Thermally enhanced solubilization and anaerobic digestion of organic fraction of municipal solid waste

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Abstract

Organic fraction of municipal solid waste (OFMSW) is an ideal substrate for biogas production; however, complex chemical structure and being heterogeneous obstruct its biotransformation in anaerobic digestion (AD) process. Thermal pre-treatment of OFMSW has been suggested to enhance the solubilization and improve the anaerobic digestibility of OFMSW. This paper critically and comprehensively reviews the characterization of OFMSW (physical, chemical, bromatological) and enlightens the valuable properties of OFMSW for waste valorization. In following sections, the advantages and limitations of AD of OFMSW are discussed, followed by the application of temperature phased AD, and various thermal pre-treatments, i.e., conventional thermal, microwave, and thermo-chemical for high rate bioenergy transformation. Effects of pre-treatment on COD, proteins, sugars and VS solubilization, and biogas yield are discussed. Formation of recalcitrant during thermal pre-treatment and the effect on anaerobic digestibility are considered. Full scale application, and techno-economic and environmental feasibility of thermal pre-treatment methods are also revealed. This review concluded that thermophilic (55°C) and temperature phased anaerobic digestion, TPAD (55+37°C) processes shows effective and stable performance at low HRTs and high OLRs and achieved higher methane yield than mesophilic digestion. The thermal pre-treatment at a lower temperature (120 ºC) improves the net energy yield. However, high-temperature pre-treatment (>150°C) result in decreased biogas yield and even lower than the non-pre-treated OFMSW, although a high degree of COD solubilization. The OFMSW solubilization in terms of COD, proteins, and sugars cannot accurately reflect thermal/hybrid pre-treatments' potential. Thus, substrate pre-treatment followed by anaerobic digestibility of pretreated substrate together can evaluate the actual effectiveness of thermal pre-treatment of OFMSW.

Keywords: Anaerobic digestion, Organic fraction of municipal solid waste, Thermal pre-treatment, Substrate solubilization, Biogas yield.

Contents

1. Introduction

Undoubtedly, one of the most important commitments on sustainable energy planning is promoting biogas production from organic-wastes to satisfy future energy requirements of our society and to achieve effective waste management. As a sustainable and renewable energy source with high-energy content, biogas is a promising alternative to fossil fuels. The application of anaerobic digestion utilizing organic wastes has increased in appeal from a policy-making standpoint as it is now considered a reliable technology (Cecchi et al., 2011). The organic fraction of municipal solid waste (OFMSW) would seem to be an ideal substrate for biogas production; however, the opportunities are missed to maximize the recovery of biogas production from the facilities due to the presence of complex organic materials in OFMSW and the heterogeneous nature of waste that obstruct the biotransformation of the substrate, i.e., rate-limiting step of the process. The organic fraction of municipal solid waste is rich in hardly degradable substances such as lignocellulose and fatty fractions, which is a barrier to the biological process of degradation. The complex structure of MSW does not provide easy access for biodegradable organics in bioreactors. Such limited accessibility causes significantly lower biogas yields. Therefore, research has focused on various pre-treatment technologies utilizing mechanical, chemical, thermal, and biological methods or combinations to solubilize the complex organics and increase the surface area and accessibility for better enzymatic hydrolysis/microbial degradation (Carrere et al., 2010; Tyagi and Lo, 2011).

The pre-treatment of the OFMSW can be an interesting option to achieve high organic matter solubilization, increase in acidogenic and methanogenic biodegradability in single or multi-stage processes, and subsequent improvement in biogas production. Pre-treatment aims to modify the complex lignocellulose structure to simpler forms by weakening the molecular bond between lignin and carbohydrate by increasing the substrate's surface area such that the degradation and the biogas generation process are simplified (Tyagi et al., 2018). Earlier studies reported that thermal, mechanical, chemical, and thermo-chemical pre-treatment systems efficiently improved digestion efficiency and biogas production (Carrere et al., 2010; Tyagi and Lo, 2011; Tyagi et al., 2018). Among the pre-treatment technologies studied, thermal pre-treatment of organic wastes at a wide range of temperatures (55-200ºC) has garnered consideration for the production of biogas (methane, hydrogen) and value-added products (bioethanol) from organic wastes. Thermal pre-treatment, through which a higher hemicellulosic fraction is removed, improves the accessibility of the enzyme to cellulose (Mosier et al., 2005; Pérez et al., 2008). Thermal pre-treatment alters the structure of the insoluble fraction to make it more amenable to biodegradability (del Rio et al., 2011). The soluble chemical oxygen demand (sCOD) increases significantly because of the degradation and dissolution of insoluble organic compounds such as carbohydrates, lipids and protein (Liu et al., 2012; Carrere et al., 2010; Ariunbaatar et al., 2014a, Yeo et al., 2019). The thermal pre-treatment is considered an environmentally-friendly process due to not using any chemicals and zero emissions. Integrating thermal pre-treatment with anaerobic digestion of OFMSW could have several potentially positive outcomes for sustainable biofuel production: increased stability of the process; increased specific biogas yields and, CH_4 content of biogas produced; maximizing the substrate availability for the microbial community; reduction in energy requirements during the digestion process; reduction in the hydraulic retention times (HRT); reduced total volume of the reactor can provide economic feasibility; reduced use of landfills; and utilization of bio-solids (digestate) as fertilizer.

The key objective of this paper is to critically and comprehensively review the application and feasibility of various thermal pre-treatments, i.e., conventional thermal, microwave, and thermo-chemical and temperature phased AD, for high rate bioenergy transformation. The effects of thermal pre-treatments on COD, proteins, sugars and VS solubilization, and biogas yield are discussed. The formation of recalcitrant during thermal pre-treatment and the effect on anaerobic digestibility are taken into account.

