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Process simulation of co-HTC of sewage sludge and food waste digestates and supercritical water gasification of aqueous effluent integrated with biogas plants

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ABSTRACT

The objective of this article is to investigate the integration of a digestate treatment with a biogas plant processing sewage sludge and food waste via process simulations: co-HTC of mixed digestates and supercritical water gasification (SCWG) of the aqueous effluent. The optimum co-HTC conditions are selected based on the energetic yields, comparing relative equipment sizes besides the hydrochar product. The selected conditions are 200 °C, 30 % solid load, and 1-h residence time for the mixing ratios in scope: energetic yields of 3.58–3.59 MJ/kg reactor inlet. These conditions result in more than 60 % K, P, and N recovery on hydrochar. SCWG of the aqueous effluent provides complete mineral recovery in the solid form and surplus energy production through syngas while causing some nitrogen loss as N₂ gas. Although the co-HTC data is calculated from individual HTC results, the synergetic effect on the energetic yield does not affect the selection of optimum conditions as investigated through co-HTC of sewage sludge and food waste (the origins of the digestates). Consequently, biogas plants can evolve into multi-product biorefineries through the proposed integration. Meanwhile, this study can guide future co-HTC experiments of food waste and sewage sludge digestates and reduce the required runs.

1. Introduction

Solid waste management has become a significant concern due to increasing waste amounts related to population growth, widespread urbanization, and the massive consumption of resources [1–7]. Two major wastes, sewage sludge (SS) and food waste (FW), are directly related to the population. According to FAO (The Food and Agriculture Organization of the United Nations), the global generation of FW and SS are 1.6 billion tonnes and 45 million dry tons per year, respectively [8, 9]. SS and FW are complex wastes containing proteins, lipids, and carbohydrates. Moreover, SS contains high ash and harmful contaminants, such as pharmaceuticals, heavy metals, and microplastics [10,11]. Similarly, conventional disposal of FW produces greenhouse gases, foul odor, and leaching [12,13]. Therefore, it is crucial to reduce and reuse waste through effective strategies.

Organic wastes are widely treated via anaerobic digestion (AD) to produce biogas: deriving 25 % of all bioenergy from biogas is possible based on EU policy estimates [14,15]. Increasing the application of AD implies increasing digestate discharge (a by-product of AD). Statistics for

EU28 pointed to approximately 180 million tons of digestate generation annually [16]. Therefore, the valorization of the digestate becomes a significant concern [17,18]. Furthermore, nutrient recovery is crucial in waste management: phosphorus and nitrogen as the basis of fertilizers. However, supply problems can occur due to limited phosphorus resources and the energy-intensive production of nitrogen-based fertilizers [19]. Efficient recovery of critical elements is essential regarding the sustainability of food supply and circular economy [20,21]. Aragón-Briceño et al. suggested hydrothermal carbonization (HTC) as a prominent option for enhanced nutrient recovery [22].

HTC gains attention due to its capability of treating wet biomass, cost-effectiveness, efficient energy consumption, and less toxicity [23–25]. It produces a coal-like, energy-dense hydrochar product at 180–250 °C temperatures in autogenous pressure [26,27]. Six companies have already applied the HTC process on an industrial scale for nitrogen and phosphorus recovery, mainly processing SS and integrating it with wastewater treatment plants [22]. Some other concepts include producing hydrochar within the ISO/TS17225-8 standards from FW via HTC coupled with AD [28] and energy recovery from SS via integrating HTC and aqueous phase reforming [29]. Reviewing sustainable

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List of abbreviation

Anaerobic digestion	AD	Higher heating value	HHV
Biochemical methane potential	BMP	Hydrothermal carbonization	HTC
Combined heat and power	CHP	Mixing ratio	MR
Chemical oxygen demand	COD	Predictive Soave-Redlich-Kwong	PSRK
Co-hydrothermal carbonization	Co-HTC	Process water	PW
Carbon retention	CR	Retention	R
Energy yield	EY	Synergistic coefficient	SC
Food waste	FW	Synergistic effect	SE
Food waste digestate	FWD	Supercritical water	SCW
Heavy metals	HMs	Supercritical water gasification	SCWG
		Sewage sludge	SS
		Sewage sludge digestate	SSD

management of food waste digestate (FWD), HTC was stated to be the most promising valorization option, including direct application to land, composting, and pyrolysis [30]. Therefore, HTC of various digestates are investigated for integrating biogas plants [31].

Since biogas plants typically process multiple wastes, HTC of mixed digestates (co-HTC) is essential and results in a synergistic effect (SE). Co-hydrothermal carbonization (co-HTC) is a favorable technique to improve the hydrochar properties, compared to HTC of a single feedstock, elevating the yield, higher heating value (HHV), and thermal behavior of hydrochar [32]. For instance, adding 50 % cellulose or hemicellulose to SS increased HHV by 111–117 % and reduced the ash content from 71.2 % to 34.7–48.5 % [33]. Co-HTC of SS and model compounds of FW resulted in only 20 % of nitrogen remaining in hydrochar [34]. The Co-HTC of SS and banana stalk improved hydrochar properties and migration of heavy metals (HMs) [35]. The feedstock mixing ratio acted as an important parameter affecting the hydrochar yield, HHV, and energy yield (EY). Increasing the SS to FW ratio showed an upward trend in hydrochar fuel properties and combustion performance.

Similarly, adding lignocellulosic biomass improved the yield and properties of hydrochar produced via co-HTC of SS and lignocellulosic biomass [36]. Kavindi et al. [37] applied hydrochar from co-HTC of rice straw and sewage sludge digestate (SSD) to remediate solid form Cr(VI) from agricultural soil, observing better microbial activity with the rice straw:SSD mass ratio of 1:1. Zhao et al. [38] observed hydrochar characteristics improved because of the SE of distillers grains and SS: fuel ratio, activity, HHV, and combustion efficiency of the hydrochar increasing with the proportion of distillers grains. To sum up, the effects of co-HTC are different and highly dependent on the composition of the feedstock combinations. Nutrient-rich SS and FW are related to population and urbanization, thus implying many large-capacity biogas plants are processing these wastes. For instance, a biogas plant in Finland (Stormossen) processes FW and SS in different digesters, thus having two digestates [39].

Meanwhile, more experimental studies on co-HTC of FWD and SSD must be conducted. However, conducting co-HTC experiments in a vast combination range of mixing ratios, temperature, residence time, and solid load would be too costly and time-consuming. Therefore, the promising sets of conditions can be selected for the experimental verification by using individual HTC results as a preliminary assessment.

HTC discharges a high amount of process water (PW) containing considerable dissolved organic compounds, minerals, and nutrients. PW contains about 15 % of the feedstock energy, 20–50 % of biomass organics, and high nutrients such as phosphorus and nitrogen [40–42]. Nutrient recovery supports the sustainable usage of resources and reduces environmental pollution by replacing conventional fertilizers. In other words, PW needs further treatment due to containing toxic compounds and needing to recover nutrients [43]. The first option is watering the fields nearby after treating PW to remove the organic content. However, this might cause overdosing of the soil with the same element

recovered dominantly in PW [44]. Another option is recirculating PW to the HTC process [45] or the AD reactor [46]. However, circulating the PW to the AD reactor introduces a risk since biological processes are sensitive to toxic compounds and heavy metals [47] despite the potential to increase biogas yield.

