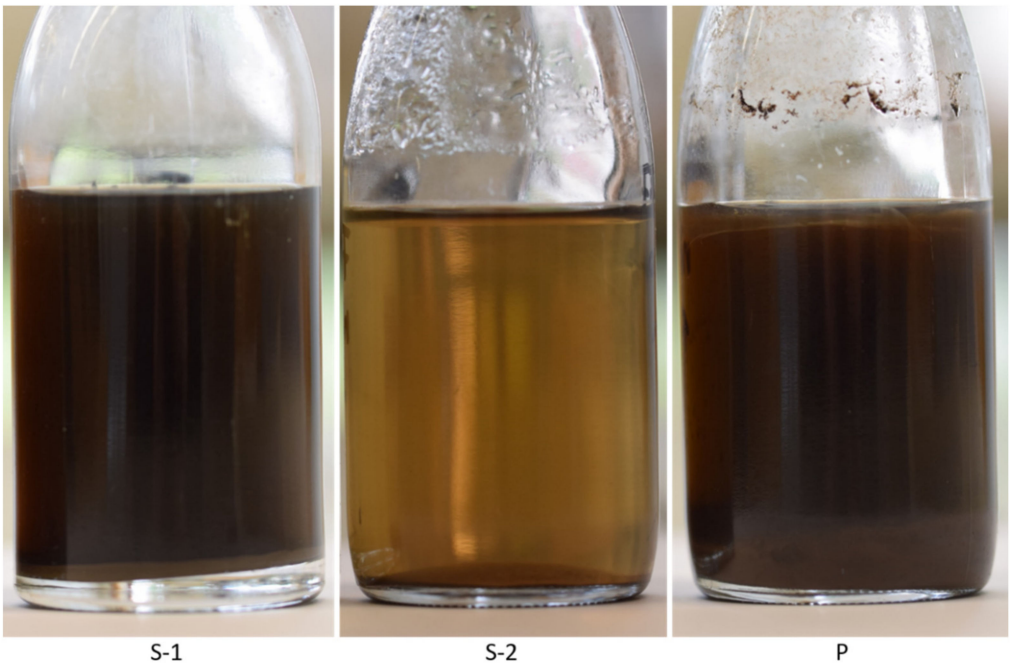

Figure 1. Press waters (PW) produced by the mechanical dewatering press and after autoclaving. Spruce-based PW (S-1 and S-2) and poplar-based PW (P).

During this study, the following sample codes were used for the PW provided through treatment of the raw materials: S-1 for the spruce with higher bark content, S-2 for the spruce with lower bark content and P for the poplar-based PW.

2.2. Dry Matter and Organic Dry Matter Concentration

To determine the DM concentration of the PW, approx. 25 g of fresh matter (FM) of the respective PW was weighed in ceramic crucibles and then dried at 105 °C in a drying oven (UNP 700, Memmert, Schwabach, Germany) with circulating air until the sample mass was stable. The procedure was executed in quadruplicate (n = 4). The crucibles were weighed back after drying, and the DM concentration was calculated based on the determination of $M_{w.b.}$ according to the ISO 18134-2 standard method [35]. The same method was used to determine the $M_{w.b.}$ of the wood chips (>300 g_{FM}) before and after treatment in the mechanical dewatering press.

To determine organic dry matter (oDM) and ash concentration, the crucibles were then heated to 550 °C in a muffle furnace (AAF 1100, Carbolite, Neuhausen, Germany) with a defined heating rate according to ISO 21656 [36]. The oDM concentration was measured in quadruplicate (n = 4).

2.3. pH-Value

The pH values were determined by using a pH meter (FiveEasy FE20, Mettler-Toledo AG, Schwarzenbach, Switzerland) in accordance with DIN 19268 [37]. The pH value determination was carried out once (n = 1) per PW sample.

2.4. Chemical Oxygen Demand

For measurement of the COD, the PW were individually mixed with double-distilled water in a dilution ratio of 1:50. For each sample, 2 mL of the dilution was then injected into a Nanocolor CSB 1500 round cuvette (small scale sealed tube method), containing potassium dichromate ($K_2Cr_2O_7$), and kept at 148 °C for 2 h using a Nanocolor Vario C2 (both Macherey-Nagel, Düren, Germany). The COD was then determined using a

compact photometer (PF-12Plus, Macherey-Nagel, Düren, Germany). This procedure was performed in accordance to ISO 15705 [38]. The COD is one of the decisive prerequisites for discharging wastewater into streams, surface waters or treatment systems. Limit values for direct discharge in surface waters are defined in the German Waste Water Ordinance, for instance [39].

2.5. Elemental Composition

For the determination of the minor and trace element concentration, the PW were analyzed in quadruplicate (n = 4) by inductively coupled plasma-optical emission spectroscopy (ICP-OES). For this purpose, each PW sample was diluted in a ratio of 1:10 with double-distilled water and acidified with 1 mL of nitric acid (HNO_3). Subsequently, they were measured by the ICP-OES system (Spectro Blue ASX-260 with an auto sampler, Spectro Analytical Instruments GmbH, Kleve, Germany). The procedure was carried out according to the standards ISO 11885 and DIN 22022-2, while the calculations were executed based on DIN 22022-6 [40–42].

In addition, a GC–MS analysis was executed with each PW sample (n = 1). For each sample, 10 mL of PW were first stirred with a polydimethylsiloxane (PDMS) Twister (Gerstel GmbH & Co.KG, Mühlheim a. d. R., Germany) for 90 min and then stirred for a further 30 min after the addition of 10 wt% of sodium chloride (NaCl). The PDMS twister was then removed from the vessel, dried and placed in a glass thermal desorption tube (4 mm in diameter and 187 mm in length) and measured by GC–MS. For thermal desorption, a thermal desorber 3.5+-system (Gerstel GmbH & Co.KG, Mühlheim a. d. R., Germany) was used. The thermal desorber unit was mounted on a HP 7890B gas chromatograph coupled to a 5977B mass-selective detector (both Agilent Technologies, Santa Clara, CA, USA) with a cooled injection system (CIS 4) programmable temperature vaporizing (PTV) inlet. The PTV was programmed to inject the solutes, and the chemical compounds were analyzed on a capillary column. Since a quantitative calibration of the GC–MS has not been carried out, the statements made in this study are valid from a qualitative perspective. Only chemical compounds with a hit probability >90% were listed in the chromatograms.

2.6. Anaerobic Digestion Experiments and Process Monitoring

The inoculum used for AD experiments was DSS collected at the local municipal sewage treatment plant (Rottenburg-Kiebingen, Germany). DSS is regarded as suitable inoculum for AD experiments as it contains a variety of different microorganisms [43]. The sampling was done from the outlet valve of the digestate container, where the DSS is stored at approx. 37 °C. After opening the outlet valve, the first approx. 10 L were discarded. The DSS was then filled into buckets, closed for transportation and handled within 1 h to avoid excess cooling and contact with ambient air. At the treatment plant, the digestate container serves as a secondary digester with an identical temperature level compared to the main AD unit, but its main purpose is to act as a buffer prior to the solid–liquid separation of the DSS.

In general, the biochemical methane potential tests were carried out with the three available PW according to VDI 4630 [43]. The volumetric biogas production was measured using glass manometers with a gas storage capacity of 1 L. The biogas composition was analyzed with a portable biogas monitor (Biogas 5000, Geotech, Coventry, UK) from biogas collected and stored in bags (Plastigas, Linde, Pullach, Germany). The specific biogas and methane production were each calculated for standard conditions (1013 hPa, 0 °C, dry gas). The configuration of each digester, the installed gas measurement system and the storage bag as used in this study are depicted in Sailer et al. [44]. The experiments were conducted in one batch test series at a mesophilic temperature of 38 °C by using a total number of 12 2L insulated glass vessels with preprogrammed heating, which were automatically stirred for 60 s/h. The AD experiment was executed for 35 d with gas analyzes conducted at day 10 and 35 of the experiment.

The experimental set-up for each digester is presented in Table 1. Approx. 1000 mL of the PW (S-1, S-2 and P) were mixed with 1000 mL DSS in triplicate (n = 3) per variant. However, the available amount of each PW for the AD experiments was not sufficient to exactly reach the targeted amount of 1000 mL for each digester, resulting in slight variations of the PW amount (Table 1). The DSS was added without further degassing, as it was already treated anaerobically at the treatment plant. Blind variants of 2000 mL DSS were carried out in triplicate (n = 3), determining the residual gas potentials of the DSS. The specific biogas yield of the mixture of the PW and DSS in each digester ($SBY_{PW\ and\ DSS}$) was calculated as

$$SBY_{PW\ and\ DSS} = \frac{BG_{PW\ and\ DSS}}{m_{oDM,PW} + m_{oDM,DSS}}, \quad (1)$$

where $SBY_{PW\ and\ DSS}$ (mL/g_{oDM}) is the specific biogas yield from the mixture of each PW and DSS, $BG_{PW\ and\ DSS}$ (mL) is the total biogas yield from the mixture of PW and DSS and $m_{oDM,PW}$ and $m_{oDM,DSS}$ (g_{oDM}) are the organic mass of each PW and DSS, respectively.

