energies

Review

Thermal Disintegration of Sewage Sludge as a Method of Improving the Biogas Potential

Sylwia Myszograj * and **Ewelina Płuciennik-Koropczuk**

Institute of Environmental Engineering, University of Zielona Góra, 65-001 Zielona Góra, Poland
* Correspondence: s.myszograj@iis.uz.zgora.pl; Tel.: +48-683282574

Abstract: Operating and research experiments indicate that the potential benefits of thermal treatment of sewage sludge before methane fermentation include increasing the biodegradability of substrates, reducing the amount and improving the dewaterability of the fermentate and its hygienization, reducing the emission of odours during stabilization, higher production of biogas, and improving the energy balance of the process. The process of disintegration (liquefaction) can be carried out, for example, through the use of mechanical homogenization, microwaves and ultrasonic waves, chemical agents, thermal methods, and biological processes. The article reviews the literature data on thermal hydrolysis research, from the first source information to the present. The thermal hydrolysis achieved enhanced hydrolysis, biogas potential, and faster sludge degradation during anaerobic digestion without compromising the quality of the end products.

Keywords: wastewater treatment plant; sewage sludge; thermal disintegration; hydrolysis; biogas potential

Citation: Myszograj, S.; Płuciennik-Koropczuk, E. Thermal Disintegration of Sewage Sludge as a Method of Improving the Biogas Potential. *Energies* **2023**, *16*, 559. https://doi.org/10.3390/en16010559

Academic Editor: Attilio Converti

Received: 27 November 2022
Revised: 21 December 2022
Accepted: 28 December 2022
Published: 3 January 2023

1. Introduction

Methane fermentation is a multi-stage process, and its main phases are hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The first stage of biodegradation of organic substrates, which is to allow the transport of nutrients inside a cell, consists of the depolymerization of macromolecular biopolymers into monomers. Microbial biomass consists of a variety of biopolymers. Dry weight of microbial cells is composed of protein (52.4%), polysaccharides (16.6%), lipid (9.4%), RNA (15.7%), and DNA (3.2%) [1]. It is important to note that the mentioned biopolymers contribute more than 95% of the total dry weight of microbial cells. Apart from the bacterial cells, 80–95% of the organic matter in the activated sludge floc consists of various types of organic material. The extracellular polymeric substances (EPS) fraction is the largest fraction (40 to 60% of the organic matter) and it consists of polysaccharides, proteins, lipids, nucleic acids, humic substances, and various heteropolymers [2]. Depolymerization is a process in which the monomers are the product. If the polymer decomposition process takes place with the generation of other products, it is called degradation. Polymer degradation can be caused by various factors, including light, temperature, as well as mechanical or chemical factors, and it always leads to significant changes in their physical and chemical properties. The degradation of biopolymers may be accompanied by their liquefaction. In the description of reactions occurring in technological processes of sewage sludge processing in aerobic (Activated Sludge Model) [3] and anaerobic (Anaerobic Digestion Model) [4] conditions, this stage is called disintegration. Disintegration consists in liquefying substrates such as dead microorganisms, preliminary sludge, excessive sludge to products in the form of molecular sugars, proteins, and fats, as well as other fractions which are most often non-degradable (inert).

The hydrolysis phase is a limitation factor for the duration of anaerobic decomposition [5–9]. Sewage sludge can be pre-treated (disintegrated) before methane fermentation.

In such a case, the process is much faster than in conventional systems, the degree of decomposition of organic substances is higher, and, consequently, the production of biogas also increases. Accelerating the liquefaction process of organic compounds into soluble forms also significantly improves the efficiency of the following stages of the process [10–12]. Another advantage is the possibility of using sewage sludge disintegration methods to obtain volatile fatty acids in order to use them as a source of easily digestible organic carbon in sewage treatment processes, including biological dephosphatation and denitrification [13–15].

Disintegration methods are divided into:

- Thermal disintegration, carried out in the temperature range from 40 to 180 °C [16,17];
- Chemical disintegration with the use of ozone, acids, or solutions [18,19];
- Mechanical disintegration using ultrasound, mills, and homogenizers, in which forces acting on the walls of microorganism cells cause stress and deformation of cells with simultaneous enzymatic hydrolysis of organic compounds [20,21];
- Biological disintegration [22,23], consisting of autolytic hydrolysis under the influence of enzymes produced by microorganisms or enzymes dosed into the system from outside;
- Freezing and melting sludge, irreversibly changing the structure of flocculation, by increasing the volume of water during freezing [24];
- Advanced oxidation processes [25].

The line between classical methods and disintegration methods is not precisely defined. Some techniques that simultaneously use different factors to enable faster hydrolysis, such as temperature, alkalization, and ultrasound, are difficult to categorize into one group of methods [26,27].

Sewage sludge is a good source of carbon, nutrients, and trace elements. When subjected to appropriate treatment before stabilization, it is an even better raw material for energy and raw material recovery. This process is most often supported by thermal disintegration, which significantly improves the characteristics of sewage sludge without compromising the quality of the end products. The article reviews the literature data on thermal hydrolysis research, from the first source information to the present. The various thermal pretreatment methods have been discussed based on sludge solubilization efficiency, and improvement in biogas yield.

2. Fundamentals of Thermal Disintegration

A clear increase in interest in the thermal disintegration of sewage sludge was initiated in the 1960s in connection with searching for highly efficient methods of dewatering [28]. Examples of technical solutions developed during this period consist in the Zimpro and Proteous processes. Most of these installations operated in the temperature range from 180 to 250 °C. Due to an increase in energy costs associated with technical and process problems (odours), these installations worked only until the early 1980s [29,30].

Currently, the methods of thermal disintegration are divided into [31]:

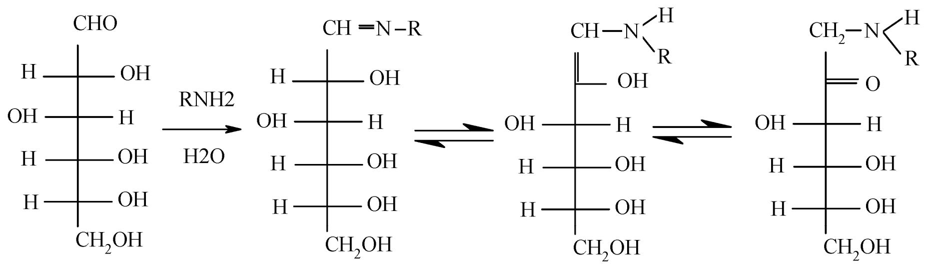
- Low-temperature, occurring at temperatures below 100 °C (35–100 °C);
- High-temperature, occurring at temperatures above 100 °C (100–275 °C).

Research regarding optimization of the process of thermal disintegration of sewage sludge focuses mainly on accelerating hydrolysis of the molecular fraction of sewage sludge, producing volatile fatty acids, biochemical hydrolysis of the remaining and not liquefied molecular fraction of sewage sludge, and increasing the amount of biogas and methane potential of sewage sludge in the methane fermentation process. Thermal treatment of sewage sludge is also used to produce an external carbon source for the denitrification process [13–15].

Bajernbruch et al. [16] also observed a reduction in foaming in anaerobic stabilization chambers of thermally disintegrated sludge (121 °C, 60 min.). In the case of thermally disintegrated preliminary and excessive sludge, their susceptibility to dewatering is increased [28,31,32]. Assuming an increase in dry weight as the assessment criterion, the literature data show that the optimal temperature for the process of thermal hydrolysis of sewage sludge should be in the range of 150 to 175 °C [33,34].