Meanwhile, circulating the PW to the HTC reactor increased the hydrochar mass yield and HHV without damaging the carbon content in hydrochar [48,49]. Some studies achieved a 5–10 % increase in hydrochar mass yield [50] and up to 15 % increase in the hydrochar energy yield [51] by recirculating PW to the HTC process. In addition, PW recirculation reduces wastewater production [43] and is an effective method for heat recovery [52]. This option is suitable when operating with a low solid load, i.e., diluting the dewatered digestate. However, the optimum reaction conditions might not require dilution of the digestate feedstock. For instance, the optimum conditions of HTC were determined as 30 % solid load when counting relative equipment size and energy requirements [53]. Alternatively, the minerals can be recovered effectively through precipitation or electrochemically [54, 55]. Meanwhile, dissolved organics are still to be treated after these methods. In other words, there is a need for PW treatment to recover minerals and nutrients in a solid form as well as energy recovery utilizing the organic content.

Considering its high water content, PW can be treated through another hydrothermal process. Supercritical water gasification (SCWG) is a promising technology that produces syngas while recovering the minerals and nutrients in a solid outlet. This method has great importance both in energy production and fertilizers. SCWG occurs at higher temperatures and pressure than the critical point of water (374 °C and 22.1 MPa). SCW becomes a suitable solvent for organics and gases but has no solubility for salts [56]. The impact of pressure is relatively minor when operating at 23–29 MPa [57,58]. Temperature is usually around 400–500 °C for catalytic SCWG (e.g., ruthenium and nickel) and higher without a heterogeneous catalyst [59]. The syngas yield increases with temperature and residence time, while too long residence time can cause char formation via repolymerization [60–62]. A main operational issue is the risk of reactor plugging due to char formation and solid deposition [63].

Nevertheless, char formation is reduced since a process's aqueous effluent contains smaller molecules than the primary feedstock [64]. Some studies investigated the integration of SCWG with the HTC process for various feedstocks, e.g., SS and FW, e.g., enhanced syngas production and complete recovery from the digestate stream [65–67]. In addition, a SCWG reactor enabling solid separation (e.g., configuration proposed by Ghavami et al. [63]) would provide the recovery of char, including minerals and nutrients. The char outlet can be transferred to the fields nearby or far away, like hydrochar.

This study aims to construct and simulate a process integration concept valorizing the digestates of a biogas plant processing SS and FW to reach circularity. The integrated process cases involve co-HTC of the digestates with or without SCWG of PW, enabling complete circularity

or only digestate treatment with a relatively low investment cost. The optimum conditions for co-HTC of SSD and FWD are selected based on combining the individual HTC results of each digestate due to the lack of experimental co-HTC data of these digestates. This selection helps obtain preliminary results of the proposed concept and directs future co-HTC experiments towards promising sets of conditions.

2. Material and method

2.1. The integration concept and the selection of optimum co-HTC conditions

The scope of this study is to integrate a co-HTC process into a biogas plant, processing SS and FW in parallel. The biogas plant generates digestates dewatered to 30 % solid content. The proposed integration scenario is depicted in Fig. 1. The aqueous effluent (i.e., PW) is the outlet stream of the simulated process when integrating only co-HTC. The other scenario is the co-HTC of digestates and SCWG of PW: recovering the nutrients and minerals in solid products and producing syngas from the organic content in PW.

The co-HTC data is generated based on the individual HTC results of the digestates. Supplementary Material 1 provides the complete list of experimental HTC results from various studies and generated co-HTC data at the matching conditions [6,31,36,68–77]. The co-HTC data cover the range of 150–250 °C, 10–30 % solid load, and 0.5–2 h of residence time. Table 1 shows the analysis of digestates used in generating co-HTC data.

The optimum conditions are selected based on the energetic yields of co-HTC data. The energetic yield is defined as the energy content of hydrochar per unit mass of the reactor inlet [53]. The co-HTC hydrochar yields and heating values are calculated as the mass-weighted average of individual results, as shown in Equations (1) and (2), where MR represents the mixing ratio. Then, the energetic yields are calculated, as shown in Equation (3). Finally, the co-HTC conditions resulting in the maximum energetic yield are selected for the simulations.

$$co-HTC Yield (\%) = \frac{(Yield_{SSD} \times MR_{SSD}) + (Yield_{FWD} \times MR_{FWD})}{MR_{SSD} + MR_{FWD}} \quad (1)$$

$$co-HTC HHV \left(\frac{MJ}{kg}\right) = \frac{(Yield_{SSD} \times HHV_{SSD} \times MR_{SSD}) + (Yield_{FWD} \times HHV_{SSD} \times MR_{FWD})}{(Yield_{SSD} \times MR_{SSD}) + (Yield_{FWD} \times MR_{FWD})} \quad (2)$$

$$Energetic yield \left(\frac{MJ}{kg \text{ reactor inlet}}\right) = \frac{(co-HTC Yield) \times (co-HTC HHV) \times Solid \text{ load} (\%)}{100 \times 100} \quad (3)$$

Table 1
Analysis of the selected digestate samples (dry basis).

Digestate	HHV (MJ/kg)	C (%)	H (%)	N (%)	S (%)	O (%)	Ash (%)	Ref.
SSD1	14.9	28.9	3.2	3.4	1.5	16.1	46.9	[31]
SSD2	14.4	33.3	4.6	4	1.2	20.3	36.7	[68]
SSD4	11.5	30.3	4.2	3.5	2.3	18.8	40.9	[6]
SSD7	14.3	29.6	4.3	4.4	1.6	20.1	40.1	[36]
FWD1	14.9	29.5	3.0	2.0	0.3	21.3	43.9	[31]
FWD3	19.7	46.0	6.5	2.7	0.3	36.6	8.0	[75]
FWD4	13.4	34.3	4	1.9	0.2	23.8	35.8	[76]

As an important parameter, the SE is evaluated by the synergistic coefficient (SC), comparing between the experimental co-HTC results and calculated values from the individual HTC results. This study introduces SC on the energetic yield (i.e., the proposed selection criteria) to evaluate the reliability of calculated co-HTC data as shown in Equation (4): where Energetic yield_{experimental} and Energetic yield_{calculated} represents the values calculated from the experimental co-HTC data and from combining the individual HTC data, respectively. However, due to the lack of experimental co-HTC data on SSD and FWD, the co-HTC data on SS and FW reported by Zheng et al. [78] are used to investigate the SC on energetic yield concerning temperature and mixing ratios.

$$SC \text{ on energetic yield} (\%) = \frac{Energetic \text{ yield}_{\text{experimental}} - Energetic \text{ yield}_{\text{calculated}}}{Energetic \text{ yield}_{\text{calculated}}} \times 100 \quad (4)$$

2.2. Process simulation

This study investigates the process integration to a biogas plant generating 300 kg/h SSD and 300 kg/h FWD on a dry basis. To investigate the possible capacity increase and SE effect, the co-HTC simulations involve the mixing ratios of SSD:FWD as.