Table 1. Experimental set-up of the anaerobic digestion experiments for each digester (D) presenting the mixtures containing digested sewage sludge (DSS) and different types of press waters (spruce: S, poplar: P).

Parameter	D 1	D 2	D 3	D 4	D 5	D 6	D 7	D 8	D 9	D 10	D 11	D 12
Inoculum type	DSS	DSS	DSS	DSS	DSS	DSS	DSS	DSS	DSS	DSS	DSS	DSS
Inoculum (mL)	2000	2000	2000	1000	1000	1000	1000	1000	1000	1000	1000	1000
Feedstock type	-	-	-	S-1	S-1	S-1	P	P	P	S-2	S-2	S-2
Feedstock (mL)	-	-	-	1019	953	738	945	962	823	969	955	832

The SBY of PW alone (SBY_{PW}) was calculated for each digester as

$$SBY_{PW} = \frac{BG_{PW\ and\ DSS} - BG_{DSS}}{m_{oDM,PW}}, \quad (2)$$

where SBY_{PW} (mL/g_{oDM}) is the specific biogas yield from PW alone, and BG_{DSS} (mL) is the biogas yield from DSS, i.e., from the corresponding blanks.

The specific methane yield of the mixture of PW and DSS ($SMY_{PW\ and\ DSS}$) was calculated as

$$SMY_{PW\ and\ DSS} = \sigma_{PW\ and\ DSS} \cdot SBY_{PW\ and\ DSS}, \quad (3)$$

where $SMY_{PW\ and\ DSS}$ (mL/g_{oDM}) is the specific methane yield from the mixture of PW and DSS and $\sigma_{PW\ and\ DSS}$ (-) is the measured volume concentration of methane in the biogas from the mixture.

The SMY of the blanks was calculated as

$$SMY_{DSS} = \sigma_{DSS} \cdot SBY_{DSS}, \quad (4)$$

where SMY_{DSS} (mL/g_{oDM}) is the specific methane yield from DSS (blanks), SBY_{DSS} (mL/g_{oDM}) is the specific biogas of the DSS and σ_{DSS} (-) is the measured volume concentration of methane in the biogas from the blanks.

Finally, the SMY of PW alone (SMY_{PW}) was calculated as

$$SMY_{PW} = \frac{\sigma_{PW\ and\ DSS} \cdot BG_{PW\ and\ DSS} - \sigma_{DSS} \cdot BG_{DSS}}{m_{oDM,PW}}, \quad (5)$$

where SMY_{PW} (mL/g_{oDM}) is the specific methane yield from PW alone.

In the experiment, the measured methane concentration has been applied for the corresponding time interval. For all experiments, SMY_{DSS}, SMY_{PW} and $SMY_{PW\ and\ DSS}$ results were plotted based on mean values (n = 3 for all variants) together with standard deviation.

3. Results and Discussion

The $M_{w.b.}$ of the raw materials (wood chips) that were used to produce PW in this study were measured before and after the treatment with the mechanical dewatering press. This analysis was performed to get a basic understanding of the efficiency of the compression process and on the production conditions that could affect the quality of the PW.

The initial $M_{w.b.}$ of S-1 was reduced from 39.4% to 37.7%, while S-2 was reduced from 37.5% to 36.5%, respectively. For spruce wood, the $M_{w.b.}$ achieved was similar to that reported by Laurila et al. [28], although the initial $M_{w.b.}$ was higher (63%). The $M_{w.b.}$ of poplar wood was initially 53.4%, which was reduced to 48.3% by the mechanical dewatering press resulting in both the highest $M_{w.b.}$ before pressing and the most pronounced $M_{w.b.}$ reduction (ΔM). Still, the compression efficiency must be considered low compared to other trials with the dewatering press observed in previous experiments (data not shown). Thereby, PW accumulations between 1000 and 2000 L/h were observed for wet sawdust-like particles during compression (according to observations of the plant operator) with a ΔM of up to 15%$_{w.b.}$ on an industrial scale. These reduction efficiencies could not be observed in the mechanical dewatering processes carried out during this study. In case of both spruce samples, this could be due to the basic characteristics as presented in Table 2. Freshly harvested wood can reach drastically higher $M_{w.b.}$ of >40 to 55 wt% [3,16,18,45]. At the same time, particle size might affect the dewatering process with smaller particles, probably leading to an overall better compressibility. In addition, all compression experiments were executed with the same settings of the mechanical dewatering press. An optimization of machine settings such as throughput rate or pressure intensity could lead to different ΔM for different wood species, approximating the respective fiber saturation points. Thus, the mechanical dewatering might deliver better values for ΔM in practice while simultaneously producing larger amounts of PW. The PW quality might be directly affected by these variations regarding its mixing ratio of water, its content of soluble compounds and with regard to the resulting DM concentration.

Table 2. Dry matter (DM) concentration based on fresh matter (FM), organic dry matter (oDM) concentration, pH value and chemical oxygen demand (COD) for digested sewage sludge (DSS) and the spruce based (S-1 and S-2) as well as the poplar based press waters (P). Mean values ± standard deviation (n = 4), (n.d. = not determined).

Sample	DM (%m/m$_{FM}$)	oDM (%m/m$_{DM}$)	pH Value (-)	COD (mg/L)
DSS	3.61 ± 0.05	61.58 ± 0.12	n.d.	n.d.
S-1	2.29 ± 0.16	88.87 ± 0.26	4.6	31,350
S-2	0.39 ± 0.01	86.71 ± 0.99	4.0	10,650
P	3.17 ± 0.01	87.90 ± 0.13	7.7	45,250

Overall, the parameter ΔM of the press operations in this study ranged between $M_{w.b.}$ 1 and 5.1 wt%, which must be considered rather low, compared to literature and observations of the press operator in other mechanical dewatering tests.

For example, Liu and Haygreen [26] pressed five different wood species in the form of wood chips and hammer milled chips with pressures ranging from 3.5 to 13.8 MPa. They found significant differences in terms of achieved drying rates by size of the particles, density of the chips and species. The $M_{w.b.}$ reduction was highest within the first 2 min of the highest pressures (13.8 MPa) while pressing relatively low-density species (aspen, balsam fir, jack pine, red maple and red oak). Yoshida et al. [27] used pressures of 10–30 MPa in a two staged roller compression apparatus, discovering significantly lower energy requirements (60–70% of the primary energy required for thermal drying alone) to dry cypress wood chips with initial $M_{w.b.}$ of 61 wt% to values of 17 wt% in combined drying (first compression, followed by thermal drying). Through mechanical compression, the $M_{w.b.}$ was decreased from 50–73 wt% to 46–57 wt% depending on the wood type and depending on the initial $M_{w.b.}$. Laurila et al. [28] investigated the compression drying

of energy wood in the form of sawdust in a cylindrical vessel with horizontal pressure from above (6–38 MPa). They only found a small influence of pressure time, while the absolute pressure levels had the most influence on $M_{w.b.}$. In this case, the resulting $M_{w.b.}$ were 30 wt% for birch, 34 wt% for pine and 35 wt% for spruce. They highlighted the lower energy demand of the process compared to conventional thermal drying. Stahl and Bentz [29] applied pressures of up to 10 MPa through gas inside a pressure chamber to wood planks. They were able to approximate to the fiber saturation point by repeating their process several times. Frodeson et al. [30] recently investigated an industrial scale dewatering press in combination with a packed moving bed dryer. For a combined drying of sawdust from 52 to 12.5 wt% $M_{w.b.}$, a reduction of thermal energy requirements of 50% was observed.

Thus, the achieved values for ΔM in this study might have influenced the characteristics of the resulting PW and must therefore be considered when evaluating the results as presented in the following sections.

3.1. Basic Characteristics of Wood Press Waters and Digested Sewage Sludge

The obtained PW showed greater differences in terms of DM concentration, pH and COD (Table 2). This is also valid between samples of the same wood species, such as S-1 and S-2, and might be directly linked to the pressing efficiency as described above. While the pressing success (ΔM) was better for S-1 compared to S-2, the DM concentration of the PW was also higher for this sample (2.3%) compared to S-2 (0.4%). The same trend was observed for P with the overall highest ΔM and the highest DM concentration ($3.17 \pm 0.01\%_{FM}$, Table 2). From these observations, a dependence of the PW's DM concentration on the ΔM and thus the original $M_{w.b.}$ may be suggested. However, this must be validated with further experiments.

