Low-Temperature Pretreatment of Biomass for Enhancing Biogas Production: A Review

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Abstract: Low-temperature pretreatment (LTPT, Temp. < 100 °C or 140 °C) has the advantages of low input, simplicity, and energy saving, which makes engineering easy to use for improving biogas production. However, compared with high-temperature pretreatment (>150 °C) that can destroy recalcitrant polymerized matter in biomass, the action mechanism of heat treatment of biomass is unclear. Improving LTPT on biogas yield is often influenced by feedstock type, treatment temperature, exposure time, and fermentation conditions. Such as, even when belonging to the same algal biomass, the response to LTPT varies between species. Therefore, forming a unified method for LTPT to be applied in practice is difficult. This review focuses on the LTPT used in different biomass materials to improve anaerobic digestion performance, including food waste, sludge, animal manure, algae, straw, etc. It also discusses the challenge and cost issues faced during LTPT application according to the energy balance and proposes some proposals for economically promoting the implementation of LTPT.

Keywords: organic waste; anaerobic digestion; thermal pretreatment; biogas; bioavailability

1. Introduction

People’s production and living produce organic waste continually, which has hot spots such as large production, complex composition, and easy-to-breed germs, posing a potential environmental threat [1,2]. Anaerobic digestion (AD) and composting are two main technical methods to treat organic waste [3]. Because it can directly convert high water content materials and recover energy [4] and fertilizer nutrients [5], anaerobic digestion receives excellent attention and has been widely studied and applied over the world [6]. To sum up, the anaerobic transformation of organic waste involves environmental protection, alternative energy production, and emission reduction of total greenhouse gases (GHG) by replacing some fossil energy fuels [7]. However, some organic wastes contain much refractory organic matter (lignocellulosic components) and various microorganisms, so it is challenging to earn ideal or economic biogas production efficiency in an actual trial [8]. Moreover, the investment in biogas projects is high and hard to be compressed because of the need for giant digestion tanks [9] and waste air treatment techniques to avoid emissions and improve biogas quality before use [10]. Therefore, many types of research have been
focusing on improving biomass material’s conversion efficiency for lower-cost biogas production [11].

Many studies have shown the hydrolysis of organic macromolecules is the key to improving anaerobic biogas production efficiency [12]. Some pretreatment methods have been developed for promoting hydrolysis, such as mechanical, chemical, and thermal [13,14]. The reported specific methods include heat [15,16], ultrasound [17], advanced oxidation [18], alkaline cracking, dry milling, hot water, steam explosion [19], degrease [20], etc. Since the energy input required for heat treatment (HP) can be supplied by choosing thermal energy with a lower energy grade (low ratio of exergy/energy), recovering waste heat energy or directly burning some fuel nearby can be used to provide energy for the HP process. This makes HP cheaper and easy to be applied to practical projects. HP has been successfully applied at an industrial scale and is one of the earliest methods recognized as having the potential to improve AD [21,22] and has been widely concerned and studied in enhancing the bioavailability of biomass. This pretreatment strategy can break up cell membranes resulting in soluble organic substrates that are easily be hydrolyzed during digestion [16–19]. Moreover, HP can also effectively weaken pathogens’ reproduction and decrease feed liquid viscosity [23].

HP is usually performed over a wide temperature range of 50–250 °C and can be divided into two categories according to temperature, as shown in Figure 1: high-temperature pretreatment (HTPT) and low-temperature pretreatment (LTPT) [21,24]. The required temperature of HTPT is above 140 °C, and its main purpose is to promote the dissolution or partial dissolution of recalcitrant components and improve the bioavailability of biomass materials [25–27]. Such as, previous studies proved that hemicellulose and cellulose solubilize at temperatures >150 °C and 200 °C, respectively [28,29]. While the LTPT employs temperatures below 140 °C (or below 100 °C) for AD improvement [21,30–32]. Protot et al. [33] stated that thermal pretreatment below 100 °C can impel the deflocculation of macromolecules. Neyens and Bayens [34] reported that LTPT resulted in the solubilization of proteins and particulate carbohydrates in sludge. Some authors proposed that pretreatment below 100 °C can be considered a biological process since biomass solubilization occurs because of a higher activity of thermophilic and hyperthermophilic bacteria populations [35,36]. In this case, exposure time also plays a more important role [37].

![Figure 1. Heat treatment interval according to temperature.](image)

Compared with HTPT, LTPT is easier to obtain positive energy production [38], and no Maillard reaction occurs in the pretreatment [39]. The Maillard reaction between amino acids and reducing sugars can produce recalcitrant products (e.g., melanoidin) that are less bioavailable for biogas production and may act as microbial inhibitors. Moreover, more aggressive pretreatments require complex operating structures and security measures not generally found at biogas plants or farms. On the contrary, any biogas plant can perform thermal pretreatment at low temperatures using the thermal energy produced by combined heat and power (CHP) [38].

Previous studies have proved that LTPT can stimulate thermophilic bacteria, solubilize organic particles, improve biodegradability [40] or promote organic-N, P mineralization [41,42]. However, the obtained effects on anaerobic performance from LTPT are
inconsistent and variable with the difference in pretreating biomass, temperature, exposure time, and AD operation [42]. Such as, both Peces et al. [43] and Ferrer et al. [44] reported a negligible methane improvement when pretreating brewer’s spent grain and water hyacinth at 80 °C. However, Menardo et al. [45] demonstrated that pretreatment at 90 °C could significantly enhance the methane production of barley straw (42%) and wheat straw (62%). Therefore, previous studies showed that LTPT does not always work, and its resultant variations have not been clearly summarized. This makes it challenging to promote and apply LTPT in practical engineering.

Thus, this work mainly addresses the heat pretreatment of biomass below 140 °C, emphasizing pretreatment benefits evaluated for different biomass types. Although several previous reviews have introduced LTPT, most of them are with other pretreatment methods lacking comprehensiveness. This study reviews the use of LTPT before AD of many biomass and fills a gap in data mining about LTPT technologies applied to biomass for biogas production.

2. General Characteristics of Heat-Treated Biomass

Hydrothermal pretreatment will change biomass material characteristics, such as pH, conductivity, nutrient release, organic matter dissolution, etc. Hren et al. [46] reported pH values decreased during the pretreatments of riverbank grass, sewage sludge, and rumen fluid. The reason may be that thermal pretreatment forms amino acids and fatty acids. Thermal treatment can promote the release of intracellular ions to the outside, which may be the main reason for the change of conductivity, especially in biomass such as grass [46], vegetables [47], microalgae [48], and sludge. Due to the accumulation of nutrients in the process of sludge formation, thermal pretreatment of sludge can effectively release nitrogen (NH$_4^+$-N) and soluble phosphorus (PO$_4^{3-}$-P) [32]. Yan et al. [49] reported the concentration of NH$_4^+$-N increased from 21.0 ± 0.6 mg·L$^{-1}$ to 200.9 ± 2.9 mg·L$^{-1}$ after pretreatment at 100 °C. Many studies have pointed out that increasing the organic matter dissolution is the main reason for pretreatment to improve the hydrolysis step in anaerobic degradation [50]. Rodriguez-Verde et al. [51] found the conducted thermal pretreatment of manure resulted in an increase in the solubilized fraction from 0.20 to 0.38 and 0.43 at 70 °C and 90 °C, respectively. Passos et al. [36] also proved that the pretreatment at 95 °C for ten hours increased VS solubilization of microalgal biomass by 1188%.