2. OFMSW characterization

The anaerobic digestion process's kinetics and energy efficiency are strongly influenced by the waste composition (Fisgativa et al., 2016). For example, waste with a high-fat content (such as food waste) negatively affects the process's kinetics (Suwannarat and Ritchie, 2015). Therefore, it is imperative to know the composition and physical-chemical characteristics of substrate to obtain good energy yields through biological processes and good quality digestate for fertilizers usage (Al Seadi and Lukehurst, 2012). The particle size also has a significant influence on this type of biological process. In fact, biogas production is slower for larger particle sizes (Zhang and Banks, 2013). Rheology is another physical parameter related to the degree of mixing of the wastes within anaerobic digesters. Wu (2012) reported that during the co-digestion of OFMSW with manure, the mixture (with solids contents around 2.5%) presented a non-Newtonian pseudoplastic fluid behavior. Finally, density is another frequently used parameter to characterize the behavior of bio-methanation processes. The density of OFMSW can range from 328 to 1052 kg/m^3. Generally, fewer unwanted substances and greater biodegradability are reported for waste with high-density values (Forster-Carneiro et al., 2008).

In order to estimate the methane potential of OFMSW or determine the viability of nutrient recovery (C, N, P), it is essential to carry out elemental analysis of the waste (Buffiere et al., 2006). In this sense, four types of OFMSW from different countries were characterized in the VALORGAS Project (VALORGAS, 2010), determining that the fraction distribution depends on each region's eating habits. However, the types of OFMSW analyzed were similar from the chemical point of view and their energy content, which is explained, taking into account that humans' energy requirements do not vary significantly from one country to another. For example, in all the samples, over 50% of the organic content is represented by fruits and vegetables (Alibardi and Cossu, 2015). Campuzano and González-Martínez (2016) analyzed the main characteristics of the OFMSW from 22 countries and 43 cities (refer Table

1). It should be noted that methane productivity not only depends on the characteristics of the OFMSW but also on the mode and operating conditions of the bio-methanation process (continuous/ semi-continuous; mesophilic/thermophilic; wet/dry digestion; etc.). Generically, methane productivity increases for a higher volatile solids (VS)/total solids (TS) ratio, i.e., between 300 and 600 NL/kgVS for a VS/TS ratio between 75 and 95%.

Table 1. Values of the main chemical and bromatological parameters and elementary composition of the OFMSW (Campuzano and González-Martínez, 2016)

Parameters	**Range**	**Average**
pH (units)	3.9 - 7.9	5.2 ± 0.95
Total solids (%)	15.0 - 50.2	27.2 ± 7.6
Volatile solids (%)	7.4 - 36.1	22.9 ± 6.3
Total phosphorus (g/kg)	0.4 - 13	1.7 ± 2.5
Kjeldahl nitrogen (g/kg)	1.0 - 28	7.9 ± 5.4
Methane production (NL/kgVS)	61 - 580	415 ± 138
COD (g/kg)	140-575	331.5 ± 121.4
Elementary composition (% of TS)		
Carbon	37.6 - 51.3	46.6 ± 4.4
Hydrogen	5.6-7.5	6.6 ± 0.62
Nitrogen	1.5-3.8	2.9 ± 0.6
Sulfur	0.1-0.9	0.3 ± 0.26
Bromatological analysis of OFMSW		
Fraction (% of VS)	**Range**	**Average**
Fat, oil and grease (FOG)	6.09-35	17.5 ± 6.6
Protein	7.7-30	17.7 ± 5.5

Raw fibre	13.6-71.9	29.2 ± 15.0
Lignin	5.2-18.5	9.7 ± 5.3
Carbohydrates	35-63.2	55.5 ± 10.1
▪ Cellulose	5-51.9	18.6 ± 15.0
▪ Hemi-Cellulose	2.9-14.6	8.6 ± 4.6
▪ Starch	13.8-20.7	17.1 ± 2.5
▪ Free sugars	5.9-22	10.5± 6.0

OFMSW is chiefly composed of food waste; thus, its bromatological composition can be described from the point of view of its carbohydrate, protein, and fat, and oil content. Based on this, biogas' potential is determined mainly by the biodegradability of the waste and its content in macromolecules such as lignocellulose, hemicellulose, and cellulose (Buffiere et al., 2006). A bromatological analysis of OFMSW was reported by Campuzano and González-Martínez (2016), taking into account 22 cities in 11 countries (Table 1). This analysis presents values for fat, oil, and grease (FOG), protein, raw fibers (lignin, cellulose, hemicellulose), and carbohydrates (cellulose, hemicellulose, starch, and free sugars). All these molecules constitute 100% of VS. According to Sanders (2001), raw fiber, soluble and non-soluble carbohydrates (cellulose, hemicellulose, and pectin), and starch are the main components. Lignin determines the degree of anaerobic biodegradability of substrates. According to Xu et al. (2014), high lignocellulosic fibre contents have adverse effects on biogas productivity since lignin cannot be hydrolyzed under anaerobic conditions. The_proteins have nitrogen and sulfur in their composition. According to Straka et al. (2007), the sulfur contained in the proteins can lead to the generation of hydrogen sulfide and free ammonia (harmful to methanogenic archaea) in the biogas during the bio-methanation process. The fats, oil and grease (FOG) fractions are mainly made up of triglycerides containing glycerol and long-chain fatty acids (LCFAs). FOGs are readily hydrolyzed to LCFAs, acetate, and hydrogen (Alves et al., 2001). These compounds are an ideal substrate for a bio-methanation process since they present a high methane yield (Li et al., 2011).