Frodeson et al. [30] mention that the DM loss during pellet production due to DM in the PW can either be considered as loss of substances with a reduction of pellet yield and possibly altered pelletization behavior, or as a new and innovative feedstock, i.e., when the DM losses are used for bio-economy purposes [30]. The results of the pressing tests in this study have shown that for wood chips, a maximum amount of only 1.62 kg_{DM} per ton_{FM} of wood chips is transferred to the pressing water (calculated with ΔM and DM concentration of P). Otherwise, if the PW should be used for bio-based process chains, the compression step may need to be optimized to increase the DM concentration in PW.

For instance, the concentration of DM in the PW has to be considered when utilized as a co-substrate in AD plants. Large amounts of PW (i.e., with a 1:1 mixture as applied in this study) may also deliver large amounts of water into the digester, which then would lead to increased heating demands while obviously delivering only a limited mass of degradable organic components. For the evaluation of PW and energy yields in AD, all reference units (FM, DM, oDM) must be considered. However, PW could also be used as a supplement with lower dosages (e.g., in a 1:10 mixture of PW and the main feedstock) to minimize the energy demand for heating.

In contrast to the DM concentration, the oDM concentrations were on similar level (approx. $88\%_{DM}$) for all three PW. Thus, the remaining mass of DM contained a variety of organic components that were pressed out of the wood while containing approx. $12\%_{DM}$ of inorganic components (ash). The oDM concentration is especially relevant when utilizing substrates energetically as only the mass of oDM can be converted to biogas in AD processes. In comparison, the pure DSS as a reference substrate achieved the highest DM with the lowest oDM concentration (approx. $3.6\%_{FM}$ and $62\%_{DM}$, respectively, Table 2). Both DM and oDM concentrations of DSS were in line with literature [44,46,47]. The concentration of C, H and N were not measured in this study, but the concentration of C correlated with the concentration of oDM and could thus be estimated. In other digestion experiments of the authors [44,46], DSS was measured with similar oDM concentrations (63.5 and $60.2\%_{DM}$) as presented in Table 2, while C was measured with 29.7 and $30.4\%_{DM}$ (with H both at $4.5\%_{DM}$ as well as with N at 3.8 and $4.1\%_{DM}$). Thus, the C concentration of approx. $30\%_{DM}$ can be

assumed for the DSS of this study while the PW can be estimated with C concentration of approx. 45%$_{DM}$. However, the concentration of C, H and N of the PW should be measured in further research. Based on C, H and N, stoichiometric methane yields in AD can be calculated [43].

The pH value of DSS was not measured in this study. Based on literature [44,47], a pH value between 6.5 and 7.5 could be expected for DSS. The results of the pH value analysis for the PW (between 4.0 and 7.7) are in accordance with results in the literature. The low pH value of spruce-based PW is already known with a similar pH value for spruce chips (pH 4.53). In addition, the ingredients of softwood typically cause lower pH values [48]. Ciesielski et al. [31] found a pH value of a PW from a 3:1 spruce:fir mixture of 4.52. The pH value of the feedstock is an important parameter in AD processes [44]. Thus, depending on the amount and type of PW in the digester, pH values can be influenced towards lower (S-1, S-2) or higher (P) pH values.

The COD of PW ranged from 10,650 to 45,250 mg/L (Table 2). Compared to the COD ranges mentioned in the literature for different industry segments (1000–100,000 mg/L), the PW of this study can be compared to industrial wastewaters from dairy or from food factories [3]. The PW from spruce and fir used by Ciesielski et al. [31] showed a COD of 4400, which is comparable to sample S-2, with a DM concentration of 0.28%.

The German Waste Water Ordinance sets limits for COD of water from different industries before it can be directly discharged in surface waters. According to this ordinance, for manufacturing of chipboard, fiberboard or wood fiber matting, maximum COD values of 200 mg/L (annual average) must not be exceeded. Therefore, as the COD values of all PW were severely above the legal limits, subsequent treatment of the water seems to be necessary if the PW should be disposed of [39]. From this perspective (disposal), a suitable utilization pathway of wood-based PW is preferable.

In addition, the theoretical biogas and methane potential can be calculated based on the COD (simplified: 1 g of degraded COD can generate in maximum 350 mL of methane) [3]. Thus, a higher level of COD might also indicate a higher energy potential.

3.2. Elemental Composition and Chemical Compounds of Wood Press Waters

For the spruce-based PW, higher absolute element concentrations were found in S-1 compared to S-2 (e.g., K with 816 mg/L vs. 222 mg/L, Table 3). This may be due to the slightly higher bark content of the S-1 sample (determined by visual inspection) as bark contains multiple inorganic charges compared to wood (see, e.g., [3,49]), but this could also be explained by the slightly higher ΔM (1.7%). The latter, however, would imply a stronger leaching effect with greater water flow compared to that of the dilution effect due to the increased water flow. Similar, the poplar-based PW contained higher amounts of minor and trace elements compared to both spruce samples (e.g., K was at 1510 mg/L).

While the elemental composition of the liquid PW (Table 3) cannot be directly compared to the elemental composition of solid wood, tendencies and correlations for the wood species spruce and poplar can be observed. The poplar-based PW was especially high in concentration of K, Ca, Mg, Na and Cu. This can partly be explained by the higher trace element concentration (K, Ca, Mg) in the raw material, as typical elemental concentrations in poplar wood are higher than those of spruce [3]. These elements have a decisive influence on the fuel properties of wood chips and their combustion behavior [3,10,50–52]. K, for example, is one of the main aerosol forming elements during combustion and strongly affects slag formation. The K concentration in the PW should theoretically lead to a decrease of the element concentration in the fuel. In the pressing process of P with the initial $M_{w.b.}$ of 53% to 48%, an equivalent of more than 165 mg/kg$_{DM}$ of K should have been flushed out of the wood with the water. Assuming an average K concentration of 3500 mg/kg$_{DM}$ in wood chips from a short rotation coppice, this corresponds to a leaching of 4.72% of the total K amount in the wood [3] and should therefore improve the combustion properties of the wood chips.

Table 3. Minor and trace element concentration of spruce-based (S-1 and S-2) and of poplar-based (P) press waters. For Sb, Ag, Ti, Li and Bi, all values were below the detection limit and thus not detected (n.d.). Mean values ± standard deviation (n = 4). The order of the elements was selected in accordance with IS0 17225-1.

Element	S-1	S-2	P
	mg/L		
Al	9.091 ± 0.152	0.615 ± 0.023	2.362 ± 0.053
Ca	198.3 ± 1.34	79.02 ± 0.82	381.2 ± 4.22
Fe	128.0 ± 1.14	12.07 ± 0.12	16.24 ± 0.42
K	815.5 ± 10.55	222.0 ± 2.03	1510 ± 5.87
Mg	119.5 ± 0.75	22.42 ± 0.23	171.6 ± 0.86
Mn	113.2 ± 0.799	7.686 ± 0.079	3.497 ± 0.056
Na	14.70 ± 0.158	2.628 ± 0.228	107.8 ± 0.705
As	0.011 ± 0.003	0.007 ± 0.005	n.d.
Cd	0.020 ± 0.000	0.007 ± 0.004	0.036 ± 0.000
Cr	0.953 ± 0.013	0.029 ± 0.003	0.030 ± 0.002
Cu	0.525 ± 0.010	n.d.	1.237 ± 0.048
Ni	1.184 ± 0.012	n.d.	0.139 ± 0.002
Pb	0.010 ± 0.004	n.d.	0.113 ± 0.004
V	0.045 ± 0.007	0.004 ± 0.005	n.d.
Zn	25.96 ± 0.323	0.601 ± 0.013	8.767 ± 0.163
Ba	2.436 ± 0.016	0.679 ± 0.010	2.079 ± 0.034
Sr	0.832 ± 0.006	0.234 ± 0.005	1.062 ± 0.013
B	1.416 ± 0.013	n.d.	0.618 ± 0.009
Mo	0.518 ± 0.007	0.004 ± 0.004	0.038 ± 0.001
Se	0.123 ± 0.009	0.030 ± 0.006	0.049 ± 0.005
Co	0.143 ± 0.002	0.012 ± 0.001	0.031 ± 0.001
Tl	0.148 ± 0.001	0.030 ± 0.004	n.d.
Be	0.007 ± 0.000	0.005 ± 0.003	0.002 ± 0.000

Dietz et al. observed contamination effects (Cr, Co, Fe, Ni, Mo) through abrasion of machine components in contact with the wood (e.g., blades constructed using heavy metal containing steels) when processing different wood chips in laboratory mills. This abrasion effect was caused by shares of mineral impurities in the sample [45]. Compared to the S-2 and P samples, S-1 showed elevated values for the above-mentioned elements (Table 3). Therefore, in addition to natural causes, increased mineral contamination and therefore increased abrasion in the processing steps (from comminution, conveying in the mechanical pressing) could also explain the measured concentrations (Table 3).