3. Municipal Solid Waste (MSW)

With the acceleration and completion of urbanization in developing countries, the amount of MSW is also rising rapidly. Statistics show that more than 4 billion tonnes of solid waste, nearly half of which is MSW, is produced worldwide each year [52]. These wastes have the characteristics of large yield, high water content, and complex composition and are potentially harmful. MSW typically consists of 46% organic content (food/kitchen waste, activated sludge, yard waste, wood, and craft residues), followed by 17% paper, 10% plastic, 5% glass, 4% metal, and 18% others [53]. Many reports have pointed out that the organic fraction of MSW (OFMSW) is an excellent raw material for biogas production because it is rich in organic substances such as starch, protein, and oil [54–56]. Thermal pretreatment can also solubilize food waste to improve the AD performance of OFMSW [57]. However, due to the different living habits, the components of OFMSW produced from other regions are various, and improving biogas production by thermal pretreatment will also vary. Thus, it is necessary to summarize the LTPT results of OFMSW to explore the empirical law more suitable for practical applications. Two typical municipal organic wastes, food/kitchen waste and waste-activated sludge (WAS), are used here as the focus of the discussion. The changes in biogas or methane production due to thermal treatment are summarized in Table 1, showing that LTPT below 120 °C is effective for both food/kitchen waste and WAS in improving biogas production, and pretreatment temperature greater than 70 °C and lasting more than 15 min are recommended. If lower than 70 °C, a longer duration may be needed for the pretreatment to obtain a significant biogas yield increase, e.g., 84 h [58].
Table 1. Biogas production of OFMSW after LTPT (Temp. ≤ 140 °C).

| Biomass                  | AD Condition   | AD Mode | Pretreatment | Biogas Yield | CH₄ Yield   | Increase   | Reference |
|-------------------------|----------------|---------|--------------|--------------|-------------|------------|-----------|
| Waste activated sludge  | Mesophilic     | Batch (20 days) | Control | 34.8 mL·g⁻¹ ODS | Negative | [37]       |           |
|                         |                |         | 70          | 15 min       | 28.3 mL·g⁻¹ ODS | Negative |           |
|                         |                |         | 30 min      |              | 33.6 mL·g⁻¹ ODS | Less evident |           |
|                         |                |         | 60 min      |              | 35.3 mL·g⁻¹ ODS |           |           |
|                         |                |         | 80 °C       | 15 min       | 21.3 mL·g⁻¹ ODS | Negative | 1.4 times |
|                         |                |         | 30 min      |              | 48.0 mL·g⁻¹ ODS | 2.2 times |           |
|                         |                |         | 60 min      |              | 75.6 mL·g⁻¹ ODS |           |           |
|                         |                |         | 90 °C       | 15 min       | 76.7 mL·g⁻¹ ODS | 2.2 times |           |
|                         |                |         | 30 min      |              | 142 mL·g⁻¹ ODS | 4 times |           |
|                         |                |         | 60 min      |              | 378 mL·g⁻¹ ODS | 11 times |           |
| Sludge                  | Mesophilic     | Batch   | Control     | 87 mL·g⁻¹ VS |            |           | [40]      |
|                         |                |         | 60 °C       | 30 min      | 93 mL·g⁻¹ VS | 7.3%      |           |
|                         |                |         | 70 °C       | 100 mL·g⁻¹ VS |            | 15.6%     |           |
|                         |                |         | 80 °C       | 108 mL·g⁻¹ VS |            | 24.4%     |           |
|                         | Semi-continuous|         | control     | 333 mL·g⁻¹ VS |            |           |           |
|                         |                |         | 70 °C       | 30 min      | 383 mL·g⁻¹ VS | 11.7%     |           |
| Kitchen waste           | Thermophilic   | Semi-continuous | Control | 1360 mL·L⁻¹ |            |           | [58]      |
|                         |                |         | 37 °C       | 1420 mL·L⁻¹ | 4.4%      |           |           |
|                         |                |         | 50 °C       | 2020 mL·L⁻¹ | 48.5%     |           |           |
|                         |                |         | 60 °C       | 2540 mL·L⁻¹ | 86.8%     |           |           |
| Kitchen waste           | Thermophilic   | Batch   | Blank       | 6.5 L·L⁻¹ |            |           | [59]      |
|                         |                |         | 120 °C      | 30 min      | 7.2 L·L⁻¹ | 10.8%     |           |
| Food waste              | Mesophilic     | Batch (14 days) | blank | 280 mL·g⁻¹ VS |            |           | [60]      |
|                         |                |         | 70 °C       | 2 h         | 290 mL·g⁻¹ VS | Less evident |           |
| Kitchen waste           | Mesophilic,    | Batch (40 days) | Blank | 614 mL·g⁻¹ VS |            |           | [61]      |
|                         | Semi-continuous|         | 55 °C       | 15 min      | 579 mL·g⁻¹ VS | Negative |           |
|                         |                |         | 70 °C       | 90 min      | 822 mL·g⁻¹ VS | 33.9%     |           |
|                         |                |         | 90 °C       | 30 min      | 781 mL·g⁻¹ VS | 27.2%     |           |
|                         |                |         | 120 °C      | 15 min      | 777 mL·g⁻¹ VS | 26.5%     |           |
| WAS                     | Mesophilic,    | Batch   | Control     | 2507 L·m⁻³ WAS |            |           | [62]      |
|                         | Semi-continuous|         | 121 °C      | 30 min      | 3390 L·m⁻³ WAS | 35.2%     |           |
| Food waste              | Mesophilic     | Batch (45 days) | Control | 555 mL·g⁻¹ VS |            |           | [63]      |
|                         |                |         | 60 °C       | 10 min      | 575 mL·g⁻¹ VS | 3.6%      | 16.2%     |
|                         |                |         | 20 min      | 645 mL·g⁻¹ VS |            |           |           |
|                         |                |         | 80 °C       | 10 min      | 653 mL·g⁻¹ VS | 17.7%     | 24.7%     |
|                         |                |         | 20 min      | 692 mL·g⁻¹ VS |            |           |           |
|                         |                |         | 100 °C      | 10 min      | 783 mL·g⁻¹ VS | 41.1%     | 31.2%     |
|                         |                |         | 20 min      | 728 mL·g⁻¹ VS |            |           |           |
| Food waste              | Mesophilic,    | Semi-continuous | control | 310 mL·g⁻¹ VS |            |           | [64]      |
|                         | Semi-continuous|         | 100 °C      | 30 min      | 383 mL·g⁻¹ VS | 23.68%     |           |
| Sewage sludge           | Mesophilic,    | Semi-continuous | control | 107 mL·g⁻¹ VS |            |           | [65]      |
|                         | Semi-continuous|         | 134 °C      | 30 min      | 210 mL·g⁻¹ VS | 2 times |           |
| Sludge                  | Mesophilic,    | Batch   | Control     | -           | 53 mL·g⁻¹ VS |            | [66]      |
|                         |                |         | 90 °C       | 1 h         | 177 mL·g⁻¹ VS | 3.33 times |           |
|                         |                |         |              | 24 h        | 195 mL·g⁻¹ VS | 3.68 times |           |
|                         |                |         |              | 36 h        | 295 mL·g⁻¹ VS | 5.56 times |           |
Table 1. Cont.

| Biomass                  | AD Condition | AD Mode | Pretreatment Temp. °C | Time  | Biogas Yield | CH₄ Yield | Increase | Reference |
|-------------------------|--------------|---------|-----------------------|-------|--------------|-----------|----------|-----------|
| Waste activated sludge  | Mesophilic   | Batch   | Control               | -     | 89 mL·g⁻¹ VSS|           |          |           |
|                         |              |         | 60 °C                 | 30 min| 101 mL·g⁻¹ VSS| 13.5%     |          | [67]      |
|                         |              |         | 80 °C                 | 30 min| 113 mL·g⁻¹ VSS| 27.0%     |          |           |
|                         |              |         | 100 °C                | 30 min| 114 mL·g⁻¹ VSS| 28.1%     |          |           |
|                         |              |         | 120 °C                | 30 min| 115 mL·g⁻¹ VSS| 29.2%     |          |           |