Li and Noike [35] tested the thermohydrolysis of sewage sludge in the temperature range from 120 to 175 °C for over 30 min. They found that the thermal treatment process had an impact on the liquefaction of the solid fraction to carbohydrates, proteins, and fats, and then to volatile fatty acids. However, the transformations of organic components are not always similar. Bougrier et al. [36], as well as Wilson and Novak [37], showed a greater impact of thermal hydrolysis on polysaccharides than on proteins. The authors suggest that polysaccharides are polymers associated with the outer structure of the solid sludge fraction, while proteins are intracellular components. Therefore, only disintegration at high temperatures leads to destroying the cell wall and releasing body protein. The structure of flocculation and cells of microorganisms is determined by electrostatic phenomena, ionic and hydrogen bonds, and probably only partial destruction of cells occurs at temperatures below 100 °C [38]. One difficulty for studying the mechanism of proteins degradation with denaturation temperatures above 100 °C is that the heat denaturation of proteins is usually irreversible at temperatures higher than 80 °C, because under these conditions proteins generally aggregate after heat denaturation [39].

Thermal treatment results in a hydrolysate with the characteristic color of "strong tea" also containing, in addition to hydrolyzed biodegradable compounds, other ingredients such as Amadori rearrangement products (early stage Maillard reaction products), or melanoids (final products), which are potential inhibitors of methane fermentation [40]. The Maillard reaction is a complex reaction between amino acids and reducing sugars, occurring at increased temperatures. The initial reaction step for, e.g., glucose and amino acid (RNH_2) can be presented as follows:



(1)

Glucose Shiff base Amadori compounds

Amadori compounds are easily polymerized into three different structures that can react in subsequent phases [40].

Typical Maillard reaction products are formed at 150 °C, reaching a quantitative maximum at 250 °C within 10 min [5]. Polymerization of these products most likely occurs at higher temperatures and longer reaction times, resulting in macromolecular compounds (Figure 1). Peterson et al. [41] determined the degradation kinetics of glucose and glycine during hydrothermal hydrolysis. The results show that Maillard-type reactions strongly affected the kinetics at 250 °C.

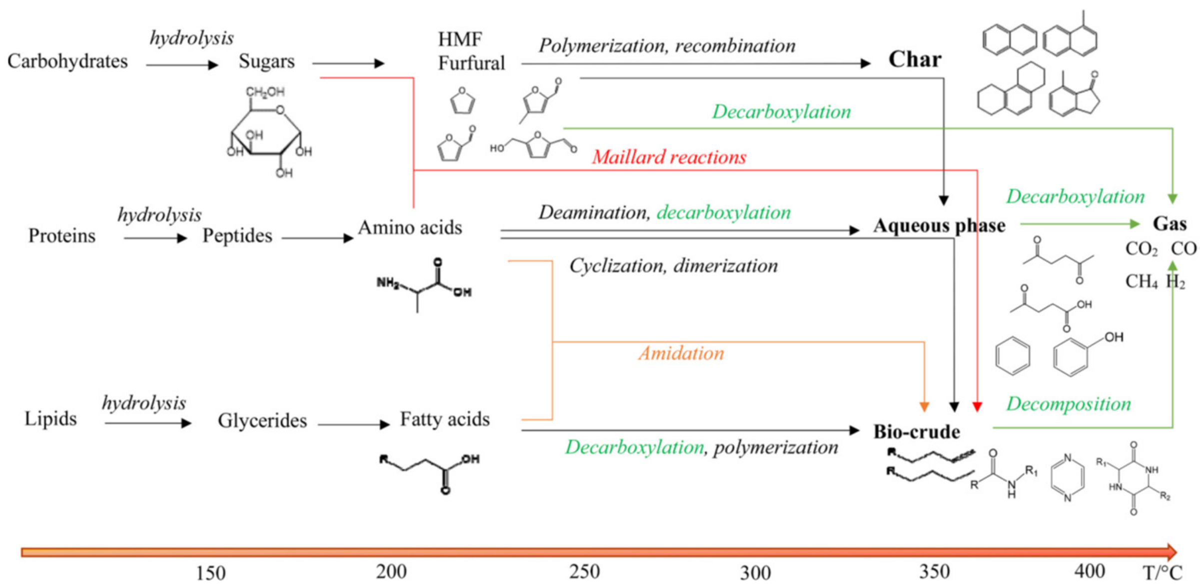


Figure 1. General reaction network for thermal disintegration of sewage sludge, [5] (Elsevier Ltd., CC BY-NC-ND license).

The decomposition of organic substrates causes an increase in the concentration of nitrogen compounds in hydrolysates. Higher processing temperatures appear to have a beneficial effect on the breakdown of fats by forming alkanes and alkenes by decarboxylation, which is a favorable reaction to improve the quality of hydrolysates. However, the formation of amides between proteins and lipids is also observed as an undesirable reaction, as it contributes to the increase of nitrogen compounds in hydrolysates and reduces the efficiency of fatty acid production [5,42,43].

Elbing and Dünnebeil [44] obtained a three-fold increase in the degree of hydrolysis, changing the temperature in the range of 100 to 180 °C, and at the same time showed a three-fold increase in the concentration of total nitrogen and ammoniacal nitrogen, without an increase in the concentration of phosphates. Under similar process conditions, Graja et al. [45] obtained only a 32% increase in the proportion of nitrogen dissolved in hydrolysates, including only 5% in the form of ammoniacal nitrogen. At low temperatures (40–70 °C) over the duration from 1 to 3 h, the degree of liquefaction of nitrogen solids ranged from 0.5 to 3.5% [46].

According to the collected data, the degree of liquefaction of sewage sludge is influenced primarily by temperature, not by the type of sludge. Wilson and Novak [37] examined the impact of disintegration in the temperature range from 130 to 220 °C on the liquefaction of preliminary sludge, obtaining an effect similar to that of excessive sludge. However, in technical conditions, disintegration methods are used almost exclusively for excessive sludge because preliminary sludge quickly undergoes enzymatic hydrolysis in the methane fermentation process. This is confirmed by research carried out by Bougrier et al. [36]. They stated that the share of the solid fraction of sludge decreases with the temperature increase from about 95% d.m. (abbrev. d.m.—dry matter) in the raw sludge to less than 50% d.m. at processing temperatures above 150 °C.

A commonly used indicator for assessing the thermal disintegration process is the change of the proportion of COD-dissolved fraction (Figure 2).

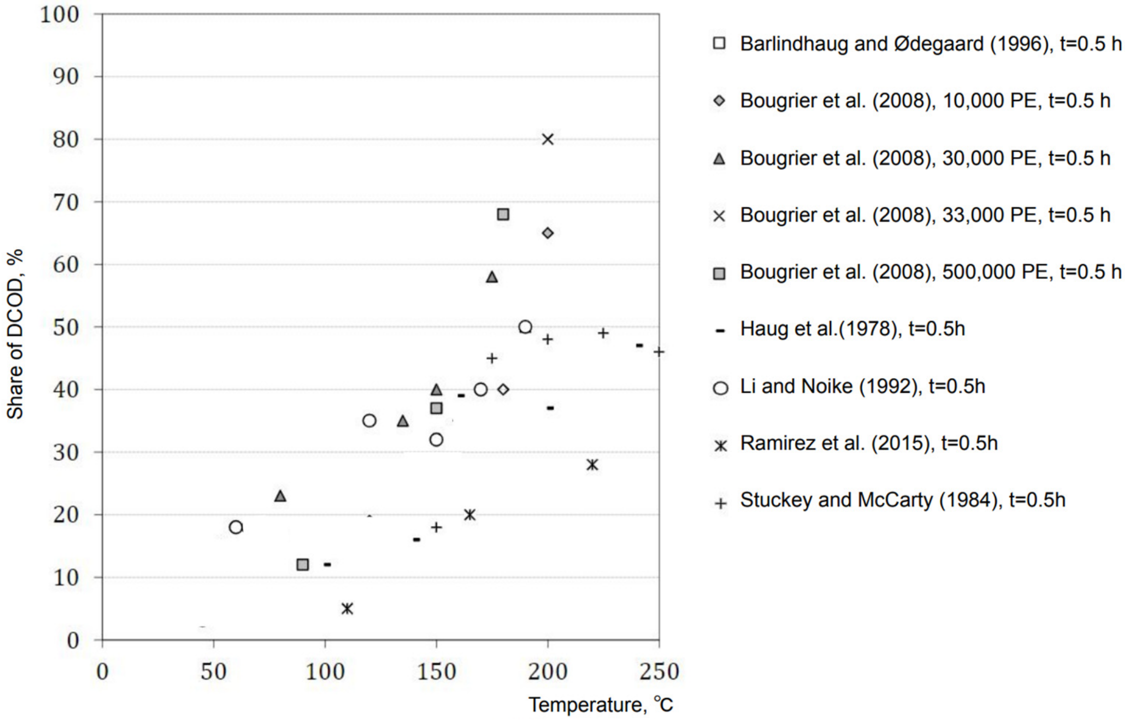


Figure 2. Share of the COD-dissolved fraction in sewage sludge COD depending on the temperature of the disintegration process (summarized literature resource by Author) [14,28,35,36,39,47].