3. Anaerobic digestion of OFMSW

Anaerobic digestion (AD) is a matured and well-established technology (Mata-Alvarez et al., 2000; Ponsá et al., 2011), and is currently viewed as the most feasible technology for biogas production from the OFMSW (Davidsson et al., 2007; Franca and Bassin, 2020; Kumar and Samadder, 2020; Tyagi et al., 2018; Zamri et al., 2021). AD systems entail the occurrence of four stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis, and consist in the microbial degradation of biodegradable organic material and its conversion into biogas (rich in methane) and a digestate (rich in nutrients) (Aboudi et al., 2015; Tyagi et al., 2021). The OFMSW obtained from MSW segregation (or source selection), although varies in characteristics depending on its origin, generally has similar biochemical properties in terms of carbohydrates, proteins, fats, minerals, etc., which give it a high biochemical methane potential (BMP) (Cabbai et al., 2013; Davidsson et al., 2007; Neves et al., 2008). Nevertheless, BMP obtained from OFMSW sourced from canteens and restaurants (mainly food wastes) had shown the highest methane yields (Banks et al., 2011; Fisgativa et al., 2016) over MSW segregated OFMSW. The bromatological characteristics of OFMSW are an important factor that affects the biogas yield and overall process performance (Vavilin et al., 2004, 2008; Zamri et al., 2021). The OFMSW with high lipids proportions had demonstrated slow biodegradation kinetics, increased oxygen demand, and risks of pipeline blockage and inhibition of the AD process, mainly due to LCFAs accumulation (Cirne et al., 2007; Liu et al., 2012; Neves et al., 2008). The OFMSW, with a high content of lignocellulose fraction, mainly originated from paper and cardboard wastes has shown that lignocellulose's recalcitrant structure hinders the hydrolysis of OFMSW (Mahmoodi et al., 2018; Yuan et al., 2014). AD of OFMSW having high protein proportions (meat and bone, fish and fishbone, animal wastes, etc.) might be underperforming due to high ammonia release and VFAs accumulation (Tyagi et al., 2018).

Anaerobic digestion of OFMSW could be carried out mainly at dry (>20 % TS), or semi-dry (10-20% TS) conditions and only a few studies were on wet AD (<10 % TS) due to the high solids content of OFMSW (Bolzonella et al., 2003; Forster-Carneiro et al., 2008, 2007). Dry AD is applied for solid organic wastes such as OFMSW and present several challenging issues due to the complexity of the operation, in comparison to wet and semi-dry conditions (Bolzonella et al., 2003; Forster-Carneiro et al., 2007; Franca and Bassin, 2020; Kothari et al., 2014; Rocamora et al., 2020), mainly for large scale applications. However, the dry AD process under a thermophilic regime has shown advantages such as the faster degradation of organics, higher pathogens removal, and enhanced methane yield concerning mesophilic and wet AD conditions (Fdez.-Güelfo et al., 2011). Semi-dry conditions have been suitable for the mesophilic AD of OFMSW with high methane generation and high OLR (Bolzonella et al., 2003). At industrial and commercial levels, the main technologies for dry AD systems are Valorga, Dranco, Kompogas, laran, Bekon, SEBAC, and Biocel, while BTA, VAGRON, AVECON are the most used technologies for wet systems (Fdéz.-Güelfo et al., 2010; Franca and Bassin, 2020; Rocamora et al., 2020; Zamri et al., 2021). The inoculum source and adaptation strategy are also important when designing an AD process for OFMSW (Forster-Carneiro et al., 2007; Rocamora et al., 2020). Forster-Carneiro et al. (2007) studied the effect of six different inoculums sources on the dry thermophilic AD of OFMSW. They found that digested sludge was the best inoculum compared to inoculums from animal (swine and cattle manures) or vegetal origins (corn silage). The highest organic matter removals were obtained when using sludge alone or in a mixture with swine manure. Moreover, shorter lagphase and high biogas production were achieved in the reactors operating with digested sludge as inoculum. The inoculum to substrate ratio (ISR) is an essential criteria in the AD of OFMSW. The ISR should be higher than 1 in terms of VS, as lower ratios can inhibit methanogenesis due to VFAs accumulation. The optimum inoculum to substrate ratio also depends on the inoculum source (Angelidaki et al., 2009; Jensen et al., 2009).

OFMSW is a complex waste stream and may not be fully degraded through the AD process. The hydrolysis stage in AD of OFMSW is considered the limiting step (Park et al., 2005). In this context, physical, chemical, and mechanical pre-treatment of OFMSW have been widely reported as a suitable practice for enhancing substrate solubilization and subsequent biogas generation and volatile solids (VS) reduction (Fdez.-Güelfo et al., 2011; Zamri et al., 2021; Tyagi et al., 2018). Nevertheless, the pre-treatment type and conditions should be carefully selected to avoid excessive hydrolysis generating intermediate inhibitory compounds (Kumar and Samadder, 2020; Labatut et al., 2011), mainly in the chemical pre-treatment of OFMSW (Panigrahi and Dubey, 2019). Among the methods studied, temperature phased AD, and thermal pre-treatment showed promising outcomes in higher substrate solubilization and resulting biogas yield and improved AD performance (Tyagi et al., 2018). Nevertheless, thermal pre-treatment conditions should be well designed and studied to avoid the high costs of the energy input or intermediary products mediated process inhibition (Cesaro and Belgiorno, 2014; Kavitha et al., 2017). Besides the characteristics of the waste, thermal pre-treatment variables such as the temperature used, the time of pre-treatment, and the heat type applied are essential to identify the best strategy to enhance AD performance and make the process economical.

4. Anaerobic digestion of OFMSW under variable temperature regimes

The anaerobic microbiome has significant differences in terms of growth rates and sensitivity to environmental conditions under different sets of conditions, e.g., temperature, which can have great consequences on the process by affecting the metabolic activity of the microorganisms of each group differently. In general, three temperature ranges (optimum) for the growth of anaerobes can be distinguished: psychrophilic (15ºC), mesophilic (35 ºC), and thermophilic, 45 ºC (Batstone et al., 2002; Chernicharo, 2007). In the case of the psychrophilic range, psychrotrophic and psychrotolerant microorganisms are considered (Wiley et al., 2011), which are capable of growing at low temperatures (<5ºC) with optimum growth temperature ranges between 20 to 30 ºC (Canganella and Wiegel, 2011). In the thermophilic range, microorganisms capable of growing in the range of 70 - 80 ºC are called extreme-thermophilic, while those that grow at temperatures above 80ºC are called hyper-thermophilic (Canganella and Wiegel, 2011).