### 3.1. Food/Kitchen Waste

Due to the high water content and biodegradability, food waste (FW) and kitchen waste (KW) are suitable for AD to produce biogas. In addition, the content of lignocellulosic compounds is low in this biomass, so gentle pretreatment can satisfy the need to improve AD performance. Ma et al. [59] reported an 11% improvement in methane production when pretreating KW at 120 °C for 30 min. Li et al. [61] displayed that temperature coupled with exposure time affects the subsequent improvement of methane production of KW. Such as, biogas production from pretreatment at 70 °C for 90 min is higher than those for a shorter duration (10–60 min), while the treatments at 90 °C and 120 °C obtained the maximum biogas yields lasting 10 and 30 min, respectively. They also pointed out the Maillard reaction would be induced when the pretreatment temperature went up to 140 °C, which reduced the methane yield. This suggests that high-temperature pretreatment is unsuitable for FW and KW, and its high sugar and protein content will lead to the formation of some adverse reactants [68]. Kuo and Cheng [58] conducted thermal treatment of KW at different temperatures to improve hydrolysis and chemical oxygen demand (COD) removal, which showed that pre-treatment at 60 °C yielded the highest total COD (TCOD) removal efficiency (79.2%) after 300 h reaction. The pretreatment at 70 °C obtained the maximum biogas yield of 822 mL·g⁻¹ VS in an investigation from 55 °C to 160 °C [61]. Sometimes, thermal treatment hardly improves the cumulative biogas yield, but it can change the biogas production rate. Wang et al. [60] pretreated FW at 70 °C for two hours, and the methane production increased only by 2.7%. Still, the pretreatment halved the time to produce the same quantity of methane compared to the anaerobic digestion of fresh FW. This suggests that heat treatment improves the kinetic features of AD.

Other studies have shown that thermal treatment coupled with chemical reagents can better solubilize KW. Seyed Abbas et al. [69] conducted a thermo-chemical pretreatment on kitchen waste (cooked rice, pasta, ground beef, apple, etc.). They showed that the pretreatment at 120 °C with NaOH 5N can provide the best conditions to increase biogas and methane production. Ma et al. [59] proved that thermal-acid pretreatment at room temperature (pH = 2) obtained a better solubilization rate of kitchen waste than other pretreatments with more severe conditions.

### 3.2. WAS

The heat treatment of WAS was shown as early as 1970 as an effective pretreatment method for AD [70]. Then many studies have proven that thermal pretreatment can accelerate hydrolysis, shorten sludge’s digestion time, and increase biogas production. It has been commercially operational at full scale since 1995 [71]. The temperature range of sludge heat treatment reported in the previous literature is also relatively wide, at 60–270 °C [30]. However, research on LTPT of sludge waste has not been well summarized, and its improvement in biogas production can be more than five times [66] (Table 1). Appels et al. [37] showed that thermal pretreatment could effectively dissolve both organic and inorganic matter, and the subsequent anaerobic digestion efficiency of sludge at 90 °C, 60 min of pretreatment can be improved 11-fold. Kim et al. [62] found that after 30 min of heat treatment at 121 °C, the damage rate of volatile solids (VS) increased by 30%,
and gas production increased by 32%. Nges et al. [72] conducted anaerobic digestion of biogas sludge through experiments and pretreated it at 50 °C for 48 h, resulting in an 11% increase in methane production. For high solid sludge, low temperature thermal pretreatment is also effective, Liao et al. [40] pretreated high solid sludge (TS = 15%) at low temperature (60–80 °C) and carried out intermittent anaerobic digestion experiment and continuous anaerobic digestion experiment and found that low-temperature pretreatment could accelerate digestion of high solid sludge and improve biogas production.

Similarly, applying heat treatment coupled with chemical reagents in sludge pretreatment has also received attention. Xiao et al. [73] conducted high-temperature thermal pretreatment (160 °C) and LTPT by adding alkali (60 °C, pH 12.0) for sludge, respectively, and obtained similar methane production and organic matter removals. This suggests that chemical assistance can compensate for the shortage of pretreatment at a lower temperature. Zheng et al. [74] also reached a similar conclusion through experiments, low-temperature thermos-alkali pretreatment (60 °C, pH 12.0) has better energy efficiency. However, the auxiliary chemical reagent will increase the treatment cost. Appropriate pretreatment only for the substrates with poorer biodegradability before mixed AD could reduce the capital and operating costs [75]. Some studies have also proven that adding chemical reagents in thermal pretreatment may produce some adverse effects. Gunerhan et al. [76] reported that increasing the concentration of NaOH and HCl in thermal pretreatment at 60–100 °C can enhance the COD solubilization of fruit and vegetable harvesting wastes. In contrast, it reduced the concentration of soluble sugar which can be directly converted to methane. It can be concluded from these precious studies that whether heat treatment alone or chemically assisted, the treatment effect depends on the specific substrate characteristics and the set operating conditions.

4. Animal Manure Biomass

Meat, egg, and milk have become essential food for human life worldwide. It is crucial for human nutrition intake and improving living standards: about 270 million dairy cows and 677 million pigs worldwide [77]. Similarly, the annual amount of fecal production is also significant, which has a tremendous potential threat to the human living environment. Recycling energy and fertilizer through AD is helpful for animal manure treatment [78]. However, those initial characteristics of high recalcitrant fibers content, high viscosity, and rich in pathogens are unfavorable to the AD of manure biomass for biogas production. Many facts have proved that the thermal pretreatment method can weaken these adverse factors [12,14,79]. However, due to the differences in chemical composition and physical properties, the reaction results of thermal treatment of different types of animal manures may also be different. For instance, the methane yield of pig manure and sewage waste increased after heating treatment, while that of dairy manure decreased by 6.9% [80]. Therefore, the present review of LTPT of manure biomass is carried out according to different categories and mainly focuses on swine/pig manure, cow/dairy/cattle manure, and chicken/poultry manure.

4.1. Pig/Swine Manure

Many studies have shown that thermal pretreatment can significantly improve the biogas production performance of swine/pig manure. Menardo et al. [38] pretreated dehydrated pig manure (PM), digested it at 120 °C and found that methane production increased by 35–171%. Increasing soluble COD may be the main reason for improving biogas production of PM after LTPT. Huang et al. [42] pretreated swine manure (SM) at 110–130 °C for 30 min and achieved a CH4 yield of 280.18–328.93 mL·g−1 VSfed increasing 14–34%. The reason may be the increase of 13–26% in soluble organic carbon concentration after pretreatment. Bonmati et al. [81] found that the concentration of soluble compounds in pig slurry rose after hydrothermal pretreatment below 90 °C, increasing methane yield. Some studies showed that the inhibitor concentration of PM liquid is low after LTPT, which did not affect biogas production. Fang et al. [39] reported that in both sludge and SM
samples, the total biogas and methane productions were enhanced by the 125 °C heating treatment but inhibited by the 225 °C treatment, and they also pointed out the pretreatments at higher temperature may produce inhibitors (e.g., melanoidin). Another study also found the treatment at 100 °C obtained the maximum biogas yield of 0.48 ± 0.02 L·g^{-1} VS, a 30% increase from the raw manure sample. In comparison, biogas production from thermally treated at 130 °C and 150 °C showed less biogas production. The speculated potential reason may be that high temperatures formed complex organic compounds which are difficult to degrade [82]. LTPT can also improve microbial distribution in the AD system of PM. Mladenovska et al. [83] pretreated the mixture of cattle and swine manure at 100–140 °C and obtained an enhancement of specific methane yield in the range of 9–24% and 10–17% for the 20- and 40-min treatment, respectively. Moreover, they also found that continuous feeding of heat-treated PM can affect microbial species richness in a continuous stirring tank reactor and give it the ability to preserve high biogas production.

However, a few studies have also found that LTPT can improve COD’s solubility. Still, biogas production is not significant, which may be due to the high fiber content in the manure samples. Raju et al. [79] found that PM improved biogas production at pretreatment temperatures of 125 °C, while pretreatment at 100 °C did not improve. They also revealed that LTPT has little effect on the cellulose and hemicellulose fractions. Carrère et al. proved that pretreatment of 70–90 °C can only increase the soluble substances and biogas production of the liquid part of PM, while improving the overall biogas production needs a higher temperature of >150 °C [84]. Table 2 summarizes the current review of LTPT of pig/swine manure and shows an increase of 7.1% to 170% in biogas or methane yield from the tests below 140 °C. Effective pretreatment is mainly concentrated above 100 °C and lasts more than 30 min.