When analyzing the impact of process parameters of experimental data published in the literature, it can be seen that temperature is the main parameter determining the efficiency of thermohydrolysis, while the time of the process is less important [18,36,45,48]. It should be noted that the degree of liquefaction is in most cases linearly dependent on temperature, especially for high-temperature disintegration. Tanaka et al. [18] obtained a 25% increase in the share of COD-dissolved fraction after treatment of excessive sludge at 130 °C for 0.5 h and 30% at the same temperature, when the treatment time was 1 h. At 170 °C, they achieved an increase in the degree of liquefaction at the level of 60% regardless of the disintegration time. Under the same conditions, disintegration of excessive sludge was carried out in the Zipperclave reactor [49,50] over 15, 30, and 60 min. During one-hour processing, the share of dissolved COD increased from 2.7% to 25.3, 43.9, and 59.5%, respectively, at process temperatures of 130, 150, and 170 °C. Bougrier et al. [51], in research at the Zipperclave reactor, also obtained COD fraction liquefaction in the range of 40 to 45% over 30 and 60 min. However, Aboulfoth et al. [52], conducting disintegration at 175 °C, determined an increase in COD liquefaction from 11.2% to 15.1% and 25.1% after increasing the treatment time respectively from 1 to 2 and 3 h.

The thermal treatment processes are associated with the simultaneous mineralization of organic components; however, it is possible to determine the conditions limiting decomposition [45]. Caraballa et al. [53] obtained a reduction in the organic mass of sludge below 5% by disintegrating at 130 °C over 60 min. Yuan et al. [54] demonstrated a significant impact of mixing on the liquefaction of sewage sludge. At temperatures of 4, 14, and 24 °C, they examined an increase in the concentration of volatile fatty acids in stirred and unstirred reactors. In stirred systems, the increase in VFA concentration in hydrolysates was higher for each of the above-mentioned experimental temperatures by 400, 100, and 20%, respectively.

3. Biogas Potential of Thermally Disintegrated Sewage Sludge

The basic objective of the conducted research in many scientific centers consists of determining the impact of thermal disintegration of sludge on the effectiveness of the methane fermentation process [3,5,8,10,15,26,48,49]. Conducting the process at low temperatures

(60–80 °C) has a positive impact on the chemical composition of the obtained hydrolysate, but it requires a longer reaction time (60–120 min) [30]. Especially excessive sludge is susceptible to the impact of low temperatures, below 100 °C [55]. The fermentation of such sludge after thermal hydrolysis takes place much faster than in conventional systems, the degree of decomposition of organic substances is higher, and, consequently, the production of biogas is also higher [56]. Low-temperature disintegration of sewage sludge in the temperature range from 60 to 80 °C was also researched by, for example, Hiraoka et al. [57], as well as Li and Noike [35], achieving an increase of more than 30% in biogas production in the methane fermentation process. Stuckey and McCarty [47] stated that the increase in susceptibility to the biodegradation of sewage sludge results not only from the liquefaction of their solid fraction, but also from the hydrolysis of dissolved compounds.

The impact of low-temperature disintegration of sewage sludge on the efficiency of methane fermentation according to studies by various authors is summarized in Table 1.

Table 1. Impact of low-temperature disintegration of sewage sludge on fermentation efficiency, (PS—Primary sludge, WAS—Waste Activated sludge, HRT—Hydraulic Retention Time).

No.	Sewage Sludge	Temperature and Time of Disintegration	Anaerobic Digestion Conditions	Effects	Source
1	PS	70 °C, 4 d	batch fermentation, 37 °C	CH_4 production increase from 21.2 to 24.7 mmol/g d.m. (16%)	[58]
2	PS	70 °C, 7 d	batch fermentation, 55 °C	CH_4 production increase from 13.7 to 25.5 mmol/g d.m. (16%)	
3	PS	70 °C, 2 d	continuous fermentation, HRT 13 d, 55 °C	CH_4 production increase from 146 to 162 mL/[L·d] (11%)	[59]
4	PS	70 °C, 2 d	continuous fermentation, HRT 13 d, 55 °C	CH_4 production increase from 13.6 to 20.1 mmol/g d.m. (48%)	[60]
5	PS	50–65 °C, 2 d	continuous fermentation, HRT 13 d, 35 °C	CH_4 production increase (25%)	[61]
6	WAS	60–70 °C, 1 d	batch fermentation, 10 d, 37 °C	biogas production increase from 200 to 300 mL/g d.m. (50%)	[62]
7	WAS	70 °C, 7 d	batch fermentation, 37 °C	CH_4 production increase from 8.30 to 10.45 mmol/g d.m. (26%)	[58]
8	WAS	70 °C, 7 d	batch fermentation, 55 °C	CH_4 production 10.9 mmol/g d.m.	
9	WAS	70 °C, 2 d	continuous fermentation, HRT 13 d, 55 °C	CH_4 production increase from 40 to 55 mL/[L·d] (28%)	[58]
10	WAS	70 °C, 9 h	batch fermentation, 55 °C	biogas production increase (58%)	[63]
11	Mixed PS + WAS	70 °C, 9, 24, 48 h	continuous fermentation, HRT 10 d, 55 °C	biogas production increase from 0.15 to 0.18 mL/g d.m. (20%)	[27]

The thermal disintegration processes can also be carried out at temperatures from 100 to 250 °C in pressure reactors. Research in this area was carried out by Haug et al. [64], Pinnekamp [65], Li and Noike [35], Dote [33], Tanaka et al. [18], Kepp et al. [29], Prechtl et al. [66], Guibelin [67], and Carballa et al. [53] (Table 2). Disintegration at temperatures exceeding 150 °C is used for substrates with a higher degree of hydration.

The published research results indicate that the treatment of preliminary sewage sludge at high temperatures (above 100 °C) has no significant impact on improving the efficiency of the methane fermentation process [68]. Haug et al. [28,64] investigated the impact of pre-treatment on the methane-generating potential of various sewage sludge types. The thermal treatment of preliminary sludge at temperatures up to 175 °C had no significant impact on the production of biogas. However, the thermal disintegration of excessive sludge at the same temperatures resulted in an increase in the production of biogas from 30 to 225%, methane by up to 60% and a reduction of at least 30% in the organic mass of stabilized sludge [30,32]. Brooks [69] studied the process of hydrolysis of excessive sewage sludge and its mixtures with preliminary sludge. The best results were obtained by keeping the sludge at a temperature of 165 to 180 °C for a duration of 10 to 30 min. The hydrolysis products were readily degradable in the fermentation process, whereas the disintegration time of sewage sludge did not affect the efficiency of biodegradation as much as the temperature. Similar conclusions were drawn by Dohanyos et al. [43] using disintegration within 1 min at 170 °C.

Table 2. Impact of high-temperature disintegration of sewage sludge on fermentation efficiency, (PS—Primary sludge, WAS—Waste Activated sludge, HRT—Hydraulic Retention Time).