- 1:1 (300 and 300 kg/h dry)
- 1:3 (300 and 900 kg/h dry)
- 3:1 (900 and 300 kg/h dry)

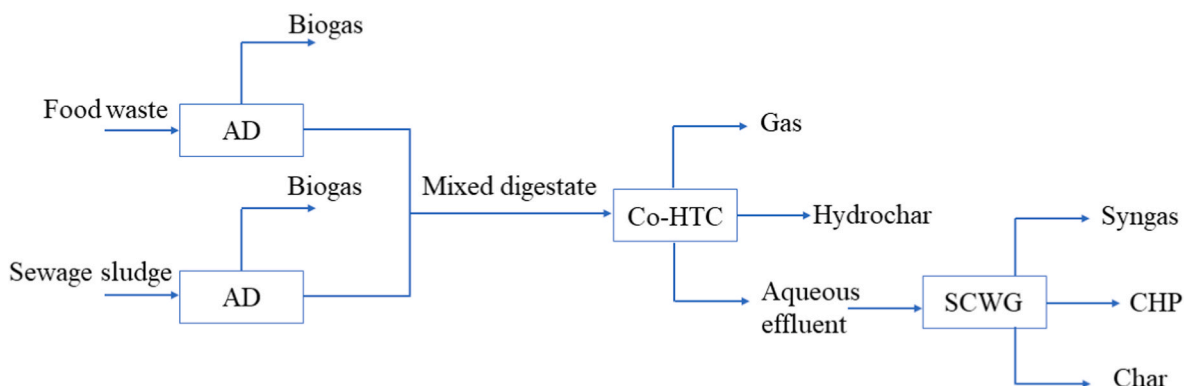


Fig. 1. Process integration block diagram.

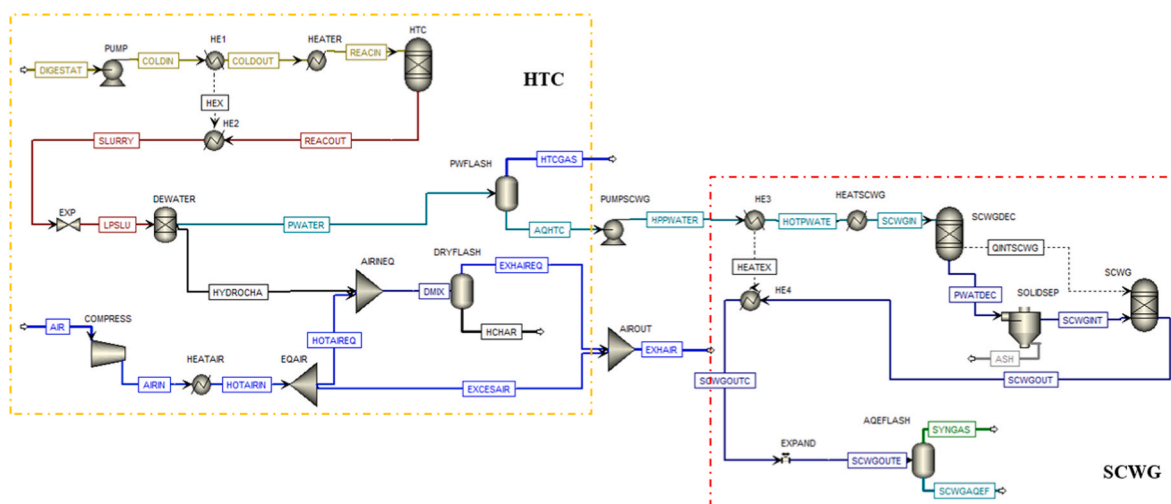


Fig. 2. Process simulation of Co-HTC and SCWG.

Fig. 2 shows the integrated processes simulated in Aspen Plus V11.1. The co-HTC section consists of pressurizing and heating the feedstock, reactor, dewatering, and thermal drying the hydrochar down to 20 % moisture content. The simulation starts with pumping and two-step feedstock heating to the reaction conditions. The first heating step is the heat exchange with the reactor outlet ('REACTOUT'), and the second is the external heat ('HEATER'). The reactor operates at a constant temperature, i.e., isothermal reactor, and is represented by a RYIELD block. After heat exchange with the feedstock ('HE1' and 'HE2'), the reactor outlet is conducted to the dewatering unit ('DEWATER') to separate hydrochar and PW, reducing the hydrochar moisture content to 50 % with the energy requirement of 79.09 kJ/kg dry solid [79]. Some portion of hydrochar remains in the PW, calculated based on the dewatering concept [79]. Finally, the hydrochar is dried by hot air ($P = 1.2$ bar, $T = 110$ °C) to reach 20 % moisture. Since industrial dryers operate with excess air, the heat requirement of thermal drying is assumed to be 3.82 MJ/kg water evaporated [80]. Therefore, the air flowrate ('AIR') is adjusted so that the heat duty of the 'HEATAIR' unit corresponds to the mentioned requirement. The air flow is split as the equilibrium ('HOTAIR') and the excess air ('EXCESAIR'). The outlet drying streams are the hydrochar product ('HCHAR') and the exhaust air ('EXHAIR'). When integrating only co-HTC, the stream 'PWATER' is an outlet stream. For the 1:1 mixing ratio, the simulation continues with the SCWG of PW, as shown in Fig. 2. The 'PWATER' stream is conducted to the flash separation to remove gases ('PWFLASH') and pressurized to 250 bars ('PUMPSCWG'). Afterwards, the stream is exposed to heat exchange with the reactor outlet ('HE3' and 'HE4') and external heating ('HEATSCWG') to reach the desired temperature. Since the SCWG reaction is endothermic, the SCWG inlet is heated to 625 °C, and the outlet of an adiabatic reactor is at 600 °C. The SCWG reactor is first simulated with a yield reactor to decompose non-conventional components into elemental ones ('SCWGDEC'). After separating the ash in the solid separator ('SOLIDSEP'), the remaining components are conducted to the SCWG reactor represented by a RGIBBS block ('SCWG'), determining the outlet by minimizing Gibbs free energy. After cooling down via heat exchange with the PW, the SCWG outlet stream is expanded to 1 bar ('EXPAND') and conducted to the flash separator to separate syngas and aqueous phase ('AQEFLASH').