**Table 2. Biogas production variation of pig manure after LTPT (Temp. ≤ 140 °C).**

| Biomass                      | AD Condition | AD Mode  | Pretreatment | Biogas Yield * | CH₄ Yield * | Increase | Reference |
|------------------------------|--------------|----------|--------------|----------------|-------------|----------|-----------|
| Swine manure                 | Mesophilic   | Batch    | Blank –      | 79             | –           | –        | [38]      |
|                              | Blank –      | 120 30 min | 213          | 170%           |             |          |           |
| Swine manure                 | Mesophilic   | Batch    | Blank –      | 0.79 COD·COD⁻¹ | 8.9%        |          | [39]      |
|                              | Blank –      | 125 4 h  | 0.86         |              |             |          |           |
| Swine manure                 | Mesophilic   | Batch    | Blank –      | 204           | –           | –        | [42]      |
|                              | Blank –      | 110 °C 30 min | 231          | 13.2%         |             |          |           |
|                              | Blank –      | 130 °C 30 min | 271          | 32.8%         |             |          |           |
| Pig manure                   | Mesophilic   | Batch    | Blank –      | 215           | –           | –        | [79]      |
|                              | Blank –      | 100 °C 15 min | 208          | –             | –          |          |           |
| Pig manure                   | Mesophilic   | Batch    | Blank –      | 234           | –           | –        | [81]      |
| Pig slurry                   | Thermophilic | Batch    | Blank –      | 348           | –           | –        |          |
| Pig manure                   | Mesophilic   | Batch    | Blank –      | 558           | –           | 60.4%    |          |
| A mixture of cattle manure    | Thermophilic | Batch    | Blank –      | 475           | –           | 30%      | [82]      |
| and swine manure             |             |          | Blank –      | 233           | –           | –        |           |
|                             | 100 °C       | 20 min 40 min | 289          | 24.0%         |             |          |           |
|                             | 120 °C       | 20 min 40 min | 254          | 9.0%          |             |          |           |
|                             | 140 °C       | 20 min 40 min | 274          | 17.6%         |             |          |           |
|                             | Continuous   | Blank –   | 238          | –             |             |          |           |
|                             | 140 °C       | Blank –   | 255          | 7.1%          |             |          |           |
Table 2. Cont.

| Biomass        | AD Condition | AD Mode | Pretreatment | Biogas Yield * | CH₄ Yield * | Increase | Reference |
|----------------|--------------|---------|--------------|----------------|-------------|----------|-----------|
| Pig manure 1   | Mesophilic   | Batch   | Blank – – –  | 112 mL·g⁻¹ COD| –           | –        | [84]      |
|                |              | (40 days)| 70 3 h –     | 98             | –           | Less evident |           |
|                |              |         | 90 3 h –     | 127            |             |          |           |
| Pig manure 2   | Mesophilic   | Batch   | Blank – – –  | 91             | –           | –        |           |
|                |              | (40 days)| 135 20 min – | 84             |             | Less evident |           |

* All data units are “mL·g⁻¹ VS” except for the different units indicated in the table.

4.2. Cattle/Dairy Manure

The high content of indigestible fiber is an essential feature that distinguishes cattle/dairy manure from other animal manures, while LTPT is hard to decompose these resistant components. As a result, the effective pretreatment temperature of cattle/dairy manure is higher than other manure. Its pretreating temperatures range from 100 to 140 °C (Table 3), and the exposure time may be required to be extended moderately. Passos et al. [85] found the only conditions that reached methane yield increments were those with long exposure times (i.e., 37 °C for 12 and 24 h), which were 3.6% and 20.5% higher than untreated dairy manure (DM), respectively. At the same time, the thermal pretreatments for 5 and 30 min did not enhance the final methane yield. Wilton et al. [22] reported that the methane production of DM increased by 37% after thermal treatment at 125 °C and for 30 min, while the treatments at a temperature below 125 °C and duration time less than 35 min showed no significant difference in methane production. The reason may be that the bovine gut had previously digested DM, a process that already alters the substrate’s lignocellulosic content, which may render thermal pretreatment redundant.

Table 3. Biogas production variation of dairy/cattle manure after LTPT (Temp. ≤ 140).

| Biomass         | Operating Condition | Operating Mode | Pretreatment | Biogas Yield | CH₄ Yield | Increase | Reference |
|-----------------|---------------------|----------------|--------------|--------------|-----------|----------|-----------|
| Dairy cow manure| Mesophilic           | Batch (40 days)| 125 °C       | 37.5 min     | 34%       | [22]     |
| Cattle manure   | Mesophilic           | Batch (27 days)| Blank – –     | 244 mL·g⁻¹ VS| –         | –        | [79]      |
| Dairy cow manure| Mesophilic           | Batch (40 days)| 37 °C        | – 12 h       | 3.6%      | [85]     |
|                 |                      |                | 100 °C       | – 15 min     | 222       | –        |          |
|                 |                      |                | 125 °C       | – 15 min     | 242       | –        |          |
| 2:1:1 mixtures  | Mesophilic           | Batch (30 days)| Blank – –     | 180.5 mL·g⁻¹ TS| –         | –        | [86]      |
| CM:CS: SBP      |                      |                | 100 °C       | – 10 min     | 196       | 8.3      |
|                 |                      |                |              | 20 min       | 216       | 19.3     |
|                 |                      |                |              | 30 min       | 236       | 30.4     |
|                 |                      |                |              | 60 min       | 256       | 41.5     |
|                 |                      |                |              | 120 min      | 242       | 34.1     |
|                 |                      |                | 120 °C       | – 10 min     | 201       | 11.5     |
|                 |                      |                |              | 20 min       | 212       | 17.4     |
|                 |                      |                |              | 30 min       | 243       | 34.5     |
|                 |                      |                |              | 60 min       | 286       | 58.1     |
|                 |                      |                |              | 120 min      | 288       | 59.6     |
Table 3. Cont.

| Biomass         | Operating Condition | Operating Mode | Pretreatment | Biogas Yield | CH₄ Yield | Increase | Reference |
|-----------------|---------------------|----------------|--------------|--------------|-----------|----------|-----------|
| Cattle manure 1 solid | Mesophilic          | Batch (30 days) | Blank | 203 mL·g⁻¹ OM |           |          |           |
| Cattle manure 1 liquid |                 |                | 140 °C, 5 min | 306          |           | 50       | [87]      |
| Cattle manure 2 solid |                 |                | Blank | 168          |           |          |           |
| Cattle manure 2 liquid |                 |                | 140 °C, 5 min | 186          |           | 11       |           |
| Blank | Cattle manure 2 solid |                | Blank | 259          |           | 15       |           |
| Blank | Cattle manure 2 liquid |                | Blank | 162          |           |          |           |

Therefore, many researchers have been exploring the heat treatment of DM at a higher temperature, mixed with other materials, or assisted by chemical reagents [79,86,87]. Şenol et al. [86] conducted a co-digestion of CM, corn silage, and sugar beet pulp (2:1:1), and pretreating at 100 °C gained a 40% increase in biogas production. Additionally, several studies reported a significant hydrolysis enhancement after heat treatment of dairy/cattle manure by adding chemical reagents (e.g., oxalic acid, sulfuric acid) [88–90].

Similarly, inhibition resulting from heat treatment also appears in the AD of dairy/cattle manure, which may occur in both low- and high-temperature cases. Raju et al. [79] found the methane potential of CM decreased by about 10% at the pretreatment condition of 100 °C. Chan et al. [91] reported preheating dairy manure at a temperature below 100 °C and with acid would decrease methane production. Budde et al. [87] found that the abundance of inhibitors and other non-digestible substances led to lower methane yields in the pretreatment at 220 °C than those obtained from untreated CM. In conclusion, higher temperatures, longer exposure time, or chemical reagent assistance may favor the thermal treatment of dairy/cattle manure. Moreover, paying attention to the generation of inhibitors is necessary.