No.	Sewage Sludge	Temperature and Time of Disintegration	Anaerobic Digestion Conditions	Effects	Source
1	PS	175 °C, 30 min	continuous fermentation HRT 15 d, 35 °C	production of CH_4 252 mL/g COD	[28]
2	WAS			CH_4 production increase from 115 to 186 mL/g COD (62%)	
3	WAS			CH_4 production increase from 205 to 234 mL/g COD (14%)	
4	WAS	175 °C, 60 min	continuous fermentation HRT 5 d, 35 °C	biogas production increase from 108 to 216 mL/g COD (100%)	[35]
5	WAS	180 °C, 60 min	batch fermentation, 8 d, 37 °C	CH_4 production increase (90%)	[18]
6	WAS	160 °C	wastewater treatment plant, HRT 15 d	biogas production increase (60%)	[70]
7	WAS	121 °C, 30 min	batch fermentation, 7 d, 37 °C	biogas production increase from 3657 to 4843 mL/g d.m. (32%)	[49]
8	WAS	170 °C, 60 min	batch fermentation, 24 d, 35 °C	biogas production increase (45%)	[50]
9	WAS	170 °C, 60 min	continuous fermentation HRT 20 d, 35 °C	CH_4 production increase from 88 to 142 mL/g COD (61%)	
10	WAS	160 °C, 30 min	Wastewater treatment plant, HRT 15 d, 35 °C	reducing of suspension concentration (45%)	[71]
11	WAS	175 °C, 40 min	continuous fermentation HRT 2.9 d, 37 °C	reducing of suspension concentration (65%)	[45]
12	WAS	170 °C, 30 min	continuous fermentation HRT 20 d, 35 °C	CH_4 production increase from 145 to 256 mL/g d.m. (51%)	[72]
13	WAS	170 °C, 30 min	batch fermentation, 24 d, 35 °C	CH_4 production increase from 221 to 333 mL/g COD (76%)	
14	WAS	170 °C, 30 min, 7 bar	batch fermentation	CH_4 production increase (50%)	[73]
15	Mixed PS + WAS	120 °C, 60 min	continuous fermentation HRT 20 d, 36 °C	CH_4 production increase from 350 to 420 mL/g d.m. (20%)	[16]
16	Mixed PS + WAS	170 °C, 1 min, 0.8 MPa	batch fermentation, 20 d	biogas production increase (49%)	[43]
17	Mixed PS + WAS	140 °C, 1 min, 0.6 MPa	wastewater treatment plant, 53–55 °C	biogas production increase from 507 to 599 mL/g d.m. (18%)	[74]

An analysis of the energy balance shows that, compared to conventional methane fermentation, systems with introduced thermal disintegration of sewage sludge are characterized by the possibility of obtaining a positive energy balance at a smaller volume of fermentation chambers. Li and Noike [35] obtained a reduction in the required fermentation time by 5 days for excessive sludge disintegrated at 170 °C for 60 min. Under these conditions, the organic compounds measured by COD were removed in 60%, with biogas production equal to 223 L/kg of COD, which was twice the value compared to raw sludge. According to these authors, the positive impact of the pre-treatment on the anaerobic biodegradation of the activated sludge was the result of a hydrolysis of the solid fraction and the production of volatile fatty acids, which were transformed into biogas.

After processing the substrates at temperatures above 175 °C, their biodegradation is difficult. Pinnekamp [65] stated that at treatment temperatures higher than 180 °C, the opposite effect is obtained. The production of gas obtained from sewage sludge disintegrated at temperatures above 180 °C was much lower than in the control samples. Fisher and Swanwick [75] showed that heating sewage sludge before fermentation to a temperature above 180 °C provides the greatest loss of dry matter, but there are refractory organic compounds in the process liquid (non-degradable COD fractions). Bougrier et al. [76] stated that the formation of refractive compounds is the result of Maillard's reaction. Stuckey and McCarty [47] observed that increasing the processing temperature to 225 °C increased the liquefaction of excessive sludge by only 51%. They found that high hydrolysis temperatures reduce the amount of dissolved organic fractions, as a result of the formation of large-molecule soluble compounds in the reaction of simple sugars with amino acids, inhibiting the methane fermentation process. The adaptation of the fermenting biomass to the substrate in the form of excessive sludge disintegrated at 225 and 250 °C was achieved only after 8 days, which suggests that refractive compounds were the main inhibitor of biodegradation and a component of sludge after treatment carried out at these temperatures.

In addition to the measurable effects associated with higher biogas production after thermal disintegration, an important aspect is the impact of temperature on microorganisms. As shown in many studies [77], only a few minutes of heat treatment causes the cells of microorganisms to die. Heating biomass for 10 min at 90 °C resulted in destroying almost 100% of active sludge bacteria cells, while a one-hour treatment at 60 °C gave a 98% effect (2% constitute the remaining active thermophilic microorganisms).

In practice, high-temperature methods are highly appreciated, including the process of thermal hydrolysis in the CAMBI technology [17,29,78]. In addition to a dozen or so sewage sludge thermohydrolysis installations, the CAMBI technology has been used to carry out an installation of pre-treatment of municipal waste (Lillehammer, Norway, 1999, 14 thousand tonnes of waste). The CAMBI technology involves the destruction of cells of excessive and preliminary sludge thickened to approx. 12% d.m., consisting in heating the sludge to 165–170 °C at a pressure of 5–6 bar, and then quickly lowering the pressure. The duration of the process is 30 min. Reducing mass in the process of fermenting the thermally disintegrated sludge is at more than 65%, while an increase in biogas production compared to classic systems reaches approx. 30%. A better final sludge dewatering is also achieved.

4. Indicators for Assessing the Degree of Disintegration

The effectiveness of disintegration processes of organic substrates is considered, first of all, in terms of assessing the change in particle size, liquefaction of the solid fraction, increase in biodegradability, formation of refractive compounds, and reduction of organic mass. So far, one single, commonly used method of assessing the effects of disintegration processes has not been established, hence comparing the results of research described by the authors with various indicators is difficult and not always possible. Moreover, the variety of test conditions used (e.g., treatment temperature, disintegration time, variable hydration of substrates) increases the problem of objective assessment of the efficiency of the process.

The indicators for assessing the degree of substrate disintegration can be divided into three groups [72,79,80]:

Group I—direct indicators: the changes in the structure and physical-chemical properties of substrates and hydrolysates is identified by determining the difference in the values of parameters in substrates before and after disintegration. Assessment indicators depend on the conditions of the disintegration process and include:

- Fragmentation of solid phase particles (change of particle size and particle size distribution, change of rheological properties, change of COD fraction) which is primarily the result of breaking the cell membranes of microorganisms and releasing organic substances into the liquid phase;
- Decrease in hydrolysis time of biopolymers by extracellular enzymes, which are formed and excreted by fermentative microorganisms.

Group II—secondary effects occurring in disintegrated substrates: Intensification of anaerobic or aerobic stabilization. For the fermentation process, important indicators consist in the biogas and methane potential, the loss of dry matter and organic matter in the input, the increase in susceptibility to dewatering processes and the decrease in final hydration. For denitrification and dephosphatation processes, the secondary effect consists in an increase in the concentration of VFA as a source of organic carbon compounds.

Group III—energy indicators: Recovery of energy spent on disintegration as a result of biogas combustion or cogeneration transformation into electricity and heat.

Based on the literature data, it can be assumed that the effectiveness of disintegration processes is commonly assessed by comparing the share of COD fraction dissolved before and after processing, in relation to total, molecular, or dissolved COD (Table 3).

Table 3. Indicators of assessment of the degree of disintegration.