In further experiments, the elemental profile of the untreated solid wood (raw state) and the treated wood (mechanically dewatered) together with the generated PW could be monitored in order to quantify the leaching effects. For spruce and poplar wood, typical elemental profiles of the solid and untreated wood can be found in literature [3,10,45].

Trace elements are of importance for AD processes. They can enhance the efficiency of AD by increasing energy yields but can also become toxic, depending on the concentration in the digester. Important trace elements for AD are reportedly Fe, Co, Mn, Mo, W, Ni, Se and Zn. Trace element requirements of AD processes depend on various parameters, which is why the ranges of optimum elemental concentrations vary in the literature. For Na, K, Ca, Mg, S, Ni, Cu, Cr, Co, Pb and Zn, inhibitory levels for AD processes are defined in the literature [44]. However, none of the mentioned elements contained in the PW seems to be critical (the most relevant could be K with an inhibitory level of 4800 mg/L). In addition to inhibitory levels, optimum ranges are defined for Ni, Co, Fe, Mn, Mo, Se and Zn [44]. None of the mentioned elements was measured within the optimum ranges as described in the literature. However, the concentration of beneficial elements are closer to the optimum ranges than the concentration of inhibitory elements to their critical concentration. From an elemental perspective, this could suggest an overall positive impact of the PW for AD

processes. In general, DSS already contains a broad variety of elements such as P, Al, Ca, Fe, K, Mg, Na, S and Si but also heavy metals that are critical with regard to legal limits [44]. PW could thus also be used as supplement delivering nutrients for other substrates with less extensive elemental profiles.

In Figure 2, the results of the GC-MS analysis of PW are shown. Table 4 complements Figure 2 by presenting the associated chemical compounds as detected by the GC–MS system. In general, the largest amounts of peaks were detected for the poplar-based PW (P) followed by the spruce-based PW (S-1 and S-2). Most of the detected peaks of the P sample could not be identified (hit probability > 90%) by the GC–MS system. Thus, different chemical compounds were detected but could not be identified with certainty. Interestingly, the chromatograms of the samples S-1 and S-2 did not show similar progressions, indicating differences in chemical compounds even among samples of the same species. Only a few overlaps were found for S-1 and S-2. For instance, alpha-terpineol was detected in both samples. This substance is commonly known as an important component in the perfume and odor industry. Furthermore, alpha-terpineol inhibits the growth of tumor cells [53,54]. It has proven to show anti-tubercular activities, is active against multi-drug-resistant tuberculosis and extensively drug-resistant tuberculosis [55]. The compound 3-carene (detected in S-1) is an antimicrobial monoterpene that has an antibacterial mechanism against foodborne pathogens and occurs naturally in a variety of plants [56]. Alpha-cadinol, which was also detected in S-1, has antifungal and hepatoprotective effects and, in addition, anti-inflammatory effects are associated with it [57–59].

Table 4. Chemical compounds detected by gas chromatography–mass spectrometry (GC–MS) for spruce-based (S-1 and S-2) and poplar-based (P) press waters.

Peak No	Compound	S-1	S-2	P
1	Acetic acid		X	
2	Silanediol, dimethyl-	X	X	X
4	Cyclotrisiloxane, hexamethyl-		X	
5	Oxime-,methoxy-phenyl-		X	
11	Cyclotetrasiloxane, octamethyl-		X	
12	3-carene	X		
15	Cyclohexene, 4-methyl-1-(1-methylethyl)- or limonene	X		
16	Benzaldehyde, 2-hydroxy-			X
26	(+)-2-bornanone/camphor	X		
29	Acetic acid, phenylmethyl ester			X
32	Endo-borneol	X		
34	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl), (R)-	X		
37	Tripen-4-ol		X	
39	Alpha-terpineol	X	X	
40	Bicyclo [3.1.1] hept-3-en-2.-one, 4,6,6- trimethyl-	X	X	
43	Acetic acid, 2-phenylethyl ester/phenethyl acetate			X
45	Phenol, 2-nitro-	X		
50	2(3H)-furanone, dihydro-5-pentyl/gamma-nonalactone		X	
56	Acetaldehyde, (3,3-dimethylcyclohexylidene)-			
60	Tau-muurolol	X		
61	Alpha-cadinol	X		
65	Hexacosane, eicosane, methyl- or heneicosane		X	

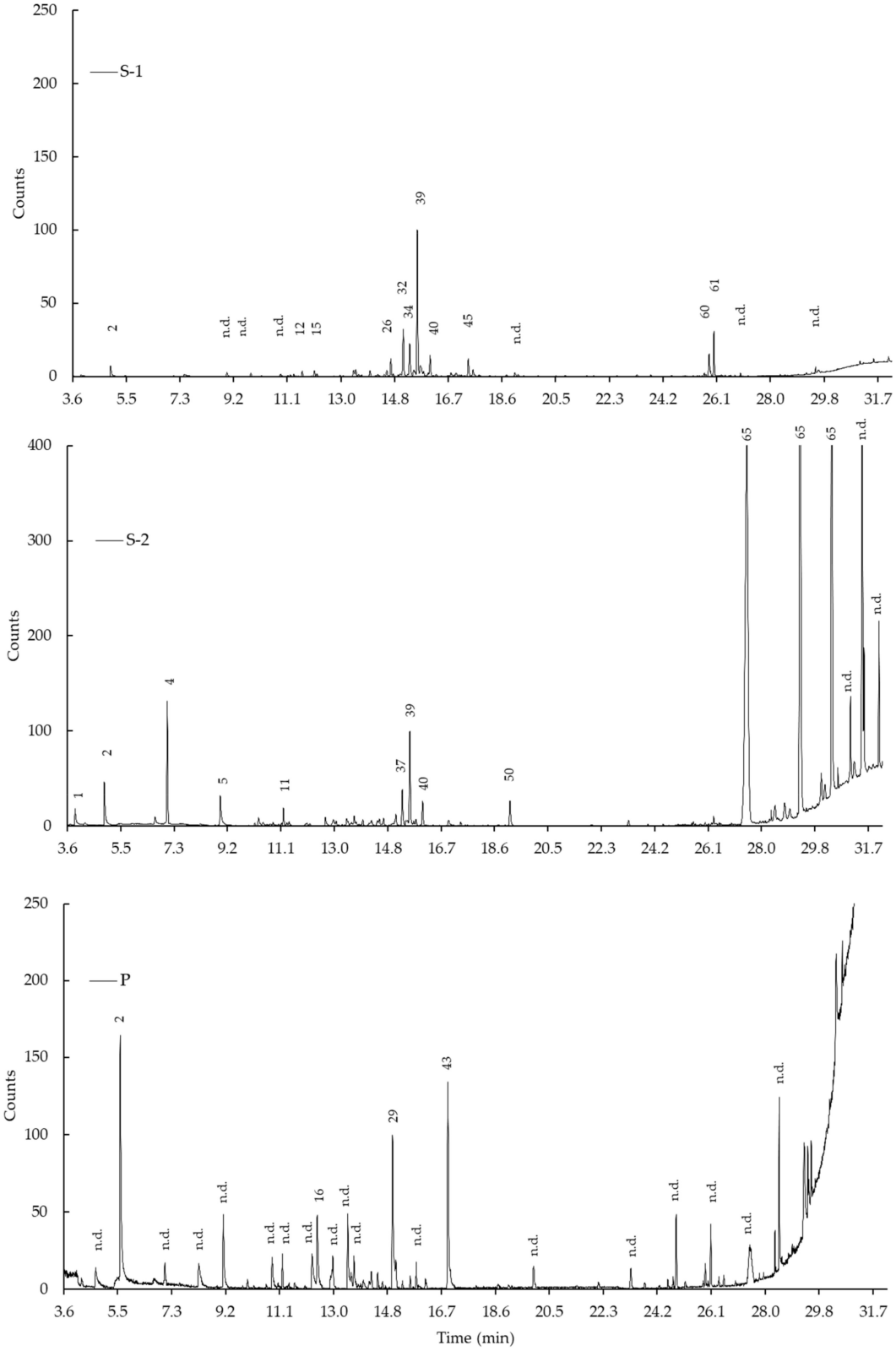

Figure 2. Chemical compounds measured by gas chromatography–mass spectrometry (GC–MS) for spruce-based (S-1 and S-2) and poplar-based (P) press waters. Detected chemical compounds were labeled with numbers. Compounds that could not be identified by the GC–MS system were labeled as not detected (n.d.). Further information on the chemical compounds can be found in Table 4.