4.3. Chicken/Poultry Manure

Chicken/poultry manure has also been proven to produce biogas. Its yield can reach more than 400 mL·g⁻¹ TS [51,92], which is better than pig manure and cow manure, probably because chicken manure (CHM) contains undigested feed. However, the content of organic nitrogen (protein, urea, uric acid) in chicken/poultry manure is higher than that in other manures, mainly due to the need for the rapid synthesis of eggs, feathers, and meat protein. During AD, the organic nitrogen is transformed into ammonia, inhibiting the anaerobic biogas generation process [93,94]. Therefore, many previous studies have focused on pretreatment to improve bioavailability and remove partial ammonia. The most common method is heating and its combination with stripping. Table 4 summarizes some of the studies and shows that the pretreatment temperature is mainly concentrated around 100 °C and below, and individual heat treatment does not perform well. The survey by Ardic and Taner [95] found pretreating CHM at 100 °C for two hours improved methane production. Rodríguez-Verde et al. [51] conducted a pretreatment of poultry-pig manure that consisted of temperature simultaneously with ammonia stripping, resulting in a nitrogen removal efficiency of 72% and a 1.2-fold higher methane production. Elasri et al. [96] obtained a biogas yield of 230.58 mL·g⁻¹ COD after two-step pretreatments of heating at 105 °C for 24 h following a fine grinding, creating 3–5 times higher than the untreated group. Yin et al. [97] performed a thermophilic pretreatment (70 °C) of CHM, including thermal and ammonia stripping, and found that the methane yield of prehydrolyzed CHM reached 518 mL·g⁻¹ VS, which was 54.6% higher than the control reactor. Yin et al. [98] created an innovative two-stage AD by combining thermal stripping pretreatment (70 °C) and an
anaerobic membrane bioreactor, bringing a hydrolysis efficiency of 72.4% and methane yield of 352 mL·g⁻¹ VS_in (growth ≈ 65%).

Table 4. Biogas production variation of chicken manure after LTPT (Temp. ≤ 140).

| Biomass        | AD Condition  | AD Mode    | Pretreatment                      | Biogas Yield   | CH₄ Yield   | Increase   | Reference |
|----------------|---------------|------------|----------------------------------|----------------|-------------|------------|-----------|
| Chicken droppings | Mesophilic   | Batch (40 days) | Blank (70 °C) | 1 d 11.2–20 m³/ton | -            | -          | [96]      |
|                |               |            | 2 d                            | 64.4 m³/ton    | -            | 3.2–4.7 fold |          |
|                |               |            | 3 d                            |                |              |            |          |
|                |               |            | 5 d                            |                |              |            |          |
| Chicken manure | Mesophilic   | Batch      | Stripping at 70 °C | 1 d 42.8% | -            |            | [97]      |
|                |               |            | 2 d                            | 63.5%          |              |            |          |
|                |               |            | 3 d                            | 54.6%          |              |            |          |
|                |               |            | 5 d                            | 7.0%           |              |            |          |
| Chicken manure | Mesophilic   | Continuous | Stripping at 70 °C | 4 d 213 mL·g⁻¹ VS | -          |            | [98]      |
|                |               |            |                                |                |              |            |          |
| Chicken manure | Mesophilic   | Batch (27 days) | Blank | 334 mL·g⁻¹ VS | -            |            | [99]      |
|                |               |            | 100 15 min                    | 317 mL·g⁻¹ VS  | -            | Less evident |          |
|                |               |            | 125 15 min                    | 314 mL·g⁻¹ VS  | -            |            |          |

Additionally, LTPT can accelerate destroying organic molecular bonds in poultry litter by adding chemical reagents. Poultry litter soluble COD increased 2–3 times after conducting a pretreatment of 0.2 g Ca(OH)₂·g⁻¹ waste at 90 °C, while that at 20 °C showed a wick COD solubilization [50]. Zahan et al. [99] obtained a 45–51% increase of biogas in the co-digesting chicken litter, food waste, and wheat straw after thermal pretreatment at 120 °C with 5% NaOH or 3% H₂SO₄. However, note the inhibitor production of the heat pretreatment of CHM. Both higher temperatures and adding chemicals are more likely to produce inhibitors. Raju et al. [79] found no significant change in the BMP of the CHM after pre-treatment at temperatures up to 200 °C. The use of sodium hydroxide is more likely to produce inhibitors (e.g., volatile fatty acid (VFA), ammonia, furfural) than using lime, and the cations Na⁺ and K⁺ were potent methanogenic inhibitors when compared with Ca²⁺ [97].

5. Algae

Algae are tiny groups whose unique morphology can only be identified under a microscope, and have chlorophyll, can conduct photosynthesis to assimilate CO₂ and produce biomass. It is a suitable bioenergy material and can be used to produce hydrogen, hydrocarbons, bioethanol, biodiesel, or methane [36]. Many studies have reported promising results using algae as an AD substrate [35]. However, the hemicellulosic component of cell walls makes algae resistant. Thus, hydrolysis has also been pointed out as a bottleneck of the AD of algae biomass [100]. Ras et al. [101] reported that 50% of the Chlorella vulgaris did not undergo anaerobic digestion without pretreatment, even under long retention times. Therefore, performing a pretreatment is critical for improving the bioavailability of algal biomass.

Heat treatment shows a good effect on promoting algae biomass conversation. Alzate et al. [35] proved that thermal hydrolysis was the most effective method for microalgae pretreatment compared with ultrasound and biological pretreatments, supporting productivity and biodegradability increases by over 60%. Heating can promote the solubilization of particulate organic fractions and hydrolysis of polymeric organic molecules in algal biomass [102]. Kinnunen et al. [48] reported that LTPT could promote protein hydrolysis of microalgae and increase its methane yields by 23–27% in the AD process operated at 20 °C. However,
Fermentation 2022, 8, 562

solubilization is not always in direct ratio to increase biogas production. The study by González-Fernández et al. [100] showed pretreating microalgae at 90 °C for two hours achieved anaerobic biodegradability of 48% and 2.2-fold methane production (170 mL·g⁻¹).

This study also found that pre-treatment at 70 °C could not disrupt the cell wall regardless of the time. In comparison, a microscopic staining observation technique demonstrated that pretreating at 90 °C destroyed the cell wall significantly. This suggests temperature is vital in affecting the LTPT of algae. Marsolek et al. [103] reported net biogas production increased when preheated at 90 °C for 1, 3.5, or 12 h, while there was no improvement at 30 °C or 60 °C. Moreover, the exposure time was also a key affecting the heat treatment of algae. Mendez et al. proved that heating Chlorella vulgaris for 40 min showed a stronger methane production potential than those for 20 min [104,105]. Passos et al. [36] reported the best results at 75–95 °C with an exposure time of 10 h, resulting in a 34–90% increase in initial methane production rate and a 12–61% increase in final methane yield. González-Fernández et al. [106] reported pretreating Scenedesmus sp. at 90 °C for one hour obtained the maximum methane production and can better adapt to the increase of organic loading rate (OLR). Schwede et al. [107] found that heating Nannochloropis salina for one hour at 100 °C achieved 58% biogas production growth, resulting in a 58% biogas yield increase. Therefore, it can be seen that pretreatments at temperature ranged from 75 °C to 120 °C and for exposure time above 40 min are more often used in the LTPT of algae biomass (Table 5).