For a given microorganism, the evolution of the specific growth rate with temperature in any of the previously mentioned ranges follows a similar trend. The growth rate around the minimum temperature is low but grows exponentially when the temperature increases until reaching the optimal temperature. From this optimum temperature, small increases in temperature cause a significant decrease in growth rate (Henze and Harremoes, 1983). The observed evolution has been proposed to correspond to a difference of two exponential functions (Eq. 1). These functions represent the increase in metabolic activity with temperature and the rate of cell decay or death due to the denaturation of cellular enzymes and proteins. Both expressions can be expressed by an Arrhenius-type expression (Chernicharo, 2007):

$$\mu = k_1\, e^{-\left(\frac{E_1}{R \cdot T}\right)} - k_2\, e^{-\left(\frac{E_2}{R \cdot T}\right)} \qquad \text{(Eq. 1)}$$

Where:

μ = specific growth rate of microorganisms (day^{-1})

k_1 microorganisms synthesis rate constant (day^{-1})

k_2 = microorganisms decay rate constant (day^{-1})

E_1 = activation energy for microbial synthesis processes ($J \cdot mol^{-1}$)

E_2 = activation energy for microbial decay processes ($J \cdot mol^{-1}$)

R = gas constant = 8.31 ($J \cdot K^{-1} \cdot mol^{-1}$)

T = absolute temperature (K)

In general, the values of E2 are higher than those of E1, which causes the growth rate curves against temperature to be asymmetric. That small increases above the optimal temperature cause a drastic decrease in viscosity. When the different ranges of temperature are compared, the maximum values of the microorganisms' specific growth rate correspond to the other ranges developed in the sense of their characteristic temperatures (van Lier et al., 1997). However, when working with mixed microbial cultures, as in the case of anaerobic digestion of OFMSW, the observed behaviour may differ. It is a consequence of temperature's effect on the selection of microorganisms that will grow in the system. Hashimoto et al. (1981) and Chen (1983) have proposed a linear relationship between the maximum specific growth rate of microorganisms and the operating temperature (Eq.2). The equation was obtained by fitting the experimental data from multiple studies. Furthermore, the equation is developed to be applied in the range of 30 - 60 ºC, which encompasses two different temperature ranges: mesophilic and thermophilic.

$$c\mu_{max}\ (day^{-1}) = 0.013 \cdot T(°C) - 0.129 \qquad \text{(Eq. 2)}$$

However, de la Rubia et al. (2005) have determined that temperatures around 45 ºC are not suitable for anaerobic digestion. They proposed a protocol to transition from the mesophilic to thermophilic process using a mixed microorganism’s culture. The protocol consists of a gradual increase in temperature but avoiding operation around 45ºC. Since, the cessation of the activity of methanogenic anaerobic microorganisms occurs at 45ºC. The authors proposed to avoid AD operation in the range of 43 ºC - 50 ºC and performing a direct transition between these two temperatures.

The mesophilic and thermophilic processes are the most widely used processes at industrial scale; however, the psychrophilic processes are excessively low in application. The thermophilic processes are considered less robust than mesophilic since temperature variations may lead to process imbalance and risk of VFAs accumulation and ammonia inhibition. On the other hand, thermophilic processes achieve higher substrate hydrolysis rates and pathogen elimination resulting in higher biogas production and volatile solids removal and generation of pathogens-free digestate, which can be used as fertilizer. The increase in temperature affects various physical-chemical aspects of the medium: It reduces the solubility of gases, especially gases with an inhibitory character such as ammonia and hydrogen sulphide. It also decreases the medium's viscosity, which reduces the energy requirements necessary for the agitation of the medium. However, high-temperature operation under a thermophilic regime requires higher energy costs for temperature maintenance. Since temperature affects the hydrolysis rate more significantly, an increase in operating temperature leads to a marked increase in the rates of hydrolysis and acidogenesis (Mata-Alvarez, 2003; Zhang et al., 2009). The above-given fact is crucial for the digestion of lignocellulosic or hardly degradable biomasses, where operation at a high temperature is preferred (Yang et al., 2015). Thus, the extreme-thermophilic and hyper-thermophilic processes have aroused much interest in recent times. Lee et al. (2008) studied the microbial diversity in hyper-thermophilic reactors fed with artificial kitchen waste. They reported that methanogenic microorganisms were predominant when working at temperatures below 65 ºC, while acidogenic microorganisms showed dominance at temperatures above 73 ºC. The increase in the hydrolysis rate along with the temperature is the basis of the temperature phased anaerobic digestion (TPAD) process, where the first stage is thermophilic or hyper-thermophilic digestion followed by mesophilic digestion in the second stage.

The anaerobic digestion of OFMSW is widely used in Europe. According to De Baere and Mattheeuws (2012), most of the full-scale systems (67%) operated under the mesophilic regime. However, the rest being operated under a thermophilic regime. Likewise, maximum numbers of the plants correspond to the digestion of high solid content (also referred to as dry digestion or solid-state digestion), i.e., representing 62% of the total. However, the start-up of dry anaerobic digestion shows complications under the mesophilic regime. Thus, thermophilic digestion is a preferable option (Li et al., 2011). In the thermophilic process, the hydrolysis of lignocellulosic fraction (cellulose) has been enhanced by five to six times higher than the mesophilic digestion (Chatterjee and Mazumdar, 2019). Digestion under thermophilic conditions causes a considerable increase in the process rate, allowing the use of lower HRT (Hartmann and Ahring, 2006). The rise in energy demand to maintain the thermophilic temperature can be compensated by the excess methane produced in the thermophilic process and the increase in the process rate (De Baere, 2000). Thus, thermophilic digestion is considered a better option for dry anaerobic digestion of OFMSW (Kim et al., 2006; Fernández-Rodríguez et al., 2013; Rocamora et al., 2020; Basinas et al., 2021). Jiang et al. (2020) have studied the anaerobic digestion of OFMSW, operating in the semi-continuous mode under mesophilic and thermophilic conditions. The authors compared the results obtained for different OLRs between 0.75 and 11.00 g VS/ L.d. The best condition corresponds to an OLR of 7.50 g VS/ L.d operating under thermophilic conditions. Based on these data and considering the annual generation of OFMSW in China, the authors (Jiang et al., 2020) estimated net output energy could be 69 970.61 GWh per year.