The component list and thermodynamic property method are selected similarly to the previous article on HTC of agricultural residue digestate [53]. The components include water, digestate, and hydrochar as dry-ash-free, ash, dissolved organics, CO_2 , H_2 , CH_4 , CO , N_2 , O_2 , H_2S , and NH_3 . Ash is an inert and a separate component to facilitate the mass balance. Additionally, the dissolved organics in PW are defined as a single, non-conventional component. The mixed digestates, organics,

and hydrochar are defined as dry-ash-free, non-conventional components. The physical properties of non-conventionals are estimated via 'HCOALGEN' and 'DCOALIGT'. The heating values of digestates and hydrochars are calculated as the mass-weighted average of the individual heating values, while the heating value of ash is introduced as 0. Heating values of dissolved organics are calculated based on a correlation valid for a wide range of substances [53]. The thermodynamic property method is selected as Predictive Soave-Redlich-Kwong (PSRK), except the RYIELD and RGIBBS reactors representing the SCWG reactor simulated with the IDEAL method.

At the selected co-HTC conditions and flow rates of each digestate, conducting a mass balance using the experimental HTC results determines the reactor yields (per unit mass of non-inert) and ultimate and proximate analysis of non-conventionals. Supplementary Material 2 shows the individual HTC mass balances and the co-HTC balance at the selected conditions and digestates. The mass balance is conducted through the following steps.

- Hydrochar, liquid, and gas flow rates based on total mass balance and yield data
- Nitrogen elemental balance to determine the nitrogen content in PW
- Gas flow rates calculated based on the assumed mole fractions as CO_2 : 0.9, H_2 : 0.01, CH_4 : 0.03, CO : 0.06 [81].
- Assuming no ash, sulfur, and nitrogen in gas product [82].
- Carbon and sulfur mass flows in PW calculated via elemental balances
- Hydrogen and oxygen contents of organics in the PW estimated based on chemical oxygen demand (COD) and biochemical methane potential (BMP)

The simulation results are evaluated regarding the products and energy requirements. The recovery measures are carbon retention and the recovery of fertilizer elements (K, P, N). The retention of elements can be calculated as shown in Equation (5) [83]. The recovery of each component can be calculated similarly, i.e., the percentage of an element in hydrochar relative to the carbon in the feedstock.

$$\text{Retention (R)} = \frac{\text{Mass of element in hydrochar}}{\text{Mass of element in feedstock}} \times 100 \quad (5)$$

The energy requirements are obtained from the simulation results. Electricity is required to operate the pumps pressurizing the reactor inlets ('PUMP' and 'PUMPSCWG'), the dewatering ('DEWATER'), and the air compressor in the HTC section ('COMPRESS'). External heat is required to heat the feedstock after heat exchange with the co-HTC reactor outlet ('HEATER'), to heat the air introduced for hydrochar

drying ('HEATAIR'), and to heat the SCWG reactor inlet after heat exchange with the outlet ('HEATSCWG'). The heat duty in HEATSCWG heater is 25 % electricity and 75 % heat requirement due to high-temperature heating compromising the combined heat and power (CHP) production via steam. The split is based on the conventional CHP plants with steam turbines: 25 % electricity, 60 % heat, and 15 % heat loss [84]. Moreover, CHP production via syngas can fulfill the energy requirement. Similarly, the energy generation from syngas combustion is 25 % electricity and 60 % heat. Meanwhile, the co-HTC reactor releases heat when operating isothermally because of exothermic reactions, thus causing a negative heat requirement.

3. Result and discussion

3.1. Selection of optimum co-HTC conditions

Selecting the optimum conditions plays a crucial role in the economic performance. Determining optimum conditions at the early stage of process development is beneficial. However, the impacts of conditions are comprehensive and interdependent. In particular, temperature has the opposite effect on the hydrochar yield and heating value: the yield decreases with temperature while the heating value increases. Moreover, increasing residence time slightly improves the yield and heating value [6,75]; however, increasing residence time can also decrease the yield at long residence times (e.g., more than 2 h) or high temperatures [75,76].

Furthermore, it is crucial to compare the relative equipment size and energy requirement, i.e., implicitly the investment and operation costs. The residence time directly affected the reactor size. In addition, the equipment size is directly influenced by the solid load of the reactor inlet, not counted in dry-based or dry-ash-free yields. The impact of solid load on the equipment size is very dominant compared to a slight effect on the product. Therefore, stating the need for a comprehensive methodology, Özdenkçi et al. (2020) [61] evaluated the conditions of SCWG based on hydrogen and energy yields with the basis of a non-inert reactor inlet.

Similarly, Ghavami et al. (2022) [53] introduced the energetic yield as the energy content of hydrochar per unit mass of the reactor inlet. The energetic yield is the first criterion when selecting the optimum conditions: compiling the impacts of conditions on the product yield, heating value, and relative equipment size. When different sets of conditions give close energetic yields, the other criteria are the residence time (comparing the reactor size), the reactor material, and the catalyst load [53,61].

The co-HTC of FWD and SSD is simulated at the optimum conditions with maximum energetic yields. Table 2 shows the energetic yields concerning the conditions and mixing ratios. The optimum energetic yields are obtained as 3.59, 3.58, and 3.59 MJ/kg reactor inlet for the

SSD1:FWD1 ratios of 1:1, 1:3, and 3:1, respectively, at 200 °C with 30 % solid load and 1-h residence time. Meanwhile, it is worth noting that the proper comparison can be made when the specific digestates of a biogas plant are exposed to co-HTC experiments, considering that the feedstocks at different studies are not identical. Nevertheless, this study is still helpful for selecting the sets of promising conditions and introducing the comparison methodology.

Due to the SE of different constituents, the feedstock types and mixing ratio are important factors influencing the hydrochar yields and heating values [32,35]. For instance, a co-HTC review listed SCs on hydrochar yield and CR for various mixed wastes, e.g., swine manure-sawdust, textile-waste paper, waste-textile-FW, and SS-pine wood [85]. However, no experimental data exists on the co-HTC of FWD and SSD. Nevertheless, the mixture of SS and FW can cause a similar synergetic impact with the mix of SSD and FWD, as the origins of these digestates. An investigation on co-HTC of SS and FW presented experimental results at 180–280 °C and 7 % solid load with the SS:FW ratios of 70:30, 50:50, and 30:70 [78]. Increasing the SS ratio resulted in higher hydrochar yield due to higher inorganic content: the highest yield with 70 % SS. Meanwhile, FW contributed to the increase in the product heating value.

Since this study selects the optimum conditions based on the energetic yield, the SC on the energetic yield indicates the role of SE in the optimum conditions. Therefore, Fig. 3 shows the SCs on the energetic yields for the experimental results of co-HTC of SS and FW presented by Zheng et al. (2019) [78]. The complete data is given in Supplementary Material 3, including the digestate analysis, hydrochar yields, and heating values. It can be seen that the SC decreases with temperature, except for 70 % SS feedstock at 280 °C. The SCs are 10.2–11.9 % at 180 °C and reduce to negative values at higher temperatures.

Nevertheless, SC on the energetic yield does not affect the choice of optimum temperature, except for 70 % SS feedstock. Furthermore, the SC would potentially be very close to zero at 200 °C, i.e., SE introducing only a minor impact on the energetic yields. Therefore, it can be expected that the optimum conditions of co-HTC of SSD and FWD can be selected by combining the individual HTC data for the preliminary investigations, which enables the selection of promising conditions for future co-HTC experiments.