### Table 5. Biogas production variation of algae biomass after LTPT (Temp. ≤ 140).

| Biomass                  | AD Condition | AD Mode      | Pretreatment Temp. (°C) | Pretreatment Time (min) | Biogas Yield (mL·g⁻¹ VS) | CH₄ Yield (mL·g⁻¹ VS) | Increase | Reference |
|-------------------------|--------------|--------------|-------------------------|-------------------------|--------------------------|------------------------|----------|-----------|
| Microalgae mixture      | Mesophilic   | Batch (60 days) | Control                 | -                       | 272                      | -                      | -        | [35]      |
|                         |              |              | 110                     | 15 min                  | 323                      | 19%                    |          |           |
|                         |              |              | 140                     | 15 min                  | 362                      | 33%                    |          |           |
| Microalgae mixture b    | Mesophilic   | Batch (60 days) | Control                 | -                       | 198                      | -                      | -        |           |
|                         |              |              | 110                     | 15 min                  | 219                      | 11%                    |          |           |
|                         |              |              | 140                     | 15 min                  | 260                      | 31%                    |          |           |
| Microspore              |              |              | Control                 | -                       | 253                      | -                      | -        | [35]      |
|                         |              |              | 110                     | 15 min                  | 413                      | 61.7%                  |          |           |
|                         |              |              | 140                     | 15 min                  | 382                      | 49.8%                  |          |           |
| Microalgae              |              | Batch        | Control                 | -                       | 180                      | -                      | -        | [48]      |
|                         |              |              | 57                      | 3.8 h                   | 221                      | 22.7%                  |          |           |
| Microalgae Scenedesmus  | Mesophilic   | Batch (33 days) | Control                 | -                       | 84                       | -                      | -        | [100]     |
| sp.                     |              |              | 70                      | 3 h                     | 76                       | -                      | -        |           |
|                         |              |              | 90                      | 3 h                     | 170                      | 220%                   | -        |           |
| Nanochloropsis oculata  | Mesophilic   | Batch (12 days) | Control                 | -                       | 0.28                     | -                      | -        | [103]     |
|                         |              |              | 30                      | 4 h                     | 0.28                     | Less evident           | -        |           |
|                         |              |              | 60                      | 4 h                     | 0.27                     | Less evident           | -        |           |
|                         |              |              | 90                      | 3.5 h                   | 0.39                     | 41%                   | -        |           |
| Nanochloropsis oculata  | Mesophilic   | Batch (12 days) | Control                 | -                       | 0.32                     | -                      | -        |           |
|                         |              |              | 90                      | 1 h                     | 0.41                     | ≈30%                   | -        |           |
|                         |              |              | 90                      | 3.5 h                   | 0.43                     | -                      | -        |           |
|                         |              |              | 90                      | 12 h                    | 0.44                     | -                      | -        |           |
Table 5. Cont.

| Biomass | AD Condition | AD Mode | Pretreatment | Biogas Yield | CH₃ Yield | Increase | Reference |
|---------|--------------|---------|--------------|--------------|-----------|----------|-----------|
|         |              |         | Temp. (°C)   | Time         |           |          |           |
| Chlorella vulgaris | Mesophilic | Batch (30 days) | Control | - | - | 139 mL·g⁻¹ COD | - | [104] |
|         |              |         | 120 | 20 min | - | 180 mL·g⁻¹ COD | 29.8% |         |
|         |              |         | 120 | 40 min | - | 268 mL·g⁻¹ COD | 92.7% |         |
| Chlorella vulgaris biomass | Mesophilic | Batch (29 days) | Control | - | - | 156 mL·g⁻¹ COD | - | [105] |
|         |              |         | 140 | 10 min | - | 220 mL·g⁻¹ COD | 40.5% |         |
|         |              |         | 20 min | - | - | 226 mL·g⁻¹ COD | 44.4% |         |
| Scenedesmus sp. | Mesophilic | Continuous (HRT 15 d) |         | 90 | 1 h | - | 97 mL·g⁻¹ tCOD | 2.9% | [106] |
|         |              |         | Continuous (HRT 15 d) |         |         | - | 11 mL·g⁻¹ tCOD | 3.4% |         |
| N. salina biomass | Mesophilic | Batch (40 days) | Control | - | - | 547 mL·g⁻¹ VS | - | - | [107] |
|         |              |         | Boiling | - | - | 487 mL·g⁻¹ VS | - | 40.3% |         |
|         |              |         | 100 | 8 h | | 549 mL·g⁻¹ VS | - | 58.2% |         |
| Chlorella sp. | Mesophilic | Batch (90 days) | 120 | 30 min | - | 0.34 L·g⁻¹ VS | Negative | [108] |
| Nannochloropsis sp. | Mesophilic | Batch (90 days) | 120 | 30 min | - | 0.36 L·g⁻¹ VS | ≈30% |         |
| T. weissflogii | Mesophilic | Batch (90 days) | 120 | 30 min | - | 0.38 L·g⁻¹ VS | ≈10% |         |
| Tetraselmis sp. | Mesophilic | Batch (90 days) | 120 | 30 min | - | 0.42 L·g⁻¹ VS | Less evident | [108] |
| Pavlova cf sp. | Mesophilic | Batch (90 days) | 120 | 30 min | - | 0.51 L·g⁻¹ VS | Negative | [109] |
| Palmaria palmata | Mesophilic | Batch (90 days) | 120 | 30 min | - | 308 mL·g⁻¹ VS | - | [109] |
|         |              |         | 20 | 24 h | - | 328 mL·g⁻¹ VS (20°C) | Less evident |         |
|         |              |         | 120 | 20 min | - | 296 mL·g⁻¹ VS | Less evident |         |

a: 40% Chlamydomonas, 20% Scenedesmus and 40% Nannochloropsis; b: 58% Acutodesmus obliquus, 36% Oocystis sp., 1% Phormidium, and 5% Nitzschia sp.

Other studies have focused on algae species’ differences in heat treatment response. Alzate et al. [35] found that different microalga species exhibited different sensitivity to heat, such as Microspora harvested from the surface of a photobioreactor showed substantial growth in methane production after pretreating at 110 °C for 15 min, while other algae biomass only increased less than 20%. Bohutskyi et al. [108] proved the differences in COD solubilization between different varieties. The wall-less Pavlova cf sp. and glycoprotein-based cell wall Tetraselmis sp. improved their solubilization after pretreatment at 120 °C for 30 min. At the same time, Chlorella sp., Nannochloropsis sp., and Thalassiosira weissflogii showed no effect on heat pretreatment. The difference in cell wall composition may be the reason why different species of algae have various sensitivities to heat treatment. Such as, Scenedesmus sp. was highlighted as the hardest digesting microalgae since its cell wall presents some polymers that confer these microalgae high resistance to bacterial attack [100,106]. Algae with a glycoprotein cell wall or a frustule-covered wall are suitable substrates for AD without pretreatment. In contrast, polysaccharide-based cell walls present a lower methane yield without pretreatment [109].

Some literature also proved that combining heat and chemical methods can improve biogas production. Bohutskyi et al. [108] reported that thermochemical pretreatment increased methane yields of Chlorella and Nannochloropsis by 30% and 40%, respectively, because of enhancing biomass solubilization. Jard et al. [109] found that heat treatment
without chemical reagent affected little on the methane potential of \textit{P. palmate.}, while the soda assisting pretreatments enhanced 11–13\% of methane production at lower temperatures ranging from 70 to 85 °C. However, some studies believe adding chemicals can improve the hydrolysis of organic matter but disagree that it will necessarily increase biogas production, and even have proven it has a negative effect. Mendez et al. [104] demonstrated thermal alkali and acid treatments had a 5-fold and a 7-fold increasing soluble carbohydrates, respectively. However, thermal treatment without chemicals created the highest methane production.

In conclusion, all temperatures, exposure time, algae species, and chemicals are essential for affecting the LTPT. Different algae biomass will lead to varying reflections for heating treatment, and chemical reagents are unnecessary. This may be a remarkable difference between algae and other biomass.

6. Lignocellulosic Biomass (LIGB)

Typical LIGB materials are characterized by the high content of hemicellulose, cellulose, and lignin, such as crop straw, grass, weeds, etc. [110]. This biomass is considered challenging to decompose and must be pretreated before fermentation [28]. Many methods have been developed to depolymerize the lignocellulose complex and improve its bioavailability, such as physical, mechanical, irradiation, and chemical [13]. Among them, thermal pretreatment can be considered an exciting choice for LIGB to achieve solubilization, improve hydrolysis, and increase biogas production [111].