In the literature, Ciesielski et al. [31] investigated the suitability of "wood juice" from a mixture of spruce/fir and a pure Douglas fir for the direct synthesis of biopolymers by bacteria. Although they found a relatively low polyhydroxyalcanoates concentration, the approach seems worth pursuing, as substrate pretreatment is not required, and a substantial amount of 3-hydroxyvalerate indicates that the obtained co-polymers provide suitable characteristics for industrial applications. In addition, Wilke et al. [32] showed a good suitability of PW in yeast production. Yeast strains nourished with PW showed significantly improved growth behavior in shake flask experiments compared to the use of a conventional industrial complex medium. Thus, initial approaches for material use of PW are already available.

Overall, this study focused on the energetic valorization of the PW in AD, but the additionally performed GC–MS analyses showed that several chemical compounds of interest for the chemical industry can be detected in PW. Quantitative investigations could be carried out to identify relevant material use potentials, exact amounts and extraction processes for the chemical compounds in PW. Thus, for each of the detected chemical compounds, measurements on the quantitative compound concentration should be performed followed by a discussion of the utilization potential and possible bio-economy pathways. In addition, the potential effects of the (detected) chemical compounds on AD processes could be a field of future research.

3.3. Anaerobic Digestion Experiments

Based on the mixtures of DSS and the different PW in each digester, both SBY and SMY are presented in Figure 3. The mixture of DSS and the PW S-2 achieved the highest $SBY_{PW\ and\ DSS}$ and $SMY_{PW\ and\ DSS}$ followed by the blank variant (DSS alone). From a digester perspective, which considers the yields per g_{oDM} of the mixtures, the combination of DSS and S-2 was the most efficient variant. The mixtures of DSS together with S-1 and P were measured with even lower $SBY_{PW\ and\ DSS}$ and $SMY_{PW\ and\ DSS}$ compared to the blank variant (containing only DSS). In the case of S-1 and P, the AD process was thus inhibited. Based on the results displayed in Figure 3, the co-digestion of DSS and PW may obviously lead to positive or negative impacts on the AD process. This can be discussed based on the digester content, which is described by physico-chemical properties of the PW such as DM and oDM concentration, pH values as well as minor and trace elements. In the case of the beneficial variant S-2, the lowest DM concentration ($0.39 \pm 0.01\%_{FM}$, Table 2) was measured, while oDM concentrations of all PW were equally between 87 and $89\%_{DM}$. The high pH value of the poplar-based PW (approx. 8) could have also inhibited the AD process (especially due to the almost 1:1 mixture). For further explanations of the results in Figure 3 and for a detailed view of the content in each digester, Table 5 delivers absolute masses for DM and oDM as well as oDM ratios and absolute biogas and methane yields.

The absolute masses of DM and oDM delivered by the DSS were identical for all variants based on 2000 mL for the blank variants and based on 1000 mL for the mixtures with the PW. However, the different DM concentration of the PW (Table 2) strongly influenced the absolute masses of DM (ranging from 3 to 30 g) and oDM (ranging from 3 to 27 g) that were present in each digester. Consequently, this led to varying $oDM_{Feedstock}$ to oDM_{DSS} ratios between 0.1 and 1.2. The digesters with the lowest oDM ratios (S-2 with ratios of 0.1–0.2) delivered the best results in terms of $SBY_{PW\ and\ DSS}$ and $SMY_{PW\ and\ DSS}$. When comparing the absolute methane yields of DSS alone (4226 ± 1155 mL) with those of the DSS and S-2 mixture (4054 ± 297 mL), similar results were achieved. However, the drastically lower mass of oDM in the digester of the mixtures (25 g vs. 45 g for DSS alone) must be considered. Thus, the mixture of DSS and S-2 delivered promising results that suggest (a) high substrate specific gas yields of the PW, and (b) an increased efficiency in utilizing the mass of oDM provided by the DSS.

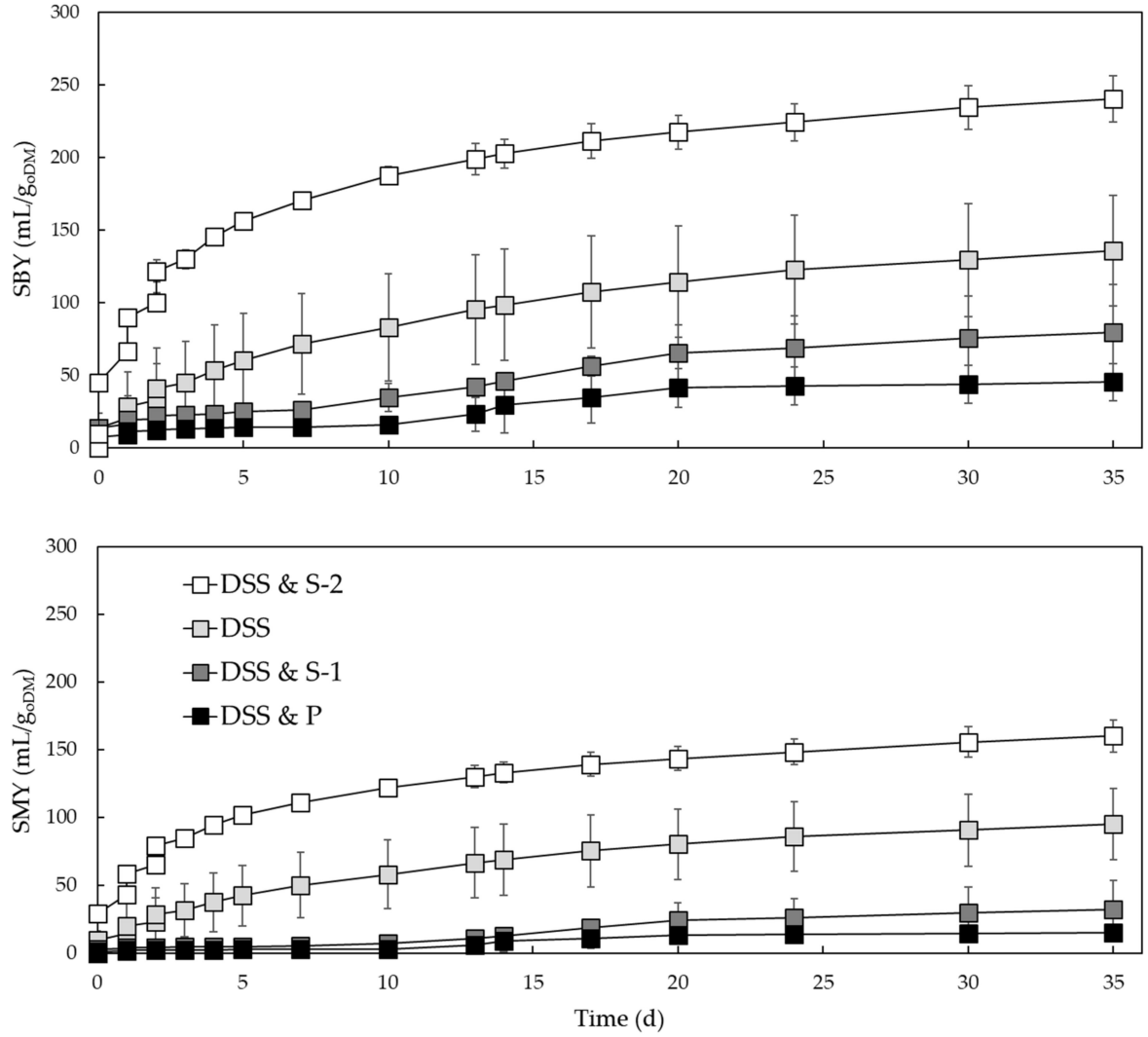

Figure 3. Specific biogas yield (SBY) and specific methane yield (SMY) based on organic dry matter (oDM) for the mixtures of digested sewage sludge (DSS) together with spruce-based (S-1 and S-2) and poplar-based (P) press waters and for DSS (inoculum) alone. Mean values ± standard deviation (n = 3).

Table 5. Masses of digested sewage sludge (DSS), spruce-based press water (S-1, S-2) and poplar-based press waters (P) based on dry matter (DM) and organic dry matter (oDM) in each digester (D). Biogas and methane volumes are related to standard conditions (1013 hPa, 0 °C, dry gas).