Assessment Indicator	Definition	Source
Solubilization of COD, ηDCOD	$\eta DCOD = [DCOD^D - DCOD^0]/PCOD$	[36,45,51,72,76,81–83]
	$\eta DCOD = [DCOD^D - DCOD^0]/COD$	[48,84–86]
	$\eta DCOD = DCOD^D/COD$	[50,87–89]
	$\eta DCOD = [DCOD^D - DCOD^0]/DCOD^D$	[90]
	$\eta DCOD = [DCOD^D - DCOD^0]/[d.m.]$	[91]
Degree of disintegration, η_D	$\eta_D = [DCOD^D - DCOD^0]/[CODmax - DCOD^0]$	[51,92,93]
	$\eta_D = [DCOD^D - DCOD^0]/CODmax$	[94]

$DCOD^0$—COD of dissolved fraction before disintegration, mg O_2/L; $DCOD^D$—COD of dissolved fraction after disintegration, mg O_2/L; PCOD—COD of particulate fraction before disintegration, mg O_2/L; COD—total COD, mg O_2/L; COD_{max}—max. value of dissolved COD after disintegration by NaOH, mg O_2/L; d.m.—dry matter, mg/L; η—effectiveness

Other indicators of direct assessment of disintegration effectiveness usually consist in comparing changes in the amount of dissolved organic substances before and after the process, such as carbohydrates, volatile fatty acids, or dissolved organic carbon concentrations [36,83]. Many authors also measure the efficiency of liquefaction of substrates by increasing the concentration of dissolved protein; however, this method is recommended mainly for assessing the disintegration of excessive sludge [95]. Regardless of the used disintegration method, the basic treatment effect is to liquefy the solid fraction of the substrate. It is assumed that the fragmentation of sewage sludge increases the availability of organic substances for microorganisms, i.e., potential biodegradability. However, literature reports do not unequivocally confirm this fact. According to studies by Wang et al. [96], the increase in the biodegradability of substrates is related to their liquefaction and reduction in particle size. Other researchers show no correlation between these parameters [63,97]. Depending on the characteristics of the raw substrates and the treatment method, the increase in the biodegradability of hydrolysates may be limited by the formation of refractive/toxic compounds [47,98,99] and by the decomposition (loss) of organic matter [80]. Inhibitors of biodegradation processes are formed, among others, in the processes of disintegration of biomass containing lignocellulose, which results in the formation of hydroxymethylfurfural (HMF) and soluble phenol compounds [80], or Amadori products or melanoids (Maillard reaction products) [5,39,92].

The assessment of the biodegradability of hydrolysates by determining the biogas and methane potentials is the most practical of all indicators for assessing the effectiveness of disintegration methods. Determining the amount of biogas that can be obtained directly indicates an improvement in the energy effect in relation to the volume or dry weight of disintegrated substrates.

Simple methods for determining the biogas potential, based on using equations by Buswell and Mueller [100], Boyle [101], or Amon et al. [102], were developed independently in different years and are based on basic organic substrates. The efficiency of the methane fermentation process is based on assessing the methane and carbon dioxide production.

Buswell and Mueller [100] assumed that if the elemental composition of organic matter is known, the production of methane and carbon dioxide can be determined with an accuracy of 5% based on the reaction:

$$C_aH_bO_c + \left(a - \frac{b}{4} - \frac{c}{2}\right)H_2O \rightarrow \left(\frac{a}{2} + \frac{b}{8} - \frac{c}{4}\right)CH_4 + \left(\frac{a}{2} - \frac{b}{8} + \frac{c}{4}\right)CO_2 \tag{2}$$

Boyle [101] supplemented the Buswell and Mueller model by introducing nitrogen and sulphur to the equation:

$$\begin{aligned} C_aH_bO_cN_dS_e + \left(a - \tfrac{b}{4} - \tfrac{c}{2} + \tfrac{3\cdot d}{4} + \tfrac{e}{2}\right)H_2O \rightarrow \\ \left(\tfrac{a}{2} + \tfrac{b}{8} - \tfrac{c}{4} - \tfrac{3\cdot d}{8} - \tfrac{e}{4}\right)CH_4 + \left(\tfrac{a}{2} - \tfrac{b}{8} + \tfrac{c}{4} + \tfrac{3\cdot d}{8} + \tfrac{e}{4}\right)CO_2 + dNH_3 + eH_2S \end{aligned} \tag{3}$$

Amon et al. [102] extended the basic division of substrates included in the equations to their bioavailability for microorganisms, especially in the case of anaerobic decomposition of agricultural waste and from animal breeding, e.g., corn, cereals, and grasses.

The effectiveness of the disintegration process can also be assessed using an indirect (energy) indicator taking into account the impact of the treatment on the methane potential of substrates [48,103]. Indirect indicators of energy and technological efficiency can be evaluated under the conditions of technical wastewater plant operation. The Cambi (forty-one installations by 2021) and the Exelys (three installations by 2021) technologies have the most experience in commercially available full-scale sludge hydrothermal pretreatment processes [104]. However, the published data relates to direct indicators (mainly an increase of biogas production and a reduction of sewage sludge) and not energy indicators and balance.

5. Conclusions

Very rich scientific studies indicate a great need to develop the modeling of the thermal disintegration of sewage sludge and processes occurring during methane fermentation of liquefied substrates, taking into account the possibility of using these models in the practice of designing and operating wastewater treatment plants. Thermal hydrolysis effectively changed the physico-chemical properties of sewage sludge, improved dewatering and biodegradability, increased biogas production and energy recovery, and decreased carbon footprint.

It is important to pay attention to the process parameters, the use of which does not result in the formation of inert substances that reduce the biogas potential and biodegradation efficiency.