3.2. Mass balances

The co-HTC reactor yields are calculated by using the data on the defined mixing ratios (SSD1:FWD1 as 1:1, 1:3, 3:1) and the selected conditions (200 °C, 30 % solid load, and 1 h for all the mixing ratios), presented by Parmar and Ross [31]. The properties of non-conventionals are also determined through these calculations, as shown in Supplementary Material 2. These are introduced as inputs to the simulation models. Table 3 shows the feedstock and hydrochar properties with

Table 2

The co-HTC results and the energetic yields for the SSD:FWD ratios of 1:1, 1:3 and 3:1, respectively.

Feedstock	Conditions			Hydrochar		Process performance	
	T (°C)	Solid load (%)	t _{res} (h)	Yield (% dry)	HHV(MJ/kg)	Energetic yield (MJ/kg reactor inlet)	
SSD1:FWD1	200	10	1	76.1 77.8 74.3	14.89 14.85 14.94	1.13	1.15 1.11
		20		78.5 79.7 77.3	15.10 15.10 15.10	2.37	2.41 2.33
		30		78.9 79.4 78.5	14.99 15.02 15.27	3.59	3.58 3.59
	250	10	68.4 69.7 67.0	15.15 14.89 15.09	1.02	1.04 1.01	
		20	69.7 70.5 68.8	15.09 15.00 15.20	2.10	2.12 2.09	
		30	71.6 72.3 70.6	15.34 15.17 15.52	3.29	3.31 3.28	
SSD7:FWD1	150	20	1	89.8 89.4 90.2	15.10 15.05 15.15	2.71	2.69 2.73
	200			79.0 79.9 78.0	15.15 15.12 15.17	2.39	2.42 2.37
	250			71.0 71.1 70.7	15.05 14.97 15.12	2.13	2.13 2.14
SSD1:FWD3	200	10	1	57.9 50.6 65.3	18.77 21.48 16.67	1.09	1.09 1.09
SSD2:FWD3	250	10	0.5	54.8 44.6 64.9	19.42 22.47 17.33	1.06	1.00 1.12
SSD4:FWD4	210	15	2	83.8 81.5 86.0	11.64 11.76 11.52	1.46	1.44 1.48
	230			75.2 74.0 76.5	11.94 12.16 11.71	1.35	1.35 1.34
	250			69.9 68.9 71.0	12.44 12.57 12.32	1.31	1.30 1.31

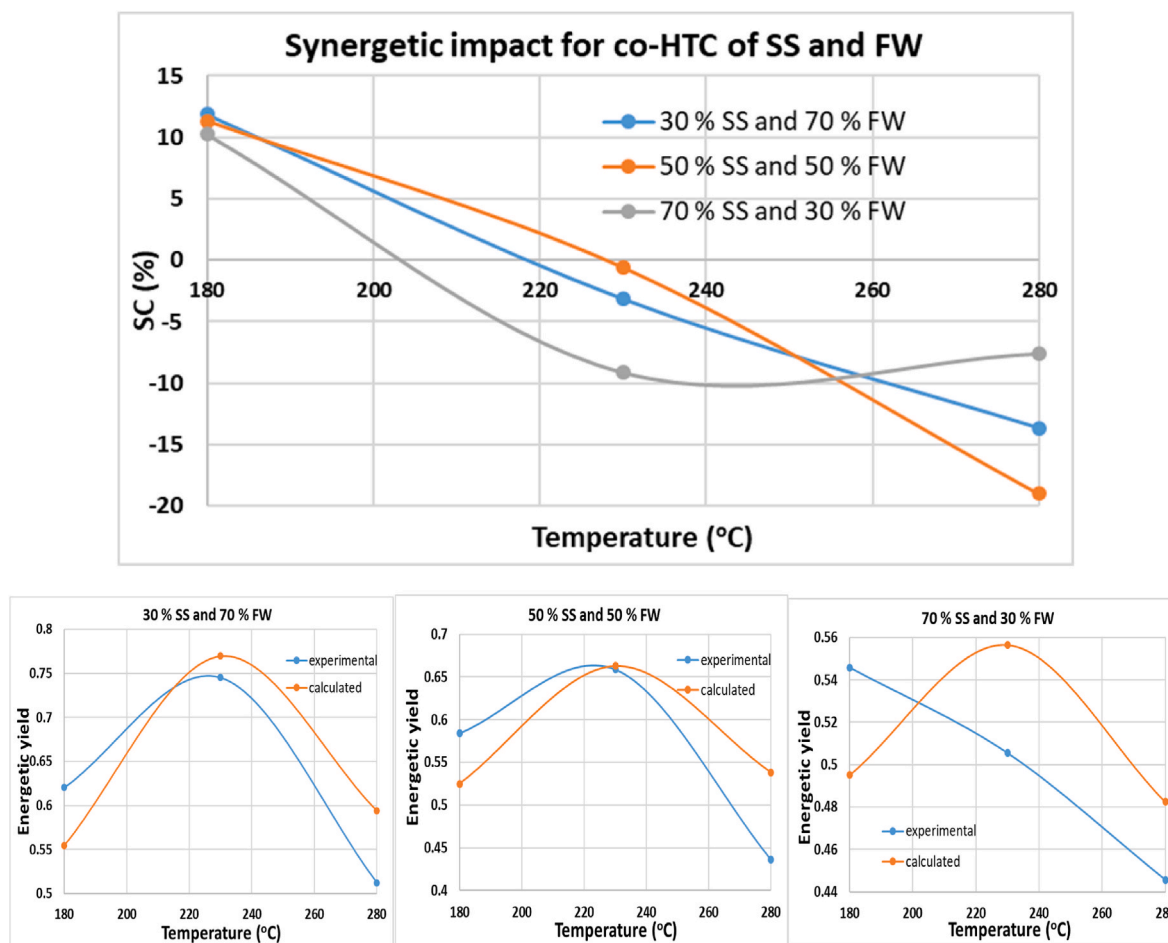


Fig. 3. The energetic yields (MJ/kg reactor inlet) and SE on the energetic yields (%).

Table 3
Feedstock and hydrochar properties (daf).

Material		C (%)	H (%)	O (%)	N (%)	S (%)	HHV(MJ/kg)
1:1	Digestate	53.48	5.68	34.25	4.94	1.65	27.29
	Hydrochar	62.73	7.14	24.67	4.13	1.34	29.31
1:3	Digestate	53.03	5.51	36.13	4.24	1.08	26.92
	Hydrochar	63.28	6.83	25.43	3.56	0.89	30.27
3:1	Digestate	53.95	5.85	32.31	5.67	2.23	27.67
	Hydrochar	62.21	7.42	23.94	4.68	1.75	28.40

Table 4
The elemental analysis and heating values of organics.

SSD:FWD ratio	Dissolved organics (dry-ash-free)					
	C (%)	H (%)	O (%)	N (%)	S (%)	HHV (MJ/kg)
1:1	24.84	2.61	58.03	10.76	3.76	4.29
1:3	28.61	2.96	57.35	8.79	2.29	6.70
3:1	19.86	2.15	58.92	13.37	5.70	1.10

different mixing ratios. Table 4 gives the elemental analysis and heating value of the dissolved organics. Table 5 reports the co-HTC reactor yields introduced in the RYIELD block in kg/kg non-inert.