Table 2 summarizes different studies on mesophilic (M), thermophilic (T) and TPAD processes applied to the treatment of OFMSW. Amodeo et al. (2021) studied TPAD of

OFMSW and digested sludge under variable temperature regimes of 37ºC, 55ºC, and 65ºC, and reported the effects on the overall process performance and biogas yield. The better hydrolysis performances were obtained at 55 ºC. The thermophilic digestion achieved higher volatile solids reductions and destruction of coliforms. With the TPAD (thermophilic-mesophilic) system, the advantages of both processes can be achieved simultaneously. Thus, combined process resulted in better specific methane production, effluent quality, and process stability in mesophilic process and higher rate of hydrolysis and VS destruction, and pathogens removal in the thermophilic process. Borowski (2015) reported that the TPAD process's operation is strongly dependent on the conditions used in the first thermophilic stage. In this study, one and two-day hydraulic retention times (HRTs) were tested in the first stage thermophilic reactor, and the best results were obtained for 1-day HRT. The operation at 2-day HRT led to a high concentration of ammonium and VFAs (propionic acid being predominant) in the thermophilic effluent, causing inhibition in the subsequent mesophilic digester. Fernández-Rodríguez et al. (2012; 2014; 2016) have studied the dry semi-continuous anaerobic digestion (20% TS) of OFMSW under mesophilic, thermophilic and TPAD conditions. Mesophilic operation were performed for HRTs of 30, 20, and 15 days (OLRs from 2.42 to 4.09 g-VS/L.d). Thermophilic tests were performed for HRTs of 15, 10, 8, 6, 5, 4, and 3 days (OLRs from 4.8 to 20.0 g-VS/L.d). Finally, two TPAD configurations were tested where first unit was operated under thermophilic range (T) and second under mesophilic range (M), using the following HRTs: 4T + 10M and 3T + 6M (OLRs of 6.40 and 9.96 g-VS/L.d, respectively). The authors compared the three processes operating at similar HRTs: (a) mesophilic at 15 days, thermophilic at 15 days and TPAD 4T + 10M, and (b) thermophilic at 10 days and TPAD 3T + 6M. The findings revealed that the TPAD process was viable in both configurations and that higher efficiencies for organic matter and VS removals and higher methane yield were obtained in the TPAD process over mesophilic or thermophilic processes. The 4T + 10M configuration was better than the 3T + 6M concerning the specific methane production (35-45% higher) and the organic matter removal efficiency (6-19% higher).

Table 2 shows that mesophilic, thermophilic, and temperature phased AD of OFMSW achieved the average methane yield of 278 and ≈350 mL CH_4/g-VS_{added}, and maximum methane yield 370 and ≈550 mL CH_4/g-VS_{added}, respectively. An overall comparison of the different processes shows that thermophilic and temperature phased anaerobic digestion offers the best conditions for the treatment of OFMSW. Both processes show effective and stable performance at low HRTs and high OLRs and achieved higher methane yield than mesophilic digestion. Nevertheless, all these processes were encountered a typical VS removal of ≤60%, high HRT (25-30 days), and low OLR of < 7.0 kg VS/ m^3. d. The average process performance of the above-discussed processes is due to the complex characteristics of OFMSW, such as, presence of lignocellulose and fatty fractions, which is a barrier to the biological process of degradation and obstructs the easy access to biodegradable organics in bioreactors. Such limited accessibility leads to reduced substrate hydrolysis followed by lower biogas production and VS removal. The substrate pre-treatment has shown the potential to solubilize the complex organics, increase the surface area and accessibility for better hydrolysis, and improve overall AD process performance. Earlier studies reported that thermal, mechanical, chemical, and thermo-chemical pre-treatment methods efficiently improved anaerobic digestion efficiency and biogas production (Carrere et al., 2010; Tyagi and Lo, 2011; Tyagi et al., 2018). Among them, thermal pre-treatment (conventional, microwave, and hybrid thermo-chemical) has received attention to significantly enhancing the substrate solubilization rate and improving the hydrolysis and biogas yield from organic wastes.

Table 2. Performance comparison of mesophilic, thermophilic and temperature phased anaerobic digestion processes

Process type	**Substrate**	**Operating conditions**	**Methane Yield**	**% VS removal**	**Reference**
Mesophilic	OFMSW+ WAS	37 °C, OLR: 1.60 kg VS/m^3.d	340 mL CH_4 /g-VS_{added}		Cavinato et al., 2013
	OFMSW+ WAS	35 °C, 4.2% TS	376 mL CH_4/ kg VS_{added}	61	Ara et al., 2015
	Hydromechanically separated OFMSW+ Sewage Sludge	35 ºC, HRT: 15 days OLR: 2.85 kg-VS/ m^3.d	230 mL CH_4/g-VS_{added}	37.23	Borowski, 2015
	OFMSW	35 ºC, HRT: 27 days OLR: 7.5 kg-VS/ m^3.d	278 mL/g-VS_{added}		Jiang et al., 2020
		35 ºC, HRT: 25 days OLR: 9.0 kg-VS/ m^3.d	250 mL/g-VS_{added}		
	OFMSW	35 ºC, HRT 20 days 20 % TS; OLR 2.95 kg-VS/ m^3.d	360 mL CH_4/g-VS_{added}		Fernández-Rodríguez et al. 2012; 2014;2016
		35 ºC; HRT 15 days 20 % TS; OLR 4.09 kg-VS/ m^3.d	242 mL CH_4/g-VS_{added}		
	OFMSW	40 ºC, HRT 122 days 23 %wt TS; OLR 4.22 kg-VS/ m^3.d	148 mL CH4/g-VS_{added}		Basinas et al., 2021