Parameter	D 1	D 2	D 3	D 4	D 5	D 6	D 7	D 8	D 9	D 10	D 11	D 12
Digester content	DSS	DSS	DSS	DSS&S-1	DSS&S-1	DSS&S-1	DSS&P	DSS&P	DSS&P	DSS&S-2	DSS&S-2	DSS&S-2
$m_{DM,\ DSS}$ (g)	72.2	72.2	72.2	36.1	36.1	36.1	36.1	36.1	36.1	36.1	36.1	36.1
$m_{DM,\ Feedstock}$ (g)	-	-	-	23.3	21.8	16.9	30.0	30.5	26.1	3.8	3.7	3.2
$m_{DM,\ Digester}$ (g)	72.2	72.2	72.2	59.4	57.9	53.0	66.1	66.6	62.2	39.9	39.8	39.3
$m_{oDM,\ DSS}$ (g)	44.5	44.5	44.5	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2	22.2
$m_{oDM,\ Feedstock}$ (g)	-	-	-	20.7	19.4	15.0	26.3	26.8	22.9	3.3	3.2	2.8
$m_{oDM,\ Digester}$ (g)	44.5	44.5	44.5	42.9	41.6	37.2	48.5	49.0	45.1	25.5	25.4	25.0
$oDM_{Feedstock}$ / oDM_{DSS}	-	-	-	0.9	0.9	0.7	1.2	1.2	1.0	0.2	0.2	0.1
Biogas (mL)	4493	5215	8399	5433	2318	2116	1802	1718	2867	6542	5570	6159
Methane (mL)	3176	3666	5835	2660	678	687	551	570	988	4390	3667	4106

In general, an increased oDM ratio of up to 0.9 and 1.2 (S-1 and P), drastically decreased the gas yields, which is in line with the ranking as presented in Figure 3. In accordance with VDI 4630 [43], the oDM ratio should remain below 0.5 to ensure a stable and efficient AD process. Therefore, the main reason for the poor performance of S-1 and P, with regard to both absolute and specific gas yields (Table 5 and Figure 3), could have been the strongly elevated oDM ratio.

In addition to the results for the mixtures, Table 6 presents the results for SBY_{DSS}, SBY_{PW}, SMY_{PW} and SMY_{DSS}. In Germany, the average SBY from sewage sludge is 520 mL/g_{oDM} [3,44]. Thus, the residual biogas potential SBY_{DSS} as measured in this study with approx. 136 ± 38 mL/g_{oDM} (25% of the average sewage sludge SBY) indicates that the inherent energy content of DSS could be exploited to a greater extend. Szaja et al. [47] used sewage sludge for co-digestion experiments and measured SMY between 230 and 270 mL/g_{oDM} for sewage sludge alone, which strengthens the statements above.

Table 6. Substrate specific biogas yield (SBY) and specific methane yield (SMY) based on fresh matter (FM), dry matter (DM) and organic dry matter (oDM) for digested sewage sludge (DSS), spruce-based (S-1, S-2) and poplar-based press waters (P). For S-1 and P, negative values indicate a reduction of the gas yield compared to the blank (Figure 3). Mean values ± standard deviation (n = 3).

Sample	SBY			SMY		
	mL/g_{oDM}	mL/g_{DM}	mL/g_{FM}	mL/g_{oDM}	mL/g_{DM}	mL/g_{FM}
DSS	136 ± 38	84 ± 24	3.0 ± 0.8	95 ± 26	59 ± 16	2.1 ± 0.6
S-1	7 ± 78	6 ± 69	0.1 ± 1.6	−48 ± 53	−42 ± 47	−1.0 ± 1.1
S-2	994 ± 145	862 ± 126	3.4 ± 0.5	628 ± 104	545 ± 90	2.1 ± 0.4
P	−34 ± 19	−30 ± 17	−0.9 ± 0.5	−55 ± 4	−49 ± 4	−1.5 ± 0.1

As explained, only the PW S-2 was able to deliver considerable gas yields above those of the blank variants. The SBY_{PW} and SMY_{PW} from S-2 alone were measured with almost 1000 mL/g_{oDM} and 630 mL/g_{oDM}, respectively. However, for the evaluation of the PW, the concentration of oDM and DM (Table 2) in the fresh state are of importance. Thus, a SBY_{PW} and SMY_{PW} of approx. 3 mL/g_{FM} and 2 mL/g_{FM}, respectively, was achieved.

After day 35 of the AD experiments, all remaining digestates of each variant were analyzed regarding DM and oDM concentration. The DSS (blank) variant was measured with concentration of 1.85 ± 0.06%$_{FM}$ (DM) and 53.92 ± 0.37%$_{DM}$ (oDM). The mixtures of DSS and PW were analyzed with DM concentrations of 1.33 ± 0.19%$_{FM}$ (S-1), 0.97 ± 0.41%$_{FM}$ (S-2) and 2.14 ± 0.21%$_{FM}$ (P), while oDM concentrations were 65.78 ± 1.39%$_{DM}$ (S-1), 52.66 ± 0.15%$_{DM}$ (S-2) and 60.45 ± 0.77%$_{DM}$ (P).

3.4. Potential of Press Waters from Wood Fuel Processing in Anaerobic Digestion

Based on the physico-chemical characteristics of the PW, expected advantages and challenges and thus their potential in AD processes can be summarized as follows:

(1) The low DM concentration of the PW (between 0.4 and 3.2%) might be challenging due to large amounts of water that are delivered into the digester (heating demand). However, PW could be used in individual and lower dosages (in this study 1:1 was applied), or it could be used to increase the amount of water in the digester for dry substrates (mashing of the feedstock) while delivering organic compounds and nutrients. An aspect worth investigating could be the process of increasing DM concentration, e.g., through thickening processes.

(2) The oDM concentration of the PW indicate suitable amounts of organic components for conversion into biogas through AD while also delivering inorganic components (ash) that could promote AD processes. The oDM concentration is similar to biowaste materials such as the organic fraction of municipal solid waste [44]. Although the oDM concentration correlates with the content of C, the C (and H as well as N)

concentration should be measured to also calculate the stoichiometric methane potentials in AD.

(3) The pH values from spruce-based PW were in the acidic milieu, while poplar-based PW were approx. pH-neutral. Thus, depending on the type and amount or dosage of PW into the digester, the pH values in AD could be adjusted.

(4) The COD can also serve as an indicator for achievable energy yields in AD. The PW levels of COD proved that a suitable treatment is necessary to fulfill legal limits.

(5) The concentration of chemical elements as well as the detection of chemical compounds can be used to develop suitable substrate mixtures in AD. Depending on the presence and concentration of certain elements, their concentration can be inhibiting or promoting. In this study, an overall positive effect of the PW on AD processes was assumed. However, low ratios of oDM_{PW} to oDM_{DSS} (< 0.5) seem to be important for an efficient AD. In addition to analyses regarding the effect of the detected chemical compounds on AD, further research could focus on material use in the sense of a bioeconomy. Therefore, extraction experiments and a quantitative analysis for evaluating the biorefinery potential of PW should be carried out.

In general, it could be a suitable option to energetically utilize PW together with DSS, as the residual gas potential of DSS could be exploited with higher efficiency. Additionally, the PW could be of interest as a supplement for further AD systems such as energy crop- or bio-waste-based AD plants. The application of wood-based PW could also be tested as diluting liquid in AD of relatively dry substrates such as bio-waste materials. This could be a field of future research. Additionally, co-digestion experiments of DSS mixed with one individual PW at different oDM ratios should be executed. Further areas of application might be the utilization of PW as fertilizer. Therefore, the elemental composition (N, P, K) and the phytotoxicity of the PW should be analyzed. Utilizing by-products from wood fuel production in the AD of DSS would lead to combined treatment approaches with a linking between forestry and AD.

4. Conclusions

This study investigated the anaerobic digestion (AD) of three different spruce- and poplar-based press waters (PW) generated as a by-product of a mechanical dewatering press. The PW were characterized with a dry matter concentration between 0.4 and 3.2%$_{FM}$ and with an organic dry matter (oDM) concentration of approx. 88%$_{DM}$. The pH value of poplar-based PW was drastically higher (pH 8) than those of the spruce-based PW (pH 4). The results of the elemental analysis suggest positive impacts regarding combustion characteristics of the mechanically pressed wood. In addition, the PW might deliver relevant trace elements for AD. Further, several chemical compounds interesting for a bioeconomy were detected by GC–MS. The mixture of spruce-based PW and digested sewage sludge (DSS) with low oDM ratios delivered highest absolute and specific methane yields. The PW could be used as co substrate to optimize wastewater treatment by enhancing the exploitation of the residual gas potential of DSS.

Author Contributions: Conceptualization, G.S., F.E., D.K. and S.P.; methodology, G.S. and F.E.; validation, G.S., F.E., D.K. and J.M.; formal analysis, G.S. and J.M.; investigation, G.S., F.E., M.S., S.L. and D.L.; writing—original draft preparation, G.S., F.E., D.K. and J.M.; writing—review and editing, G.S., F.E., D.K. and J.M.; visualization, G.S., F.E. and M.S.; supervision, D.K., S.P. and J.M.; project administration, D.K. and S.P.; funding acquisition, D.K. and S.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the German Federal Ministry of Food and Agriculture (BMEL, grant numbers 22023217 and 22016717) on decision of the German Bundestag. Grant distribution was administered by Fachagentur Nachwachsende Rohstoffe e.V. (FNR). The APC was funded by the Baden-Württemberg Ministry of Science, Research and Culture in the funding program Open Access Publishing.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available upon request from the corresponding author.