An essential aspect of processing the experimental data is prioritizing the characterization data and determining the simulation inputs [53]. For instance, the mass balance in this study reveals a mismatching carbon amount in the organics when calculated through carbon elemental balance or nitrogen elemental balance and the C:N ratio of PW, i.e., affecting the hydrogen and oxygen calculations and causing

Table 5
Calculated yields in kg/kg non-inert.

Component	SSD:FWD ratio		
	1:1	1:3	3:1
Hydrochar	0.140	0.136	0.145
Water	0.809	0.809	0.808
Organics	0.033	0.038	0.029
CO ₂	0.017	0.016	0.017
H ₂	8.479E-06	8.291E-06	8.668E-06
CH ₄	0.0002	0.0002	0.0002
CO	0.0007	0.0007	0.0007

errors in elemental balances. Therefore, this study conducts mass balance with the priority as follows: total mass balance to define the flow rates, digestate and hydrochar analysis to identify the feed and product, assumed gas composition, elemental balances around the reactor to determine elemental contents of PW, and finally using COD and BMP data to determine the content of hydrogen and oxygen in the dissolved organics and water amount in the PW stream. The ultimate and proximate analysis on solid samples is reliable because drying at low temperatures avoids further decomposition. In contrast, CHNS analysis of PW can involve higher uncertainty since light organics can evaporate during sample drying. The accuracy can be improved further through a more profound analysis of PW and ash. Ash is considered inert despite including some inorganic carbon.

Meanwhile, equilibrium reactions also convert inorganic carbon to carboxylates [86]. Consequently, it is beneficial regarding accuracy to utilize the most reliable characterization data first. The mass balance

results are sufficient for the preliminary assessment despite requiring further improvements.

After determining the inputs, the process is simulated to determine the mass and energy balances. Table 6 shows the flowrates following the block diagram depicted in Fig. 4. The digestate flowrate is based on the mass balance calculation (determined in Supplementary Material 2), and the rest is extracted from the simulation. The mass balance indicates several aspects of the process. Comparing the reactor inlet and outlet shows that water participates in the reactions. A small amount of water is consumed in the SSD:FWD ratios of 1:1 and 3:1 while being generated to a smaller extent in the mixing ratio of 1:3. Additionally, SSD causes slightly more gas formation than FWD while FWD results in slightly more dissolved organics. Dissolved organics is another aspect since PW has 4.7–5.4 % of organics, i.e., potential for further recovery. Furthermore, Table 6 indicates that most ash is recovered within the hydrochar, which encourages the usage of hydrochar as a fertilizer.

After flash separation, the PW is conducted to SCWG, removing the dissolved gases for the mixing ratio 1:1. The stream information is reported in Table 7. The mass balance indicates that 61 kg/h water is consumed in SCWG reactions, thus contributing to the formation of syngas. This observation is consistent with the experimental studies observing hydrogen and oxygen gasification efficiencies as more than 100 %, i.e., elements of water being converted besides the solid content [86]. The carbon representing char is observed in negligible amounts in the simulation. It is beneficial to process the aqueous effluent in SCWG to reduce the char formation, rather than the original feedstock, because it includes dissolved and smaller molecules. Meanwhile, the SCWG reactor may not reach equilibrium, and unhydrolyzed content (e.g., hydrochar in PW) may cause a char outlet. Therefore, SCWG results are to be validated through experimenting with the specific stream, although simulations are helpful for preliminary assessment.

The mineral and nutrient recovery is improved through the ash outlet of the SCWG reactor. In the case of a reactor configuration enabling solid separation, the minerals can be recovered entirely because of no solubility in SCW. Meanwhile, nitrogen recovery introduces a challenge due to distribution into different phases. Nitrogen gas is observed due to ammonia decomposition as an equilibrium reaction. The PW includes 9.2 kg/h nitrogen element. According to the SCWG results, most nitrogen element is converted into gas, 8.85 kg/h of N₂. Despite observing fewer amounts, the experimental studies also reported nitrogen gas formation. For instance, a survey of SCWG of SS

Table 6
Mass flows of the co-HTC process in kg/h.

Stream	Component	SSD:FWD ratio		
		1:1	1:3	3:1
DIGESTATE	Digestate	327.6	664.2	646.2
	Ash	272.4	535.8	553.8
	Water	1400	2800	2800
HTCOUT	Hydrochar	242.75	470.66	500.34
	Ash	272.4	535.8	553.8
	Water	1396.95	2803.1	2784.7
	Organics	57.30	130.44	98.76
PWATER	Gases	30.60	60.00	62.40
	Water	999.43	2000.44	1997.27
	Ash	45.35	57.86	123.54
	Hydrochar	72.34	145.94	143.20
HC SLUDGE	Organics	57.30	130.44	98.76
	Gases	30.60	60.00	62.40
	Water	397.52	802.66	787.43
	Ash	227.05	477.94	430.26
AIR	Hydrochar	170.41	324.72	357.14
	Air	17550	35450	34760
EXHAUSTAIR	Air	17550	35450	34760
	Water	298.43	603.7	591.02
HC PRODUCT	Water	99.09	198.96	196.41
	Ash	227.05	477.94	430.26
	Hydrochar	170.41	324.72	357.14

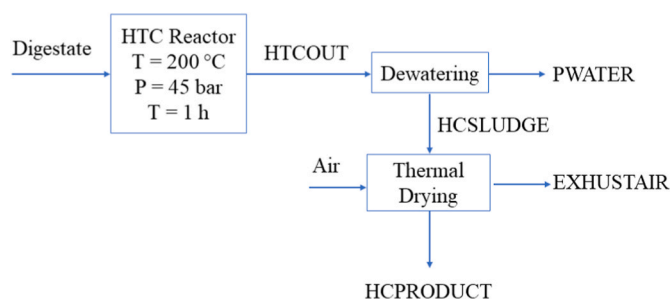


Fig. 4. The block diagram of the integrated co-HTC process.

Table 7
Mass flows in the SCWG section for 1:1 SSD:FWD ratio.

Component	Flow rates (kg/h)		
	HPPWATER	SYNGAS	SCWGAQEF
Water	997.71	51.06	885.79
Ash	45.35	-	-
Hydrochar	72.34	-	-
Organics	57.30	-	-
CO ₂	0.62	143.40	0.15
CO	0.0009	2.14	0.0001
CH ₄	0.0004	26.27	0.002
H ₂	1.26e-5	6.63	0.0004
N ₂	-	8.85	0.0003
H ₂ S	-	3.31	0.01
NH ₃	-	0.17	0.19

observed 10–20 % nitrogen recovery in the solid phase, the majority in liquid, and significant amounts in gas [87]. Thus, a considerable portion of nitrogen is converted into gas, although the ammonia decomposition might not reach equilibrium. Nitrogen transformation depends on the feedstock composition during the HTC and SCWG processes. Nitrogen retention rate is higher in co-HTC of SS with different feedstocks, including FW and garden waste, due to various interactions between organic waste and SS [41]. In the co-HTC of SS and FW model compounds, about 67 % of the nitrogen is transformed to the PW in the form of NH₄⁺ and organic-N, and only 20 % to the hydrochar, resulting in a 36–60 % rise in nitrogen retention [34].