	FW	37 ºC, HRT: 30 days	477 mL CH4/g VS_{added}	83.22	Xiao et al., 2018
Thermophilic	OFMSW +WAS	55 °C, OLR: 2.21 kg VS/m^3.d	570 mL CH_4 /g VS_{added}		Cavinato et al., 2013
	OFMSW	55 ºC, HRT: 27 days OLR: 7.5 kg-VS/ m^3.d	302 mL/g-VS_{added}		Jiang et al., 2020
		55 ºC, HRT: 25 days OLR: 9.0 kg-VS/ m^3.d	273 mL/g-VS_{added}		
	OFMSW	55 ºC, HRT 15 days 20 % TS; OLR 4.8 kg-VS/ m^3.d	302 mL CH_4/g-VS_{added}		Fernández-Rodríguez et al., 2012, 2014, 2016
		55 ºC; HRT 10 days 20 % TS; OLR 5.9 kg-VS/ m^3.d	342 mL CH_4/g-VS_{added}		
		55 ºC; HRT 5 days, 20 % TS; OLR 13.0 kg-VS/ m^3.d	322 mL CH_4/g-VS_{added}		
	OFMSW	55 ºC, HRT 138 days, 24.7 %wt TS, OLR 2.26 kg-VS/ m^3.d	176 mL CH_4/g-VS_{added}		Basinas et al., 2021
	FW	55ºC, HRT: 30 days;	461 mL CH_4/g VS_{added}	81.68	Xiao et al., 2018
Temperature Phased Anaerobic Digestion (TPAD)	Hydromechanically separated OFMSW+ Sewage Sludge	55 ºC + 35 ºC, HRT: 1 + 14 days	333 mL CH_4/g VS_{added}	52.10	Borowski, 2015
		55 ºC + 35 ºC	267 mL CH_4/g VS_{added}	44.89	

	HRT: 1 + 9 days			
OFMSW+ Sewage sludge	Acidogenic reactor-70°C Methanogenic reactor-55°C, OLR: 1.01 kg VS/m^3.d	350 mLCH$_4$/ g VS$_{added}$	49.6	Lee et al., 2009
OFMSW	55-57 º C + 35-37 ºC, HRT: 4 days + 10 days, 20 % TS, OLR: 6.40 kg-VS/ m^3.d	339 mL CH$_4$/g-VS$_{added}$		Fernández-Rodríguez et al. 2012, 2014,2016
	55-57 º C + 35-37 ºC HRT: 3 days + 6 days 20 % TS, OLR: 9.96 kg-VS/ m^3.d	246 mL CH$_4$/gVS$_{added}$		
OFMSW (FW) + Sewage Sludge	55ºC + 35ºC, 10 % TS; OLR: 6 kg-VS/ m^3.d	200 mL CH$_4$/g-VS$_{added}$	44.2	Kim et al., 2011
OFMSW (FW) + paper waste	55 ºC + 35 ºC, With recirculation HRT: 6 + 24 days	1.67 L CH$_4$/L.d	86.2	Li et al., 2020
	55 ºC + 35 ºC, Without recirculation, HRT: 6 + 24 days	1.91 L CH$_4$/L.d	81.58	
FW	55 ºC + 37 ºC, HRT: 3 + 17 days	0.47 L CH$_4$/Lr/d	75.2	Gabi et al., 2017
	55 ºC + 37 ºC, HRT: 3 + 10 days	0.87 L CH$_4$/L.d	73.9	

OFMSW (FW)	55ºC + 37.5 ºC, HRT: 6 + 24 days	447 mL CH_4/g VS_{added}		Qin et al., 2018
OFMSW	55ºC + 37.5 ºC, HRT: 1.3 + 5 days OLR: 7.7 kg-VS/ m^3.d	464 mL CH_4/g VS_{added}	95.7	Chu et al., 2008
FW	55ºC + 35 ºC HRT: 5 + 9 days OLR: 5.7 kg-VS/ m^3.d	510 mL CH_4/g VS_{added}	80	Algapani et al., 2019
FW	55ºC + 37 ºC, HRT: 6 + 24 days	454 mL CH_4/g VS_{added}	78.55	Xiao et al., 2018
OFMSW (FW) + SS	55ºC + 35 ºC, HRT: 10 days OLR: 2.7 kg-VS/ m^3.d	280 mL CH_4/g VS_{added}	61.3	Kim et al., 2004
	35ºC + 35 ºC, HRT: 10 days; OLR: 2.7 kg-VS/ m^3.d	190 mL CH_4/g VS_{added}	40.1	
OFMSW + Primary Sludge	55ºC + 35 ºC, HRT: 15 days OLR: 2.4 kg-VS/ m^3.d	418 mL CH_4/g VS_{added}	69.8	Scmit and Ellis, 2001

WAS: waste activated sludge; OFMSW: organic fraction of municipal solid waste; HRT: hydraulic retention time; OLR: organic loading rate; TS: total solids concentration; L: reactor volume (L); VS: volatile solids concentration

5. Thermal pre-treatment of OFMSW

5.1 Conventional heating

Conventional heating is one of the simplest forms of thermal pre-treatment of OFMSW, where a sealed reactor like an autoclave or a thermal-pressure vessel (with/without a mixer) is used to provide thermal energy to the substrate. Figure 1 depicts the effect of conventional heating on the breakage of lignocellulosic bonds in the OFMSW and subsequent solubilization of recalcitrant organic matter. Table 3 summarizes the findings of various studies conducted on thermal pre-treatment of OFMSW. The complex lignocellulosic structure of OFMSW is characterized by the enclosure of cellulose and hemicellulose in lignin, restricting the enzymatic effects on the substrate particles and limiting the hydrolysis of OFMSW. Thus, breakage of this complex structure of OFMSW is necessary to enhance the hydrolysis and methane yield during anaerobic digestion. As OFMSW is treated under high pressure and high temperature using steam, the cell wall gets ruptured, leading to cleavage of the bond between the lignin, cellulose, and hemicellulose (disintegration of the particle structure), followed by dissolution of chemical oxygen demand (COD), proteins, carbohydrates, humic acids, lignin, cellulose, and hemicellulose. It enhances the disintegration of the organic particulate matter and solubilizes the biomass within the temperature ranges from 50-270ºC (Carrere et al., 2008). A temperature range of 110-180ºC and a reaction time of 20-60 min were suggested as an effective temperature-time combinations for conventional thermal pre-treatment (Lu et al., 2008).