Although many studies have shown the effective hydrolysis temperature of hemicellulose and cellulose is above 150 °C, the thermal pretreatment to LIGB below 140 °C still received some attention. Once a part of lignin could be dissolved in water at a lower temperature, more cellulose and hemicellulose may become available for degradation, further increasing net energy production [112,113]. In addition, higher temperature pretreatment of LIGB may produce inhibitors. Wang et al. [114] conducted a hydrothermal pretreatment at a higher temperature (>210 °C), but it did not improve the anaerobic digestion of rice straw and showed severe inhibition. Thus, they recommended pretreating at a lower temperature. Therefore, it is necessary to summarize the LTPT studies of LIGB to explore more economical pretreatment methods.

Luo et al. [112] pretreated rice straw at temperatures from 90 °C to 130 °C for 150 min, improving lignocellulose degradation and VFAs production, and pretreating at 100 °C earned the maximum methane yield. Rajput et al. [115] found that biogas production from 120 °C and 140 °C thermally pretreated wheat straw was 22\% and 29.2\% higher than the raw wheat straw. In their another study, co-digesting preheating wheat straw and sunflower meal obtained a similar result [116]. Li et al. [110] found that thermal pretreatment increased \textit{Pennisetum}’s maximum biogas production rate and total methane yield, with an improvement of around 7\% and 8\%, respectively. Montoya-Rosales et al. [117] reported that thermal pretreating bean straw at 121 °C for one hour showed effectiveness in hemicellulose degradation (60\%), cellulose solubilization (51\%), and improving biogas yield (145.4 mL·g⁻¹ COD). Bolado-Rodriguez preheated Wheat straw and Sugarcane bagasse at 121 °C for 60 min, resulting in a 29\% and 11\% increase in biogas production, respectively. Switchgrass increased the biogas yield by 25.9\% after being pretreated at 100 °C for six hours [118]. The above study results show heat treatment below 140 °C positively affects the biogas production of LIGB. Table 6 shows that the increase in biogas production caused by LTPT ranged from 3.0\% to 29\%, and treatment temperatures above 100 °C with more than one hour of exposure were recommended.
Given the weak removal ability of hemicellulose or lignin by LTPT with individual hot water, some studies proposed to strengthen the effect of LTPT by adding chemicals, such as using alkali, acid, or peroxide. Cao et al. [121] pretreated the dry sweet sorghum bagasse at 121 °C for 60 min within 2% (w/v) sodium hydroxide solution, resulting in 84.52% lignin removal. At the same time, only using chemical reagents without heating showed weak lignin dissolution. Jin et al. [118] found that alkaline pretreatment (0.5% wt/vol) of switchgrass at 100 °C for six hours can significantly enhance digestion efficiency. Pedersen et al. [122] also proved that alkaline pretreatments at lower temperatures induced high enzymatic glucose and xylose release in wheat straw. As for acids addition, sulfuric acid is the most commonly used. Taherdanak et al. [123] conducted a pretreatment of wheat plant with 1% (v/v) acid at 121 °C for 10–120 min, which destroyed the surface layer of the wheat plant and increased pore size, resulting in 15.2% lignin removal, 91.5% xylan degradation, and an increment of 15.5% in methane yield. Organic acids can also promote thermal pretreatment. Amnuaycheewa et al. [124] pretreated rice straw using three organic acids of acetic acid, citric acid, and oxalic at 100–140 °C for 30–60 min. The obtained biogas production yields reached 314.7, 322.1, and 318.3 mL·g⁻¹ VS, respectively, are higher than untreated case (50.84 mL·g⁻¹ VS) and hydrochloric acid pretreatment (132.8 mL·g⁻¹ VS). They also conducted an FTIR test and proved that the lignin characteristic peaks of 1732 cm⁻¹ [125], 1604 cm⁻¹, and 1510 cm⁻¹ [126] were reduced compared to the untreated sample, indicating that pretreatments removed lignin from the solid residues. Crystalline cellulose characteristic peaks of 1430 cm⁻¹ and 1105 cm⁻¹ [126] were decreased in pretreated solid residuals, which might be the result of the removal of crystalline cellulose and hemicellulose from the biomass.

### Table 6. Biogas production variation of lignocellulosic biomass after LTPT (Temp. ≤ 140).

| Biomass                  | AD Condition | AD Mode          | Pretreatment | Biogas Yield     | CH₄ Yield | Increase | Reference |
|-------------------------|--------------|------------------|--------------|------------------|-----------|----------|-----------|
| **Pennisetum grass**    | Mesophilic   | Batch (33 days)  | Control      | 190 mL·g⁻¹ VS    |           |          | [110]     |
| **Rice straw**          | Mesophilic   | Batch (35 days)  | Control      | 104 L·kg⁻¹ TS    |           |          | [112]     |
|                         |              |                  | 100          | 128 L·kg⁻¹ TS    |           | 22.8%    |           |
|                         |              |                  | 130          | 125 L·kg⁻¹ TS    |           | 19.8%    |           |
| **Rice straw**          | Mesophilic   | Batch (50 days)  | Control      | 298 mL·g⁻¹ TS    |           | 3.0%     | [114]     |
| **Wheat straw**         | Mesophilic   | Batch (45 days)  | Control      | 404 mL·g⁻¹ VS    |           |          | [115]     |
| **Wheat straw** and Sunflower meal (33%) | Mesophilic | Batch (45 days)  | Control      | 340 mL·g⁻¹ VS    |           |          | [116]     |
| **Wheat straw** Sugarcane bagasse | Mesophilic | Batch (30 days)  | 121          | 29%              |           |          | [119]     |
|                         |              |                  | 120          | 370 mL·g⁻¹ VS    |           | 8.8%     |           |
|                         |              |                  | 140          | 390 mL·g⁻¹ VS    |           | 14.7%    |           |
| **Bean straw**          | Mesophilic   | Continuous (HRT 4.5 d) | Control | 142 mL·g⁻¹ COD   | -         | -        | [117]     |
| **Switchgrass**         | Mesophilic   | Batch (1100 h)   | 121          | 145 mL·g⁻¹ COD   | -         | -        | [118]     |
| **Rice straw**          | Mesophilic   | Batch (30 days)  | 80           | 372.5 mL·g⁻¹ VS  |           | 12.4%    | [120]     |
It is concluded that adding chemical reagents can increase biomass solubility during thermal pretreatment, mainly since alkali addition substantially affects lignin removal. However, several studies found that although chemical assistance can promote the solubilization of COD, it is ineffective in improving biogas production. Du et al. [120] reported sole thermal pretreatment, and adding acid, alkaline or alkaline-peroxide can release inhibitors of formic, acetic acids, and phenolic compounds, only the thermal pretreatment at 121 °C for 60 min without chemicals improved methane yield. A study obtained a higher lignin component removal rate and reduced sugar yield in the acid assistance treatment (2% HCl, two hours, 123 °C) of bean straw. Still, the biogas production did not increase significantly comparing the sole thermal pretreatment (1 h, 121 °C) [117].

In conclusion, higher temperature pretreatment is more conducive to dissolving lignocellulosic matter, but it also produces by-product inhibitors. Adding chemical reagents can help to improve the bioavailability of LIGB at a low preheating temperature. The effect of chemical reagents on the decrease of lignocellulosic recalcitrant mainly depends on the temperature and used dosage [127]. Therefore, optimizing preheating parameters is necessary to reduce the dose of chemicals. LTPTs with and without adding chemicals are recommended to improve the bioavailability of lignocellulosic biomass. However, constantly exploring suitable operating conditions is imperative.

7. Energy Balance

The equipment investment and pretreating costs mainly affect whether it can be enlarged and applied. In other words, pretreatment can increase the biogas production of biomass, but it may not bring energy benefits. So the energy feasibility for any biomass or wastewater pretreatment technology is a crucial variable determining its implementation at an industrial scale [128]. Such as, Mendez et al. [104] reported energy input of pretreatment to Chlorella vulgaris at 120 °C for 40 min was higher than the extra energy gain from the increased biogas. Marsolek et al. [103] pretreated Nanochloropsis oculata at 90 °C for 12 h, resulting in a net energy yield of 351 kJ L⁻¹ algal suspension that is 25% lower than the control. Therefore, although biogas production has increased in many pre-treatment cases, the net energy output may be weak.