Acknowledgments: The authors would like to thank Viktoria Knappe and Michael Russ for laboratory assistance.

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Abbreviations

AD	anaerobic digestion
DM	dry matter
DSS	digested sewage sludge
FM	fresh matter
M	moisture content
oDM	organic dry matter
SBY	specific biogas yield
SMY	specific methane yield
PW	press water(s)
w.b.	wet basis
ΔM	moisture content reduction

References

1. Arbeitsgruppe Erneuerbare Energien (AGEE). *Zeitreihen zur Entwicklung der Erneuerbaren Energien in Deutschland: Unter Verwendung von Daten der Arbeitsgruppe Erneuerbare Energien-Statistik (AGEE-Stat.)*; Bundesministerium für Wirtschaft (BMWi): Berlin, Germany, 2021. Available online: https://www.erneuerbare-energien.de/EE/Redaktion/DE/Downloads/zeitreihen-zur-entwicklung-der-erneuerbaren-energien-in-deutschland-1990-2020.pdf (accessed on 25 December 2021).
2. *DIN EN ISO 17225-1:2021-10*; Solid Biofuels-Fuel Specifications and Classes-Part. 1: General Requirements (ISO 17225-1:2021). Beuth Verlag GmbH: Berlin, Germany, 2021.
3. Kaltschmitt, M.; Hartmann, H.; Hofbauer, H. *Energie Aus Biomasse*; Springer: Berlin/Heidelberg, Germany, 2016; ISBN 978-3-662-47437-2.
4. Schön, C.; Hartmann, H. *Nutzer- und Brennstoffeinflüsse auf Feinstaubemissionen aus Kleinfeuerungsanlagen*; Berichte aus dem TFZ No. 36; Technologie- und Förderzentrum im Kompetenzzentrum für Nachwachsende Rohstoffe (TFZ): Straubing, Germany, 2014. Available online: https://www.tfz.bayern.de/mam/cms08/festbrennstoffe/dateien/36_bericht.pdf (accessed on 30 December 2021).
5. Quintero-Marquez, A.; Bernard, C.; Zoulalian, A.; Rogaume, Y. Improving the operation of an automatic wood chip boiler by optimizing CO emissions. *Energy Fuels* **2014**, *28*, 2152–2159. [CrossRef]
6. Schön, C.; Kuptz, D.; Mack, R.; Zelinski, V.; Loewen, A.; Hartmann, H. Influence of wood chip quality on emission behaviour in small-scale wood chip boilers. *Biomass Conv. Bioref.* **2019**, *9*, 71–82. [CrossRef]
7. Nord-Larsen, T.; Bergstedt, A.; Farver, O.; Heding, N. Drying of firewood—The effect of harvesting time, tree species and shelter of stacked wood. *Biomass Bioenergy* **2011**, *35*, 2993–2998. [CrossRef]
8. Shen, G.; Xue, M.; Wei, S.; Chen, Y.; Zhao, Q.; Li, B.; Wu, H.; Tao, S. Influence of fuel moisture, charge size, feeding rate and air ventilation conditions on the emissions of PM, OC, EC, parent PAHs, and their derivatives from residential wood combustion. *J. Environ. Sci.* **2013**, *25*, 1808–1816. [CrossRef]
9. Kuptz, D.; Hartmann, H. The effect of raw material and machine setting on chipping performance and fuel quality—A German case study. *Int. J. For. Eng.* **2015**, *26*, 60–70. [CrossRef]
10. Döring, S. *Pellets als Energieträger*; Springer: Berlin/Heidelberg, Germany, 2011; ISBN 978-3-642-01623-3.
11. Stenström, S. Drying of biofuels from the forest - A review. *Dry. Technol.* **2017**, *35*, 1167–1181. [CrossRef]
12. Yi, J.; Li, X.; He, J.; Duan, X. Drying efficiency and product quality of biomass drying: A review. *Dry. Technol.* **2020**, *38*, 2039–2054. [CrossRef]
13. Li, H.; Chen, Q.; Zhang, X.; Finney, K.N.; Sharifi, V.N.; Swithenbank, J. Evaluation of a biomass drying process using waste heat from process industries: A case study. *Appl. Therm. Eng.* **2012**, *35*, 71–80. [CrossRef]
14. Kuptz, D.; Schreiber, K.; Schulmeyer, F.; Lesche, S.; Zeng, T.; Ahrens, F.; Zelinski, V.; Schön, C.; Pollex, A.; Borchert, H.; et al. Evaluation of combined screening and drying steps for the improvement of the fuel quality of forest residue wood chips—Results from six case studies. *Biomass Conv. Bioref.* **2019**, *9*, 83–98. [CrossRef]
15. Jirjis, R. Storage and drying of wood fuel. *Biomass Bioenergy* **1995**, *9*, 181–190. [CrossRef]