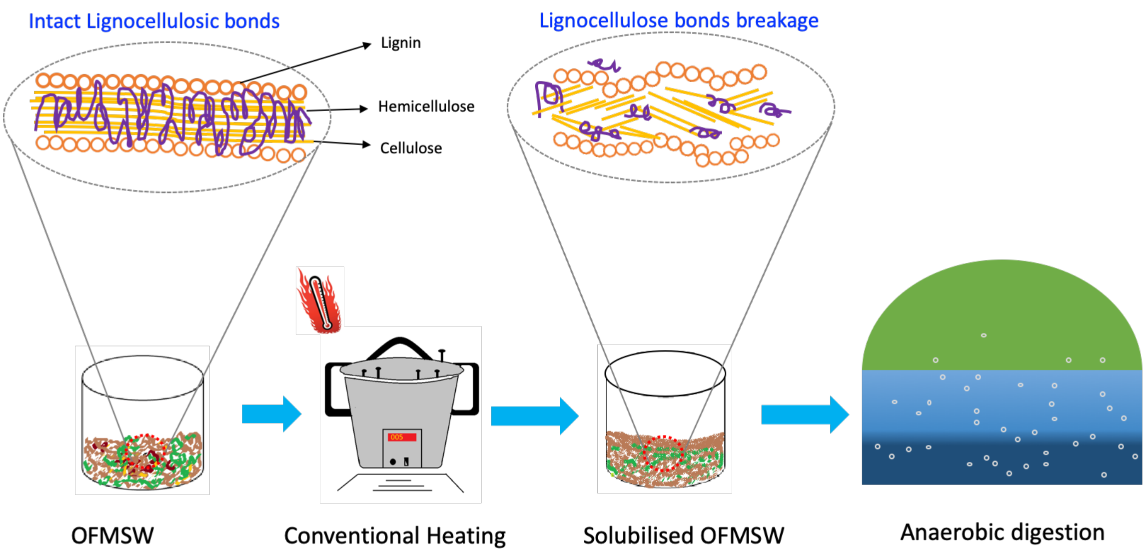

Fig. 1. Conventional thermal pre-treatment of OFMSW

The pre-treatment temperature and reaction time have a significant effect on substrate solubilization and overall process performance. The effectiveness of thermal pre-treatment has been evaluated for enhancement in substrate solubilization (COD, proteins, carbohydrate, and lignocellulosic fraction) and AD process performance (biogas yield and VS removal). The effect of thermal pre-treatment has been studied at a wide temperature range of 65ºC to 200ºC and variable reaction time. Amiri et al. (2017) studied the thermal pre-treatment of OFMSW, leachate, and sludge mix at 65ºC for 60 min and reported the maximum biogas yield of 450 mL/gCOD, i.e., 7% higher than control. Ariunbaatar et al. (2014b) studied the thermal pre-treatment of OFMSW at 80ºC for 1.5 h, resulting in a 52% increase in methane production compared to control (untreated OFMSW). Contrarily, Gonzalez-Fernandez et al. (2012) reported no enhancement in the biogas production for household waste pre-treated at 70ºC for 60 minutes. Li and Jin (2015) pre-treated the food waste (FW) at 70 ºC (70 min) and 90ºC (70 min) and observed the 25% and 29% higher biogas yield over control, respectively. However, maximum methane yield of 899 $mLCH_4/gVS_{added}$, i.e., 48% higher than control, was observed for 120ºC (50 min). Deepanraj et al. (2017) observed that pre-treatment of OFMSW at 120ºC for 30 min showed a VS removal of 62%, COD removal of 50%, and a 4.67% higher biogas production over control. Similarly, a 24% increase in methane production was observed on pre-treatment of OFMSW at 120ºC (Ma et al., 2011). Thus, low thermal pre-treatment (<100°C) was not effective for the enhanced biogas production. However, thermal pre-treatment at 120ºC showed a notable increase in biogas generation over control.

Thermal pre-treatment of organic substrates above 150ºC has been reported to increase the substrate solubilization. However, it decreases the biogas yield due to the formation of recalcitrant phenolic compounds inhibitory to anaerobes (Hendrik et al., 2009). Earlier studies reported the formation of melanoidin, a complex co-polymer formed due to the Maillard reaction between the carbohydrates and amino acids, which are difficult to degrade. The studies on thermal pre-treatment of OFMSW at 175ºC showed only a 3% and 11% increase in biogas yield compared to control (Schieder et al., 2000; Liu et al.,2012). In most of the studies carried out above 160ºC, despite achieving higher COD solubilization, the AD process was unable to significantly transform the solubilized fraction into biogas due to the presence of recalcitrant, formed at the higher temperature. However, sometimes it turns out to be in negative energy yield gain. In another study, a mixture of OFMSW and slaughter house waste (SHW) showed a 53% decrease in biogas yield as compared to control due to accumulation of volatile fatty acids (VFAs), long-chain fatty acids (LCFAs), and fats accumulation, which was perceived to form due to refractory compounds (Cuetos et al., 2010). Similarly, the biogas and methane generation was reduced by 3.4% and 7.5%, respectively, during anaerobic digestion of food waste, pre-treated at 170 ºC for 1 h (Qiao et al., 2011). Tampio et al. (2014) studied the thermal autoclaving of OFMSW at 160ºC and 6.2 bar pressure. They reported a 22% increase in NH_4-N and a 16% increase in soluble COD (sCOD). However, 11% lower CH_4 production in comparison with control (untreated OFMSW). Thus, higher temperature (>150 ºC) pre-treatment with longer reaction time triggered the Maillard reaction and recalcitrant formation, which inhibited the process performance and reduced the biogas yield. The melanoidins formation shows a positive correlation with protein and carbohydrate concentration in OFMSW (Liu et al., 2012). Thus, thermal pre-treatment at high temperature is less favourable for process enhancement. Thermal pre-treatment of OFMSW at a lower temperature (≈120 ºC) can improved the AD process performance and enhanced biogas yields.