Net energy production (NEP) can be used to evaluate this energy balance between the energy input and output. It can be roughly calculated by subtracting energy consumed in pretreatment from the increment energy of biogas or methane production. The ratio of energy input to energy output (Eᵢ/Eₒ) or Eₒ/Eᵢ was also considered an energy balance signal [36,129]. Energy balance is often affected by the interaction and variability between raw material characteristics [36], biogas yield [130], pretreatment intensity, performing temperature [128], and HRT [128,131]. Such as, the solid concentration of the pretreated liquid may be a key to the energy balance. Passos et al. [36] reported that 1.17% VS resulted in a negative energy balance, but when the algal feedstock was concentrated to 2% or 3% VS, it results in a neutral or positive energy balance. Carrillo-Reyes et al. [128] reported the longest HRT of 30 d resulted in a positive energy balance, while a short HRT of 15 d showed a negative balance. Sun et al. [131] also proved that mesophilic conditions operated in the longest HRT of 30 d obtained the highest Eₒ/Eᵢ.

Although thermal pretreatment is an available approach used in the pilot- and full-scale implementation [127], it also needs to consume energy. Thus, considering thermal pretreatment’s energy balance is necessary to evaluate its efficiency and benefits [103]. Table 7 summarizes the energy balance results from the previous literature.
Table 7. Energy balance between thermal pretreatment and biogas production.

| Biomass               | Pretreatment Condition | Energy Yield | Energy for Pretreatment | NEY   | Reference |
|-----------------------|------------------------|--------------|-------------------------|-------|-----------|
|                       |                        | Pretreatment | Control                 |       |           |
| Nanochloropsis oculara| 1 h, 90 °C             | 593          | 125                     | -167  | [105] a  |
|                       | 3.5 h, 90 °C           | 641          | 173                     | -120  |           |
|                       | 2 h, 90 °C             | 644          | 176                     | -117  |           |
| Chlorella vulgaris    | 120 °C for 40 min      | 16489        | -                       | 7969  | 6894      | 1075 | [104] b |
| Algal mixture         | 15 °C for 15 h         | -            | -                       | 2.0   | 116       | -0.9 | [36] c  |
| Dewatered pig manure  | 125 °C for 15 min      | -            | -                       | 36    | 116       | -80  | [79] d  |
| Algal                 | 105–165 °C for 30 min  | -            | -                       | 1     | 1–10      | Negative | [129]    |
|                       | 50 °C, 120 °C for 30 min| 12.4         | 0.5                     | 18    | -17.5     |       |           |
|                       | 80 °C, 120 °C for 30 min| 13.6         | 1.7                     | 18    | -16.3     |       |           |
|                       | 120 °C for 30 min      | 14.3         | 2.4                     | 8     | -5.6      |       |           |
| Brewer’s spent grain  | 60 °C, 12 h            | -            | -                       | 0.4   | 1         | Negative | [43] |
| Kitchen waste         | 120 °C, 30 min         | -            | -                       | 9.2   | 8.5       | +0.7  | [59] e  |
| Food waste            | 60 °C, 10 min          | 13.35        | -                       | -     | 46.06     | Negative | [63] |
|                       | 80 °C, 10 min          | 15.15        | -                       | -     | 76.66     | Negative | [63] |
|                       | 100 °C, 10 min         | 18.16        | 12.9                    | -     | 107.46    | Negative | [63] |
| Sewage sludge         | 134 °C, 30 min         | 2.49         | 2.00                    | -     | -         | 0.49  | [65] e  |
|                       | 90 °C, 1 h             | 312.0        | 221                     | 46.6  | 174.64    |       |           |
|                       | 90 °C, 24 h            | 348.2        | 257                     | 82.6  | 174.75    |       |           |
|                       | 90 °C, 36 h            | 484.9        | 394                     | 91.5  | 302.54    |       |           |
|                       | 60 °C, 30 min          | -            | -                       | 322   | 401       | -78   | [67] d  |
|                       | 80 °C, 30 min          | -            | -                       | 645   | 750       | -105  |           |
|                       | 100 °C, 30 min         | -            | -                       | 672   | 1098      | -426  |           |
|                       | 120 °C, 30 min         | -            | -                       | 699   | 1447      | -748  |           |

Note: Energy yield equal to gas volume × calorific value; Increment was calculated as energy yield minus that of control (data not shown); net energy yield was the difference between increment and required energy for pretreatment; The units are not unified for keeping the original data unit. a: Energy unit is KJ; b: energy unit is kWh; c: energy unit is MJ. Other unmarked units are uncertain.

From all the previous research, proper pretreatment methods and conditions can improve the gas production characteristics of biomass anaerobic digestion. However, the increased part of biogas production may not recover the energy input in the pretreatment. Although LTPT looks less promising, some effective measures are recommended to achieve a positive thermal or electrical balance (or revenue). These suggestions are summarized or proposed in this paper as follows:

1. Pretreatment below 100 °C is preferred to be adopted for more feasibility. The pretreating performing conditions of temperature, exposure time [100], material particle size [45], and the chemical catalyst interact with each other. It is necessary to consider process optimization fully.

2. When the pretreated biomass liquid has a low dry matter content, it is necessary to recover the previous heating energy to heat the next batch of fluid using a heat exchange device [133].

3. The residual heat of the CHP can pretreat the feedback and then be followed by biogas production [63, 134]. Moreover, other clean and sustainable energy technology can be used to cover the extra consumed energy in pretreatment, such as solar panels, windmills, or biogas engineering [128].

4. The solid concentration of biomass is crucial to improve the energy balance of biogas production. Budde et al. [135] reported that thermodynamic hydrolysis is feasible for
solid cattle wastes and mixtures of solid and liquid cattle wastes and is not suitable for liquid cattle waste to obtain positive energy yield.

8. Conclusions

Because heat energy is easily produced and utilized, LTPT is considered good feasibility for engineering practice. However, LTPT is greatly affected by biomass types, temperature, exposure time, and solids content in the pretreating liquid. Pretreatments below 75 °C can activate some microorganisms or enzymes that promote hydrolysis, while that above 75 °C may break up the cell wall, promote hydrolysis and solubilization, destroy hydrogen bonds, and eliminate pathogenic or competitive bacteria, etc. Energy balance can evaluate the practical application potential of heat pretreatment and is determined by whether the biogas increment can recover the energy input in pretreatment. However, this review found most studies obtain an increase in biogas production, but the increased energy is difficult to cover the energy consumption in pretreatment. Increasing the solids content in pretreating liquid, prolonging exposure time, reducing solid particle size, and adding chemicals are recommended to improve the energy balance. Finally, although LTPT has been widely studied and lacks some new ideas, it deserves continued attention and research because of its good application potential.

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Abbreviation

| AD    | Anaerobic digestion | LTPM  | Low-temperature pretreatment |
|-------|---------------------|-------|-------------------------------|
| CHP   | Combined heat and power | MSW | Municipal Solid Waste |
| CM    | Cattle manure       | NEP   | Net energy production |
| CHM   | Chicken manure      | ODS   | Organic dry sludge |
| COD   | Chemical oxygen demand | OFMSW | Organic fraction of MSW |
| DM    | Dairy manure        | OLR   | Organic loading rat |
| FW    | Food waste          | PM    | Pig manure |
| GHG   | Greenhouse gases    | SM    | Swine manure |
| HP    | Heat pretreatment   | TCOD  | Total COD |
| HRT   | Hydraulic retention time | TS | Total solids |
| HTPT  | High temperature pretreatment | VFA | Volatile fatty acid |
| KW    | Kitchen waste       | VS    | Volatile solids |
| LIGB  | Lignocellulosic biomass | WAS | Waste activated sludge |

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