Additionally, nitrogen transformation is affected by temperature: 47.3 % of nitrogen transformed to the aqueous product at 160 °C, while 69.2 % transformed at 250 °C during HTC of SS [88]. In this study, about 38 % of the nitrogen in the digestate is transferred to the dissolved organics, i.e., inlet to the SCWG reactor. Table 8 reports the retentions in hydrochar for carbon, potassium, phosphorus, and nitrogen. It can be seen that increasing SSD promotes carbon, potassium, and nitrogen retention; however, FWD promotes phosphorus retention.

Regarding the usage of hydrochar from co-HTC of SSD and FWD, its utilization as a fertilizer is potentially more suitable than energetic usage. The heating values of hydrochar products are relatively low due to high ash content, causing a longer time for complete combustion [89]. Meanwhile, the hydrochar recovers most the nutrients and minerals, thus making it feasible to transport to the agricultural fields. The remaining nutrients and minerals in PW can also be recovered as a solid product, e.g., through SCWG, as in this study or a precipitation process.

Table 8
Retention of different elements.

Retention (%)	SSD-FWD ratio		
	1:1	1:3	3:1
Carbon	86.91	84.56	89.28
Potassium	66.97	63.97	70.77
Phosphorus	67.23	75.70	62.92
Nitrogen	61.93	59.35	63.92

3.3. Energy balance

The energy balances are obtained from the simulation results. Table 9 and Table 10 present the energy requirements of the co-HTC process for all the mixing ratios and the SCWG section for the 1:1 mixing ratio, respectively. The energy requirements in the co-HTC section are proportional to flow rates with only minor deviations regarding the mixing ratio, except for the reactor. Contrarily, the reactor heat duty differs sharply depending on the mixing ratio: the heat of reaction increases with SSD proportion. From the heat integration viewpoint, the released heat from the reactor can sufficiently cover the heat required for thermal drying in all the mixing ratios. Heating the feedstock requires different utility due to the temperature range; nevertheless, the heat released from the reactor exceeds the sum of this requirement and the need in drying, except the SSD:FWD ratio of 1:3. The SCWG of PW provides more energy production. The net heating value of the syngas is 9.03 MJ/kg. The CHP production from syngas covers most of the power consumption and provides extra heat. The net power requirement reduces from 124.9 to 16 kW and increases the net heat production by 331 kW.

It is essential to determine the heat of reaction and verify it with the literature. It can be determined through Hess's law (the enthalpy change between reactants and products) or by calculating the system's exchanged thermal power [90]. The calculated heat duty of the reactor is reported in Table 9, which is equivalent to -2.56 , -2.12 , and -2.90 MJ/kg_{dry-solid} and -4.69 , -3.83 , -5.39 MJ/kg_{dry-ash-free-solid} for 1:1, 1:3, and 3:1 SSD:FWD ratios, respectively. There is a lack of information about the heat of reaction of co-HTC of SSD and FWD; nevertheless, the obtained heat of reactions is within the reported ranges for FW (-1.19 MJ/kg_{dry-solid}), SS (-2.62 MJ/kg_{dry-solid}), organic waste (-7.30 MJ/kg_{dry-solid}), and glucose (-1.06 MJ/kg_{dry-solid}) [90–92]. The heat of reaction differs depending on the severity of conditions and the feedstock composition. For instance, the experimental condition for SS was 250 °C and 20 h residence time [91].

The energy balance calculations are functional for preliminary analysis despite uncertainties in non-conventional properties. The possible errors due to non-conventional properties are minimized since water dominates the streams. For instance, the duties of heat exchangers involve a minor uncertainty because of the specific heat of non-conventionals calculated via the empirical correlations meant for coal. Moreover, the dissolved organics introduce uncertainty in its heating value. Therefore, the results help select the optimum conditions and feasibility assessments. At the same time, further improvements can include validating of energy balances on a pilot scale and a more accurate representation of non-conventional properties, e.g., temperature dependence of specific heats.

3.4. Operational aspects and simulation limitations

Although this study provides a valuable methodology to select the optimum conditions and process simulation models, it is worth noting

Table 9
Co-HTC energy requirements.

	SSD-FWD ratio		
	1:1	1:3	3:1
Electricity requirement (kW)			
PUMP	3.5	7	7
COMPRESS	110.0	222.3	217.9
DEWATER	11.4	22.1	23.2
Total	124.9	251.4	248.1
Heat requirement (kW)			
HAETER	40.45	80.8	80.9
HEATAIR	316.26	638.8	626.4
CO-HTC	-427.2	-706.8	-967.1
Total	-70.49	12.8	-259.8

Table 10
SCWG energy balance.

Electricity requirement (kW)		Heat requirement (kW)	
HTC Section	124.9	HTC Section	-70.49
PUMPSCWG	31.5	-	-
0.25 × HEATSCWG	11	0.75 × HEATSCWG	33
Electricity outcome (kW)	-	Heat outcome (kW)	-
0.25 × SYNGAS	151.68	0.6 × SYNGAS	364.05

the limitations: verification of synergetic impact, stream rheology, and SCWG limitations, including solid recovery, thermodynamic method, and reaching the equilibrium state.

The reliability of the generated co-HTC data is verified by using the experimental co-HTC of the origins (SS and FW) due to the lack of data on the co-HTC of these digestates. The SCs on the energetic yield are near zero around the optimum temperature, and the waste types have similar constituents. Meanwhile, verifying the synergetic impacts of those digestates through the co-HTC experiments is essential.

Stream rheology is important for biorefinery design, specifically in piping, heat requirement, and pumping. Slurries with more than 20 % solid load behave like non-Newtonian fluids [93]. It depends on the feedstock, particle size, and sludge concentration, and the energy requirement increases with sludge concentration [94]. This study simulates co-HTC integration with a 30 % solid load. If the feedstock slurry is too viscous to pump, the process can be integrated with a lower solid load (e.g., 20 %). However, lowering the solid load will proportionally increase the energy requirements and equipment sizes due to the increased flow rates. Another alternative is transporting the feedstock through screwdrivers and modifying the heat integration, as shown in Fig. 5. This increases the external heat requirement and requires extra power for the screwdriver while saving investment and operation costs through relatively smaller equipment sizes.