Author Contributions: Conceptualization S.M.; methodology; validation, S.M. and E.P.-K.; resources, S.M. and E.P.-K.; writing—original draft preparation S.M. and E.P.-K.; writing—review and editing, S.M.; visualization, S.M.; supervision, S.M.; project administration, S.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: No new data were created or analyzed in this study. Data sharing is not applicable to this article.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Stouthamer, A.H. A theoretical study on the amount of ATP required for synthesis of microbial cell material. *Antonie Van Leeuwenhoek* **1973**, *39*, 545–565. [CrossRef] [PubMed]
2. Nielsen, P.H.; Thomsen, T.R.; Nielsen, J.L. Bacterial composition of activated sludge—Importance for floc and sludge properties. *Water Sci. Technol.* **2004**, *49*, 71–78. [CrossRef]
3. Henze, M.; Gujer, W.; Mino, T.; van Loosedrecht, M. *Activated Sludge Models ASM1, ASM2, ASM2d and ASM3*; IWA Publishing: London, UK, 2006; ISBN 9781780402369. [CrossRef]
4. Batstone, D.; Keller, J.; Angelidaki, I.; Kalyuzhnyi, S.; Pavlostathis, S.; Rozzi, A.; Sanders, W.; Siegrist, H.; Vavilin, V. Anaerobic digestion model No 1 (ADM1). *Water Sci. Technol.* **2002**, *45*, 65–73. [CrossRef] [PubMed]
5. Yujie, F.; Hornung, U.; Dahmen, N. Hydrothermal liquefaction of sewage sludge for biofuel application: A review on fundamentals, current challenges and strategie. *Biomass Bioenergy* **2022**, *165*, 106570. [CrossRef]
6. Myszograj, S. Biogas and Methane Potential of Pre-Thermally Disintegrated Bio-Waste. *Energies* **2019**, *12*, 3880. [CrossRef]
7. Gong, L.; Yang, X.; Wang, Z.; Zhou, J.; You, X. Impact of hydrothermal pre-treatment on the anaerobic digestion of different solid–liquid ratio sludges and kinetic analysis. *RSC Adv.* **2019**, *9*, 19104–19113. [CrossRef] [PubMed]
8. Yan, W.; Xu, H.; Lu, D.; Zhou, Y. Effects of sludge thermal hydrolysis pretreatment on anaerobic digestion and downstream processes: Mechanism, challenges and solutions. *Bioresour. Technol.* **2022**, *344*, 126248. [CrossRef]
9. Myszograj, S. The influence of thermo-chemical treatment of primary sludge on methane fermentation process. In *Environmental Protection into the Future*; Bień, J.J., Nowak, W., Eds.; Wydawnictwo Politechniki Częstochowskiej: Częstochowa, Poland, 2007; pp. 228–237.
10. Kor-Bicakci, G.; Eskicioglu, C. Recent developments on thermal municipal sludge pretreatment technologies for enhanced anaerobic digestion. *Renew. Sustain. Energy Rev.* **2019**, *110*, 423–443. [CrossRef]
11. Kuglarz, M.; Karakashev, D.; Angelidaki, I. Microwave and thermal pretreatment as methods for increasing the biogas potential of secondary sludge from municipal wastewater treatment plants. *Bioresour. Technol.* **2013**, *134*, 290–297. [CrossRef]
12. Pilli, S.; Pandey, A.K.; Katiyar, A.; Pandey, K.; Tyagi, R.D. Pre-treatment technologies to enhance anaerobic digestion. In *Sustainable Sewage Sludge Management and Resource Efficiency*; Taşeli, B.K., Ed.; IntechOpen: London, UK, 2020.
13. Smith, G.; Göransson, J. Generation of an effective internal carbon source for denitrification through thermal hydrolysis of pre-precipitated sludge. *Water Sci. Technol.* **1992**, *25*, 211–218. [CrossRef]
14. Barlindhaug, J.; Odegaard, H. Thermal hydrolysis for the production of carbon source for denitrification. *Water Sci. Technol.* **1996**, *34*, 371–378. [CrossRef]
15. Djandja, O.S.; Yin, L.-X.; Wang, Z.-C.; Duan, P.-G. From wastewater treatment to resources recovery through hydrothermal treatments of municipal sewage sludge: A critical review. *Process Saf. Environ. Protect.* **2021**, *151*, 101–127. [CrossRef]
16. Barjenbruch, M.; Hoffmann, H.; Tränker, J. Minimizing of foaming in digesters by pre-treatment of the surplus sludge. *Water Sci. Technol.* **1999**, *42*, 235–242. [CrossRef]
17. Kepp, U.; Solheim, O.E. Meeting increased demands on sludge quality—Experience with full scale plant for thermal disintegration. In Proceedings of the 9th World Congress, Anaerobic Conversion for Sustainability, Antwerpen, Belgium, 2–6 September 2001.
18. Tanaka, S.; Kobayashi, T.; Kamiyama, K.; Bildan, M.L. Effects of thermochemical pre-treatment on the anaerobic digestion of waste activated sludge. *Water Sci. Technol.* **1997**, *8*, 209–215. [CrossRef]
19. Syed-Hassan, S.S.A.; Wang, Y.; Hu, S.; Su, S.; Xiang, J. Thermochemical processing of sewage sludge to energy and fuel: Fundamentals, challenges and considerations. *Renew. Sustain. Energy Rev.* **2017**, *80*, 888–913. [CrossRef]
20. Kennedy, K.J.; Thibault, G.; Droste, R.L. Microwave enhanced digestion of aerobic SBR sludge. *Water SA* **2007**, *33*, 0378–4738. [CrossRef]
21. Ruiz-Hernando, M.; Vinardell, S.; Labanda, J.; Llorens, J. Effect of ultrasonication on waste activated sludge rheological properties and process economics. *Water Res.* **2022**, *208*, 117855. [CrossRef]
22. Thomas, L.; Jungschaffer, G.; Sprössler, B. Improved sludge dewatering by enzymatic treatment. *Water Sci. Technol.* **1993**, *28*, 189–192. [CrossRef]
23. Guellil, A.M.; Boualam, H.; Quiquampoix, P.; Ginestet, P.; Audic, J.; Block, C. Hydrolysis of wastewater colloidal enzymes extracted from activated sludge flocs. *Water Sci. Technol.* **2001**, *43*, 33–40. [CrossRef]
24. Gao, W. Freezing as a combined wastewater sludge pretreatment and conditioning method. *Desalination* **2011**, *268*, 170–173. [CrossRef]
25. Liu, S.; Zhua, N.; Li, L. The one-stage autothermal thermophilic aerobic digestion for sewage sludge treatment. *Chem. Eng. J.* **2011**, *174*, 564–570. [CrossRef]
26. Bauman-Kaszubska, H.; Sikorski, M. The impact of selected sewage treatment methods on the change in parameters of sewage sludge originating from municipal sewage treatment plants. *J. Ecol. Eng.* **2018**, *19*, 199–207. [CrossRef]
27. Ferrer, I.; Ponsab, S.; Vazquezc, F.; Font, X. Increasing biogas production by thermal sludge pretreatment prior to thermophilic anaerobic digestion. *Biochem. Eng. J.* **2008**, *42*, 186–192. [CrossRef]
28. Haug, R.T.; Stuckey, D.C.; Gossett, J.M.; Mac Carty, P.L. Effect of thermal pretreatment on digestibility and dewaterability of organic sludges. *J. Water Pollut. Control.* **1978**, *50*, 73–85.
29. Kepp, U.; Machenbach, I.; Weisz, N.; Solheim, O.E. Enhanced stabilization of sewage sludge through thermal hydrolysis, 3 years of experience with full-scale plant. *Water Sci. Technol.* **2000**, *42*, 89–96. [CrossRef]