16. Hofmann, N.; Mendel, T.; Schulmeyer, F.; Kuptz, D.; Borchert, H.; Hartmann, H. Drying effects and dry matter losses during seasonal storage of spruce wood chips under practical conditions. *Biomass Bioenergy* **2018**, *111*, 196–205. [CrossRef]
17. Noll, M.; Jirjis, R. Microbial communities in large-scale wood piles and their effects on wood quality and the environment. *Appl. Microbiol. Biotechnol.* **2012**, *95*, 551–563. [CrossRef] [PubMed]
18. Hofmann, N.; Mendel, T.; Kuptz, D.; Schulmeyer, F.; Borchert, H.; Hartmann, H. *Lagerung von Holzhackschnitzeln: Trockenmasseverluste, Änderungen der Brennstoffqualität und Kosten*; Berichte aus dem TFZ No. 55; Technologie- und Förderzentrum im Kompetenzzentrum für Nachwachsende Rohstoffe (TFZ): Straubing, Germany, 2017. Available online: https://www.tfz.bayern.de/mam/cms08/festbrennstoffe/dateien/tfz_bericht_55_lagerung_hackschnitzel_ges.pdf (accessed on 22 December 2021).
19. Barontini, M.; Scarfone, A.; Spinelli, R.; Gallucci, F.; Santangelo, E.; Acampora, A.; Jirjis, R.; Civitarese, V.; Pari, L. Storage dynamics and fuel quality of poplar chips. *Biomass Bioenergy* **2014**, *62*, 17–25. [CrossRef]
20. Ferrero, F.; Malow, M.; Noll, M. Temperature and gas evolution during large scale outside storage of wood chips. *Eur. J. Wood Prod.* **2011**, *69*, 587–595. [CrossRef]
21. Koppejan, J.; Lönnermark, A.; Persson, H.; Larsson, I.; Blomqvist, P.; Arshadi, M.; Valencia-Reyes, E.; Melin, S.; Howes, P.; Wheeler, P.; et al. Health and safety aspects of solid biomass storage, transportation and feeding. IEA Bioenergy: Paris, France, 2013.
22. Whittaker, C.; Yates, N.E.; Powers, S.J.; Misselbrook, T.; Shield, I. Dry matter losses and greenhouse gas emissions from outside storage of short rotation coppice willow chip. *Bioenergy Res.* **2016**, *9*, 288–302. [CrossRef] [PubMed]
23. Hofmann, N.; Borchert, H. Influence of fuel quality and storage conditions on oxygen consumption in two different wood chip assortments–Determination of the storage-stable moisture content. *Fuel* **2022**, *309*, 122196. [CrossRef]
24. Erber, G.; Kanzian, C.; Stampfer, K. Modelling natural drying of European beech (*Fagus sylvatica* L.) logs for energy based on meteorological data. *Scand. J. For. Res* **2016**, *31*, 294–301. [CrossRef]
25. Filbakk, T.; Høibø, O.; Nurmi, J. Modelling natural drying efficiency in covered and uncovered piles of whole broadleaf trees for energy use. *Biomass Bioenergy* **2011**, *35*, 454–463. [CrossRef]
26. Liu, Z.; Haygreen, J.G. Drying rates of wood chips during compression drying. *Wood Fiber Sci.* **1985**, *2*, 214–227.
27. Yoshida, T.; Sasaki, H.; Takano, T.; Sawabe, O. Dewatering of high-moisture wood chips by roller compression method. *Biomass Bioenergy* **2010**, *34*, 1053–1058. [CrossRef]
28. Laurila, J.; Havimo, M.; Lauhanen, R. Compression drying of energy wood. *Fuel Processing Technol.* **2014**, *124*, 286–289. [CrossRef]
29. Stahl, M.; Bentz, M. High-pressure treatment of wood-combination of mechanical and thermal drying in the "I/D Process". *Chem. Eng. Technol.* **2004**, *27*, 1216–1221. [CrossRef]
30. Frodeson, S.; Berghel, J.; Ståhl, M.; Granström, K.; Romlin, C.; Thelander, A. The potential for a pellet plant to become a biorefinery. *Processes* **2019**, *7*, 233. [CrossRef]
31. Ciesielski, S.; Adu, E.O.; Mozejko-Ciesielska, J.; Pokoj, T.; Wilke, A. Wood juice valorization through production of poly(3-hydroxybutyrate- co -3-hydroxyvalerate) using *Bacillus* sp. G8_19. *ACS Sustain. Chem. Eng.* **2021**, *9*, 16870–16873. [CrossRef]
32. Wilke, A.; Jochum, J.; Turad, S. EBIPREP–Effiziente Nutzung von Biomasse zur schadstoffarmen Erzeugung erneuerbarer Energie und biotechnologischer Wertstoffe. *Chem. Ing. Tech.* **2020**, *92*, 1262. [CrossRef]
33. Bohnert Technik GmbH. Pressen statt Föhnen: Die Hackschnitzelquetsche: Effiziente mechanische Trocknung im industriellen Dauerbetrieb: Innovativ, wirtschaftlich, robust. Available online: http://www.hackschnitzelquetsche.de/index.html (accessed on 29 December 2021).
34. Kompetenz-und Informationszentrum Wald und Holz (KIWUH) bei der Fachagentur Nachwachsende Rohstoffe e V. *Basisdaten Wald und Holz 2019*; Kompetenz-und Informationszentrum Wald und Holz (KIWUH) bei der Fachagentur Nachwachsende Rohstoffe e V: Gülzow, Germany, 2019. Available online: https://www.fnr.de/fileadmin/Projekte/2019/Mediathek/Basisdaten_KIWUH_web_2te_Auflage_Okt_mio.pdf (accessed on 30 December 2021).
35. *DIN EN ISO 18134-2:2017-05*; Solid Biofuels-Determination of Moisture Content-Oven Dry Method-Part 2: Total Moisture-Simplified Method (ISO 18134-2:2017). Beuth Verlag GmbH: Berlin, Germany, 2017.
36. *DIN EN ISO 21656:2021-06*; Solid recovered fuels-Determination of ash content (ISO 21656:2021). Beuth Verlag GmbH: Berlin, Germany, 2021.
37. *DIN 19268:2021-10*; pH-Measurement of Aqueous Solutions with pH Measuring Chains with pH Glass Electrodes and Evaluation of Measurement Uncertainty. Beuth Verlag GmbH: Berlin, Germany, 2021.
38. *DIN ISO 15705:2003-01*; Water Quality-Determination of the Chemical Oxygen Demand Index (ST-COD)-Small-Scale Sealed Tube Method (ISO 15705:2002). Beuth Verlag GmbH: Berlin, Germany, 2003.
39. *German Waste Water Ordinance-Verordnung über Anforderungen an das Einleiten von Abwasser in Gewässer (Abwasserverordnung-AbwV): AbwV*; Bundesministerium für Umwelt, Naturschutz und Reaktorsicherheit: Berlin, Germany, 1997.
40. *DIN 22022-2:2001-02*; Solid Fuels-Determination of Contents of Trace Elements-Part 2: ICP-OES. Beuth Verlag GmbH: Berlin, Germany, 2001.
41. *DIN 22022-6:2001-12*; Testing of Solid Fuels-Determination of Trace Elements-Part 6: Evaluation and Declaration of Results. Beuth Verlag GmbH: Berlin, Germany, 2001.
42. *DIN EN ISO 11885:2009-09*; Water Quality-Determination of Selected Elements by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (ISO 11885:2007). Beuth Verlag GmbH: Berlin, Germany, 2009.
43. *VDI 4630*; Fermentation of Organic Materials: Characterisation of the Substrate, Sampling, Collection of Material Data, Fermentation Test. Beuth Verlag GmbH: Berlin, Germany, 2016.

44. Sailer, G.; Eichermüller, J.; Poetsch, J.; Paczkowski, S.; Pelz, S.; Oechsner, H.; Müller, J. Optimizing anaerobic digestion of organic fraction of municipal solid waste (OFMSW) by using biomass ashes as additives. *Waste Manag.* **2020**, *109*, 136–148. [CrossRef] [PubMed]
45. Dietz, E.; Kuptz, D.; Blum, U.; Schulmeyer, F.; Borchert, H.; Hartmann, H. *Qualität von Holzhackschnitzeln in Bayern: Gehalte ausgewählter Elemente, Heizwert und Aschegehalt*; Berichte aus dem TFZ No. 46; Technologie- und Förderzentrum im Kompetenzzentrum für Nachwachsende Rohstoffe (TFZ): Straubing, Germany, 2016. Available online: https://www.tfz.bayern.de/mam/cms08/service/dateien/tfz_bericht_46_holzhackschnitzel.pdf (accessed on 22 December 2021).
46. Sailer, G.; Silberhorn, M.; Eichermüller, J.; Poetsch, J.; Pelz, S.; Oechsner, H.; Müller, J. Influence of digester temperature on methane yield of organic fraction of municipal solid waste (OFMSW). *Appl. Sci.* **2021**, *11*, 2907. [CrossRef]
47. Szaja, A.; Montusiewicz, A.; Lebiocka, M.; Bis, M. A combined anaerobic digestion system for energetic brewery spent grain application in co-digestion with a sewage sludge. *Waste Manag.* **2021**, *135*, 448–456. [CrossRef]
48. Jung, B.; Roffael, E. Über die Acidität einheimischer Holzarten. *Eur. J. Wood Prod.* **2002**, *60*, 154. [CrossRef]
49. Werkelin, J.; Lindberg, D.; Boström, D.; Skrifvars, B.-J.; Hupa, M. Ash-forming elements in four Scandinavian wood species part 3: Combustion of five spruce samples. *Biomass Bioenergy* **2011**, *35*, 725–733. [CrossRef]
50. Feldmeier, S.; Wopienka, E.; Schwarz, M.; Schön, C.; Pfeifer, C. Applicability of fuel indexes for small-scale biomass combustion technologies, Part 1: Slag formation. *Energy Fuels* **2019**, *33*, 10969–10977. [CrossRef]
51. Feldmeier, S.; Wopienka, E.; Schwarz, M.; Schön, C.; Pfeifer, C. Applicability of fuel indexes for small-scale biomass combustion technologies, Part 2: TSP and NOx emissions. *Energy Fuels* **2019**, *33*, 11724–11730. [CrossRef]
52. Sommersacher, P.; Brunner, T.; Obernberger, I. Fuel indexes: A novel method for the evaluation of relevant combustion properties of new biomass fuels. *Energy Fuels* **2012**, *26*, 380–390. [CrossRef]
53. Bhatia, S.P.; Letizia, C.S.; Api, A.M. Fragrance material review on alpha-terpineol. *Food Chem. Toxicol.* **2008**, *46* (Suppl. 11), S280–S285. [CrossRef]
54. Hassan, S.B.; Gali-Muhtasib, H.; Göransson, H.; Larrson, R. Alpha terpineol: A potential anticancer agent which acts through suppressing NF-κB signalling. *Anticancer Res.* **2010**, *30*, 1911–1919. [PubMed]
55. Kaura, T.; Sharmaa, P.; Guptac, G.K.; Ntie-Kangd, F.; Kumara, D. Treatment of tuberculosis by natural drugs: A review. *Plant. Arch.* **2019**, *19*, 2168–2176.
56. Shu, H.; Chen, H.; Wang, X.; Hu, Y.; Yun, Y.; Zhong, Q.; Chen, W.; Chen, W. Antimicrobial activity and proposed action mechanism of 3-Carene against *Brochothrix thermosphacta* and *Pseudomonas fluorescens*. *Molecules* **2019**, *24*, 3246. [CrossRef]
57. Martins, R.C.C.; Alegrio, L.V.; Castro, R.X.; Godoy, R.L.O. Constituents of the essential oil of *Eugenia nitida* Camb. (Myrtaceae). *J. Essent. Oil Res* **1999**, *11*, 724–726. [CrossRef]
58. Ho, C.-L.; Liao, P.-C.; Wang, E.I.-C.; Su, Y.-C. Composition and antifungal activities of the leaf essential oil of *Neolitsea parvigemma* from Taiwan. *Nat. Prod. Commun.* **2011**, *6*, 1357–1360. [CrossRef] [PubMed]
59. Yang, J.; Choi, W.-S.; Kim, J.-W.; Lee, S.-S.; Park, M.-J. Anti-inflammatory effect of essential oils extracted from wood of four coniferous tree species. *J. Korean Wood Sci. Technol.* **2019**, *47*, 674–691.