The simulation of SCWG assumes reaching equilibrium. The minerals accelerate some reactions in the conversion mechanism, e.g., water gas shift reaction and steam reforming, and suppress the char formation [59]. The feasible residence time of SCWG is relatively short, e.g., up to 5 min, due to high pressure causing high reactor cost [61,95]. Meanwhile, the decomposition of intermediates might be kinetic-limited. Moreover, char is negligible in the simulation results; however, the unhydrolyzed portion of organics can form char, of which the

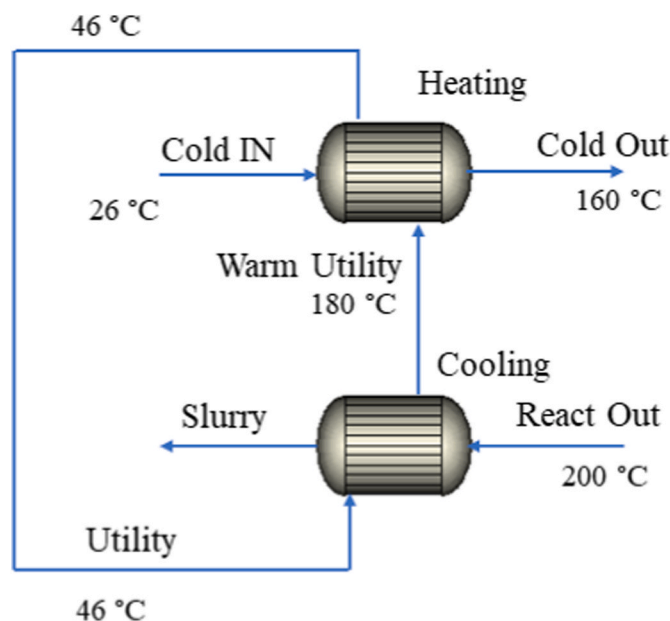


Fig. 5. Alternative heat integration for a viscous slurry [53].

gasification is kinetic-limited. In addition, ammonia decomposition depends on the residence time and might not reach equilibrium, e.g., less nitrogen loss in syngas. Therefore, the results of SCWG of PW can be experimentally verified as a further investigation. Another simulation limitation is the assumption of complete solid recovery. This requires a particular reactor configuration, e.g., multiple riser tubes for gas outlets while precipitating the solids [63].

Despite high pressure and polar compounds, the SCWG reactor units are simulated with the IDEAL method. However, several other methods implied exothermic reactions. Only the IDEAL method provided reasonable results regarding temperature, dropping from 625 to 600 °C. This is consistent with the simulations of SCWG of black liquor conducted using the PSRK method, resulting in a 15 °C temperature drop [95].

4. Future aspects

Co-HTC of FWD and SSD is an innovative approach for waste management and energy production. Integrating the co-HTC process with SCWG enables nutrient recovery and syngas production. This study is a preliminary assessment of integrating co-HTC of SSD and FWD followed by SCWG of PW with biogas plants. It can enlighten the further process development steps.

- Experimental data

Simulation studies provide a practical preliminary assessment, while experiments can verify the simulations. First, experiments at promising condition sets can address the lack of experimental data on the co-HTC of SSD and FWD. The experiments can cover a range of conditions around the selected optimum values: e.g., 180–220 °C, 0.5–1.5 h, and 20–30 % solid load. Furthermore, experiments on the SCWG of PW are needed for more accurate syngas and solid outlets. The accuracy can be improved through deeper characterization of ash and PW and temperature dependences of physical properties of non-conventional components.

- Feedstock variation and co-processing with other wastes

SSD and FWD have various compositions affecting the process efficiency and product. The particular waste should be specifically experimented with when developing the process integration. Furthermore, a biomass supply chain requires multi-feed-multi-product processes to enable a circular economy [96]. Therefore, a future aspect can involve another waste into the SCWG or the co-HTC reactor. This can increase the capacity and improve the economic performance but requires co-processing experiments [97].

- Commercial viability and scale-up

The proposed integration has shown promise at the lab-scale and industrial implementations. HTC is applied commercially by several companies, i.e., being economically feasible [41,98]. Similarly, SCWG was stated as economically feasible even for a more challenging feedstock (black liquor), provided the operational issues are addressed effectively [95]. SCWG introduces high investment costs due to high flow rates and pressure [99]. The integration of co-HTC can be an option when preferring lower investment costs. Meanwhile, SCWG of PW enables a high amount of CHP production and complete recovery of the minerals despite increasing the investment cost.

5. Conclusion

This study investigates integrating co-hydrothermal carbonization of digestates and supercritical water gasification of process water with a biogas plant processing sewage sludge and food waste in parallel. The

hydrochar can be used as a fertilizer, while syngas can provide CHP production. The optimum co-hydrothermal carbonization conditions are selected based on the energetic yields, comparing the relative equipment sizes besides the product yield and quality. The co-hydrothermal carbonization data is generated from the individual HTC data of each digestate due to the lack of co-hydrothermal carbonization experimental data on sewage sludge digestate and food waste digestate. The results indicated that the optimum co-hydrothermal carbonization conditions were 200 °C, 1-h residence time, and 30 % solid load. These conditions provide remarkable nutrient recovery on the hydrochar: 64–71 % potassium, 63–76 % phosphorus, and 59–64 % nitrogen recoveries.

Furthermore, supercritical water gasification of process water provides further mineral and nutrient recovery in solid form, which is beneficial for logistics. Additionally, using syngas for combined heat and power production provides surplus energy. Meanwhile, supercritical water gasification sharply increases the investment cost, i.e., it is an effective option for large-capacity plants.

Despite the uncertainties, this study is helpful for preliminary assessment and directing the co-hydrothermal carbonization experiments towards promising ranges of conditions. Two main uncertainty factors are the heat of reaction in the co-hydrothermal carbonization and the synergistic effect of mixing feedstocks. The heat of reaction from the simulations (−2.56, −2.12, and −2.90 MJ/kg_{dry-solid} and −4.69, −3.83, −5.39 MJ/kg_{dry-ash-free-solid} for 1:1, 1:3, and 3:1 sewage sludge digestate and food waste digestate ratios, respectively) matches with the literature values for similar feedstocks and model compounds. Similarly, this study investigates the synergistic coefficient on the energetic yields for co-hydrothermal carbonization of sewage sludge and food waste due to a lack of data on co-hydrothermal carbonization of sewage sludge digestate and food waste digestate. The synergistic coefficient decreases with temperature in all mixing ratios and is close to zero at the selected co-hydrothermal carbonization temperature. This investigation determines that the SE has no significant impact on choosing the optimum conditions. Therefore, future co-hydrothermal carbonization experiments can involve narrow ranges of conditions around the selected optimum (e.g., 180–220 °C, 20–30 % solid load, and 0.5–1.5 h residence time) rather than numerous experiments.

In conclusion, biogas plants can be evolved into multi-product biorefinery facilities through the hydrothermal treatment of digestate. Hydrothermal carbonization is the option for producing hydrochar with a relatively low investment cost, followed by supercritical water gasification of process water providing energy production and further recovery of nutrients.

CRediT authorship contribution statement

Niloufar Ghavami: Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Data curation, Conceptualization. **Karhan Özdenkçi:** Writing – review & editing, Writing – original draft, Validation, Supervision, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Cataldo De Blasio:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.energy.2023.130221>.

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