30. Neyens, E.; Baeyens, J. A review of thermal sludge pre-treatment processes to improve dewaterability. *J. Hazard. Mater.* **2003**, *98*, 51–67. [CrossRef]
31. Gahlot, P.; Balasundaram, G.; Tyagi, V.K.; Atabani, A.E.; Suthar, S.; Kazmi, A.A.; Štěpanec, L.; Juchelková, D.; Kumar, A. Principles and potential of thermal hydrolysis of sewage sludge to enhance anaerobic digestion. *Environ Res.* **2022**, *214*, 113856. [CrossRef]
32. Sawayama, S.; Inoue, S.; Ogi, T.; Yokoyama, S. Thermochemical anaerobic treatment of dewatered sewage sludge. *J. Ferment. Bioeng.* **1995**, *79*, 300–302. [CrossRef]
33. Dote, Y.; Yokoyama, S.; Minowa, T.; Masuta, T.; Sato, K.; ShinjiItoh, S.; Suzuki, A. Thermochemical liquidization of dewatered sewage sludge. *Biomass Bioenergy* **1993**, *4*, 243–248. [CrossRef]
34. Myszograj, S.; Stadnik, A.; Płuciennik-Koropczuk, E. The influence of trace elements on anaerobic digestion process. *Civ. Environ. Eng. Rep.* **2018**, *28*, 105–115. [CrossRef]
35. Li, Y.Y.; Noike, T. Upgrading of anaerobic digestion of waste activated sludge by thermal pre-treatment. *J. Water Sci. Technol.* **1992**, *26*, 857–866. [CrossRef]
36. Bougrier, C.; Delgenes, J.P.; Carrere, H. Effects of thermal treatments on five different waste activated sludge samples solubilisation, physical properties and anaerobic digestion. *Chem. Eng. J.* **2008**, *139*, 236–244. [CrossRef]
37. Wilson, C.A.; Novak, J.T. Hydrolysis of macromolecular components of primary and secondary wastewater sludge by thermal hydrolytic pretreatment. *Water Res.* **2009**, *43*, 4489–4498. [CrossRef] [PubMed]
38. Paul, E.; Camacho, P.; Sperandio, M.; Ginestet, P. Technical and economical evaluation of a thermal, and two oxidative techniques for the reduction of excess sludge production. *Process Saf. Environ. Prot.* **2006**, *84*, 247–252. [CrossRef]
39. Matsuura, Y.; Takehira, M.; Joti, Y.; Ogasahara, K.; Tanaka, T.; Ono, N.; Kunishima, N.; Yutani, K. Thermodynamics of protein denaturation at temperatures over 100 °C: CutA1 mutant proteins substituted with hydrophobic and charged residues. *Sci. Rep.* **2015**, *5*, 15545. [CrossRef] [PubMed]
40. Ramirez, I.; Motteta, A.; Carrerea, H.; Delerisb, S.; Vedrenneb, F.; Steyera, J.P. Modified ADM1 disintegration/hydrolysis structures for modeling batch thermophilic anaerobic digestion of thermally pretreated waste activated sludge. *Water Res.* **2009**, *43*, 3479–3492. [CrossRef]
41. Peterson, A.A.; Lachance, R.P.; Tester, J.W. Kinetic evidence of the Maillard reaction in hydrothermal biomass processing: Glucose-glycine interactions in high-temperature, high-pressure water. *Ind. Eng. Chem. Res.* **2010**, *49*, 2107–2117. [CrossRef]
42. Myszograj, S.; Płuciennik-Koropczuk, E. COD and nitrogen compounds balance in mechanical-biological wastewater treatment plant with sludge treatment. *Desalination Water Treat.* **2020**, *186*, 443–449. [CrossRef]
43. Dohanyos, M.; Zabranska, J.; Kutil, J.; Jenicek, P. Improvement of anaerobic digestion of sludge. *Water Sci. Technol.* **2004**, *49*, 89–96. [CrossRef]
44. Elbing, G.; Dünnebeil, A. Thermal disintegration with subsequent digestion lab-scale investigation. *Korresp. Abwasser* **1999**, *46*, 538–547.
45. Graja, S.; Chauzy, J.; Fernandes, P.; Patria, L.; Cretenot, D. Reduction of sludge production from WWTP using thermal pretreatment and enhanced anaerobic methanisation. *Water Sci. Technol.* **2005**, *52*, 267–273. [CrossRef] [PubMed]
46. Xue, T.; Huang, X. Releasing characteristics of phosphorus and other substances during thermal treatment of excess sludge. *J. Environ. Sci.* **2007**, *19*, 1153–1158. [CrossRef] [PubMed]
47. Stuckey, D.C.; McCarty, P.L. The effect of thermal pre-treatment on the anaerobic biodegradability and toxicity of waste activated sludge. *Water Res.* **1984**, *18*, 1343–1353. [CrossRef]
48. Myszograj, S. Effects and mathematical modelling of thermal pretreatment of waste activated sludge. *Pol. J. Environ. Stud* **2010**, *2*, 166–170.
49. Kim, J. Effects of various pretreatments for enhanced anaerobic digestion with waste activated sludge. *J. Biosci. Bioeng.* **2003**, *95*, 271–275. [CrossRef]
50. Valo, A.; Carrère, H.; Delgenès, J. Thermal, chemical and thermo-chemical pre-treatment of waste activated sludge for anaerobic digestion. *J. Chem. Technol. Biotechnol.* **2004**, *79*, 1197–1203. [CrossRef]
51. Bougrier, C.; Carrere, H.; Battimelli, A.; Delgenes, J.P. Effects of various pretreatments on WAS in order to improve matter solubilization and anaerobic digestion. In Proceedings of the 10th World Congress on Anaerobic Digestion (AD10-2004), Montreal, QC, Canada, 29 August–2 September 2004.
52. Aboulfoth, A.M.; El Gohary, E.H.; El Monayeri, O.D. Effect of thermal pretreatment on the solubilization of organic matters in a mixture of primary and waste activated sludge. *J. Urban Environ. Eng.* **2015**, *9*, 82–88. [CrossRef]
53. Carballa, M.; Omil, F.; Lema, M. Improvement of anaerobic digestion operation and digested sludge character-istics using a chemical and thermal pre-treatment. In Proceedings of the 10th World Congress on Anaerobic Digestion (AD10-2004), Montreal, Canada, 29 August–2 September 2004.
54. Yuan, Q.; Sparling, R.; Oleszkiewicz, J.A. Technical Note: VFA generation from waste activated sludge: Effect of temperature and mixing. *Chemosphere* **2011**, *82*, 603–607. [CrossRef]
55. Jolis, D. High-solids anaerobic digestion of municipal sludge pretreated by thermal hydrolysis. *Water Environ. Res.* **2008**, *80*, 654–662. [CrossRef]
56. Dohanyos, M.; Zabranska, J.; Jenicek, P. Enhancement of sludge anaerobic digestion by using of a special thickening centrifuge. *Water Sci. Technol.* **1997**, *36*, 145–153. [CrossRef]

57. Hiraoka, M.; Takeda, N.; Sakai, S.; Yasuda, A. Highly efficient anaerobic digestion with thermal pre-treatment. *Water Sci. Technol.* **1985**, *17*, 529–539. [CrossRef]
58. Gavala, H.N.; Angelidaki, I.; Ahring, B.K. Kinetics and modelling of anaerobic digestion process. *Adv. Biochem. Eng. Biotechnol.* **2003**, *81*, 57–93. [CrossRef] [PubMed]
59. Skiadas, I.V.; Gavala, H.N.; Lu, J.; Ahring, B.K. Thermal pre-treatment of primary and secondary sludge at 70 degrees C prior to anaerobic digestion. *Water Sci. Technol.* **2005**, *52*, 161–166. [CrossRef] [PubMed]
60. Lü, F.; Chen, M.; He, P.J. Effects of ammonia on acidogenesis of protein-rich organic wastes. *Environ. Eng. Sci.* **2008**, *25*, 114–122. [CrossRef]
61. Ge, H.Q.; Jensen, P.D.; Batstone, D.J. Pretreatment mechanisms during thermophilic-mesophilic temperature phased anaerobic digestion of primary sludge. *Water Res.* **2010**, *44*, 123–130. [CrossRef]
62. Hasegawa, S.; Shiota, N.; Katsura, K.; Akashi, A. Solubilization of organic sludge by thermophilic aerobic bacteria as a pretreatment for anaerobic digestion. *Water Sci. Technol.* **2000**, *41*, 163–169. [CrossRef]
63. Climent, M.; Ferrer, I.; Baeza, M.D.; Artola, A.; Vazquez, F.; Font, X. Effects of thermal and mechanical pretreatments of secondary sludge on biogas production under thermophilic conditions. *Chem. Eng. J.* **2007**, *133*, 335–342. [CrossRef]
64. Haug, R.T.; LeBrun, T.J.; Tortorici, L.D. Thermal pre-treatment of sludge-a field demonstration. *J. Water Pollut. Control Fed.* **1983**, *55*, 23–34.
65. Pinnekamp, J. Effects of thermal pre-treatment of sewage sludge on anaerobic digestion. *Water Sci. Technol.* **1989**, *21*, 97–108. [CrossRef]
66. Prechtl, S.; Scheneider, R.; Bischof, F.; Faulstich, M. Digestion of waste water after pre-treatment by the process of thermal pressure hydrolysis. In Proceedings of the 9th World Congress, Anaerobic Conversion for Sustainability, Antwerpen, Belgium, 2–6 September 2001.
67. Guibelin, E. Sustainability of thermal oxidation processes: Strengths for the new millennium. *Water Sci. Technol.* **2002**, *46*, 259–267. [CrossRef]
68. Zhen, G.; Lua, X.; Katoc, H.; Zhaod, Y.; Lia, Y.Y. Overview of pretreatment strategies for enhancing sewage sludge disintegration and subsequent anaerobic digestion: Current advances, fullscale application and future perspectives. *Renew. Sustain. Energy Rev.* **2017**, *69*, 559–577. [CrossRef]
69. Brooks, R. Heat treatment of sewage sludge. *Water Pollut. Control Fed.* **1970**, *69*, 221–231.
70. Fjordside, C. An operating tale from Naeested Sewage Treatment plant. In Proceedings of the Municipal Wastewater Treatment Nordic Conference, Copenhagen, Denmark, 17–19 January 2001; pp. 17–19.
71. Chauzy, J.; Graja, S.; Gerardin, F.; Cretenot, D.; Patria, L.; Fernandes, P. Minimization of excess sludge production in a WWTP by coupling thermal hydrolysis and rapid anaerobic digestion. *Water Sci. Technol.* **2005**, *52*, 255–263. [CrossRef] [PubMed]
72. Bougrier, C.; Albasi, C.; Delgenes, J.P.; Carrere, H. Effect of ultrasonic, thermal and ozone pre-treatments on waste activated sludge solubilisation and anaerobic biodegradability. *Chem. Eng. Process.* **2006**, *45*, 711–718. [CrossRef]
73. Fernández-Polanco, F.; Velazquez, R.; Perez-Elvira, S.I.; Casas, C.; Del Barrio, D.; Cantero, F.J.; Fdz-Polanco, M.; Rodriguez, P.; Panizo, L.; Serrat, J.; et al. Continuous thermal hydrolysis and energy integration in sludge anaerobic digestion plants. *Water Sci. Technol.* **2008**, *57*, 1221–1226. [CrossRef]
74. Zabranska, J.; Dohanyos, M.; Jenicek, P.; Kutil, J.; Cejka, J. Mechanical and rapid thermal disintegration methods of enhancement of biogas production-full-scale applications. In Proceedings of the IWA Specialized Conference on Sustainable Sludge Management: State-of-The-Art, Challenges and Perspectives, Moscow, Russia, 29–31 May 2006; pp. 235–241.
75. Fisher, R.A.; Swanwick, S.J. Hight temperature treatment of sewage sludges. *Water Pollut.Control* **1971**, *71*, 255–370.
76. Bougrier, C.; Delgenes, J.P.; Carrère, H. Impacts of thermal pre-treatments on the semi-continuous anaerobic digestion of waste activated sludge. *Biochem. Eng. J.* **2007**, *34*, 20–27. [CrossRef]
77. Lang, N.L.; Smith, S.R. Time and temperature inactivation kinetics of enteric bacteria relevant to sewage sludge treatment processes for agricultural use. *Water Res.* **2008**, *42*, 2229–2241. [CrossRef]
78. Weisz, N.; Kepp, U.; Norli, M.; Panter, K.; Solheim, O.E. Sludge disintegration with thermal hydrolysis-cases from Norway, Denmark and United Kingdom. In Proceedings of the 1st IWA World Congress, Paris, France, 3–7 July 2000; pp. 288–295.
79. Tiehm, A.; Nickel, K.; Zellhorn, M.; Neis, U. Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization. *Water Res.* **2001**, *35*, 2003–2009. [CrossRef] [PubMed]
80. Hendriks, A.T.; Zeeman, G. Pretreatments to enhance the digestibility of lignocellulosic biomass. *Bioresour. Technol.* **2009**, *100*, 8–10. [CrossRef]
81. Carlsson, M.; Lagerkvist, A.; Morgan-Sagastume, F. The effects of substarte pre-treatment on anaerobic digestion systems: A review. *Waste Manag.* **2012**, *32*, 1634–1650. [CrossRef] [PubMed]
82. Mottet, A.; Steyera, J.P.; Délérisb, S.; Vedrenneb, F.; Chauzyc, J.; Carrèrea, H. Kinetics of thermophilic batch anaerobic digestion of thermal hydrolysed waste activated sludge. *Biochem. Eng. J.* **2009**, *46*, 169–175. [CrossRef]
83. Salsabil, M.R.; Laurent, J.; Casellas, M.; Dagot, C. Techno-economic evaluation of thermal treatment, ozonation and sonication for the reduction of wastewater biomass volume before aerobic or anaerobic digestion. *J. Hazard. Mater.* **2010**, *174*, 323–333. [CrossRef] [PubMed]
84. Apples, L.; Baeyens, J.; Degrève, J.; Dewil, R. Principles and potential of the anaerobic digestion of waste-activated sludge. *Prog. Energy Combust. Sci.* **2008**, *34*, 755–781. [CrossRef]

85. Marin, J.; Kennedy, K.J.; Eskicioglu, C. Effect of microwave irradiation on anaerobic degradability of model kitchen waste. *Waste Manag.* **2010**, *30*, 1772–1779. [CrossRef]
86. Myszograj, S. Thermal and chemical disintegration to improve sewage sludge solubilisation. *Acta Nat. Sci.* **2014**, *1*, 1–10.
87. Eskicioglu, C.; Kennedy, K.J.; Droste, R.L. Characterization of soluble organic matter of WAS before and after thermal pretreatment. *Water Res.* **2006**, *40*, 3725–3736. [CrossRef]
88. Perez-Elvira, S.I.; Fdz-Polanco, M.; Plaza, F.I.; Garralon, G. Ultrasound pretreatment for anaerobic digestion improvement. *Water Sci. Technol.* **2009**, *60*, 1525–1532. [CrossRef]
89. Wett, B.; Phothilangka, P.; Eladawy, A. Systematic comparison of mechanical and thermal sludge disintegration technologies. *Waste Manag.* **2010**, *30*, 1057–1062. [CrossRef]
90. Ma, J.; Duong, T.H.; Smits, M.; Verstraete, W.; Carballa, M. Enhanced biomethanation of kitchen waste by different pre-treatments. *Bioresour. Technol.* **2011**, *102*, 592–599. [CrossRef]
91. Apul, O.G.; Sanin, F.D. Ultrasonic pretreatment and subsequent anaerobic digestion under different operation-al conditions. *Bioresour. Technol.* **2010**, *101*, 8984–8992. [CrossRef] [PubMed]
92. Müller, J.A. Disintegration as a key step in sewage sludge treatment. *Water Sci. Technol.* **2000**, *41*, 123–130. [CrossRef]
93. Naddeo, V.; Belgiorno, V.; Landi, M.; Zarra, T.; Napoli, R.M.A. Effect of sonolysis on WAS solubilization and anaerobic biodegradability. *Desalination* **2009**, *249*, 762–767. [CrossRef]
94. Braguglia, C.M.; Gianico, A.; Mininni, G. Comparison between ozone and ultrasound disintegration on sludge anaerobic digestion. *J. Environ. Manag.* **2010**, *19*, 596–600. [CrossRef] [PubMed]
95. Schmitz, U.; Berger, R.; Orth, H. Protein analysis as a simple method for the quantitative assessment of sewage sludge disintegration. *Water Res.* **2000**, *34*, 3682–3685. [CrossRef]
96. Wang, Q.; Kuninobo, M.; Kakimoto, K.; Ogawa, H.I.; Kato, Y. Upgrading of anaerobic digestion of WAS by ultrasonic pretreatment. *Bioresour. Technol.* **1999**, *68*, 309–313. [CrossRef]
97. Strong, P.J.; Gapes, D.J. Thermal and thermo-chemical pre-treatment of four waste residues and the effect on acetic acid production and methane synthesis. *Waste Manag.* **2012**, *32*, 1669–1677. [CrossRef]
98. Lissens, G.; Thomsen, A.B.; De Baere, L.; Versraete, W.; Ahring, B. Thermal wet oxidation improves anaero-bic digestion of raw and digested biowaste. *Environ. Sci. Technol.* **2004**, *38*, 3418–3424. [CrossRef]
99. Carrere, H.; Rafrafi, Y.; Battimelli, A.; Torrijos, M.; Delgenes, J.P.; Motte Cassini, C. Improving methane pro-duction during the codigestion of waste-activated sludge and fatty wastewater: Impact of thermo-alkaline pretreat-ment on batch and semi-continuous processes. *Chem. Eng. J.* **2012**, *10*, 404–409. [CrossRef]
100. Buswell, A.M.; Mueller, H.F. Mechanism of methane fermentation. *Ind. Eng. Chem.* **1952**, *44*, 550–552. [CrossRef]
101. Boyle, W.C. Energy recovery from sanitary landfills a review. In *Microbial Energy Conversion*; Schlegel, H.G., Barnea, J., Eds.; Pergamon Press: Oxford, UK, 1976; pp. 119–138.
102. Amon, B.; Kryvoruchko, V.; Amon, T.; Zechmeister-Boltenstern, S. Methane, nitrous oxide and ammonia emissions during storage and after application of dairy cattle slurry and influence of slurry treatment. *Agric. Ecosyst. Environ.* **2006**, *112*, 153–162. [CrossRef]
103. Yoshida, H.; Tokumoto, H.; Ishii, K.; Ishii, R. Efficient, high-speed methane fermentation for sewage sludge using subcritical water hydrolysis as pretreatment. *Bioresour. Technol.* **2009**, *100*, 2933–2939. [CrossRef] [PubMed]
104. Kakar, F.L.; Tadesse, F.; Elbeshbishy, E. Comprehensive Review of Hydrothermal Pretreatment Parameters Affecting Fermentation and Anaerobic Digestion of Municipal Sludge. *Processes* **2022**, *10*, 2518. [CrossRef]