5.2 Microwave (MW) heating

The key advantage of microwave (MW) thermal pre-treatment over conventional heating is rapid and selective heating, accelerated reaction rates, instant On-Off control, and improved energy efficiency. In contrast, conventional thermal pre-treatment involves high energy consumption (Tyagi and Lo., 2013). The microwave (MW) generally operates at a frequency of 2.45 GHz and a wavelength of 0.12 m. The polar molecules (e.g., water) within and outside the substrate are the targets of the electromagnetic radiations. In this way, the microwave dipoles align in the radiation field, causing a displacement inside the substrate, generating heat, and the organic matter in the complex substrates like OFMSW releases into the soluble phase, thus increasing the easily biodegradable fraction into the medium. Figure 2 shows the mechanism of disintegration of the complex structure of the OFMSW particles. The bipolar components like water, fat, proteins and carbohydrate in the OFMSW are influenced by microwave pre-treatment. The main factors which influence the pre-treatment of OFMSW are temperature, MW power, and irradiation time. The pre-treatment power applied ranges between 440-500 W, temperature between 30-175ºC, and irradiation time between 1-10 minutes, although few studies reported the irradiation time > 10 minutes (Tyagi and Lo, 2013; Aguilar-Reynosa et al., 2017; Tyagi et al., 2018). Microwave pre-treatment has two kinds of treatment effects: thermal and athermal. The thermal effect is caused by an increase in temperature, while the athermal effect occurs when the electric field can force the polarised side of organics to break the hydrogen bond and change their structure. The substrates solubilization and increment in biogas production are taking place adhered to both the thermal and athermal effect of microwave treatment (Aguilar-Reynosa et al., 2017).

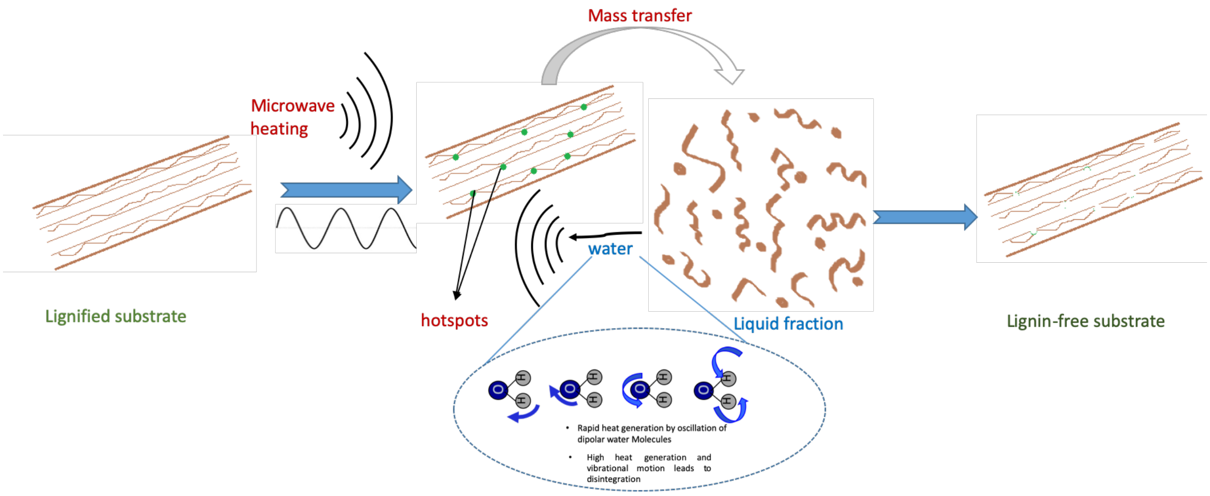

Fig. 2. Microwave pre-treatment of OFMSW leading to delignification of complex structure (Redrawn from Aguilar-Reynosa et al., 2017 and Tyagi and Lo, 2013)

Shahriari et al. (2013) observed that microwave pre-treatment of OFMSW (145ºC, ramp rate of 2.7ºC/min.) increased the sCOD by 26%, and achieved 7% higher biogas yield over control. However, biogas generation was reduced for OFMSW MW pre-treated at 175ºC, owing to the formation of recalcitrant and inhibitory compounds (melanoidins and humic acids). Ara et al. (2014) reported a biogas yield of 1760 mL/gVS$_{added}$ on anaerobic co-digestion (AcoD) of mixed OFMSW- primary sludge (PS) and WASMW (microwave pre-treated WAS: 135ºC, 1 min. at a rate of 25ºC/min.) at a ratio of 75:12.5:12.5, which was 11% higher than the control. The COD solubilization of 104% was observed at the above pre-treatment conditions as compared to the control. Percorini et al. (2016) studied the effect of MW pre-treatment of OFMSW on solubilization of COD, carbohydrates and proteins, and cumulative methane production. An increase in sCOD by 219% was observed in MW pre-treated OFMSW, while methane production was increased by 8.5% for MW substrate over control. Marin et al. (2010) performed MW pre-treatment of OFMSW at 175ºC for 1 min before anaerobic digestion. The solubilization of 82%, 78%, and 88% was observed for sugar, protein, and humic acid with a methane yield of 340 mL/gVS_{added} (14% increase from control). The MW radiations released bound water in the soluble fraction and enhanced hydrolysis of OFMSW. Bundhoo et al. (2017) observed that MW pre-treatment of OFMSW at 6946 kJ/kg TS specific energy resulted in 11000 mg/L sCOD over 6000 mg/L sCOD for untreated OFMSW. On the contrary, MW pre-treatment was unable to enhance bio-hydrogen production due to the formation of recalcitrant/ inhibitory compounds, accumulation of VFAs in the reactor, thus, incomplete bio-transformation of organic matter into bio-hydrogen production during anaerobic digestion. Savoo and Mudhoo (2018) observed that MW pre-treatment of OFMSW at 350 watt (W) for 15 min resulted in 39% COD solubilization, 271 mL /g $VS_{removed}$ biogas yield, and 35% VS removal. Zhang et al. (2016) observed the methane yields of 316 and 338 mL/gVS_{added} for MW pre-treated OFMSW-non-pre-treated SS, and non-pre-treated OFMSW with microwave pre-treated SS at 100ºC, respectively. On the other hands, microwave pre-treatment at specific energy ranges from 2333 to 12000 kJ/kg has led to the formation of inhibitory/refractory compounds, thus inhibiting the anaerobic digestion (Rani et al., 2013).

The key advantages of MW pre-treatment of OFMSW are the fast heat transfer, lower energy demand, and short reaction time, leaving it with little or no degradation products; the heating is all around the material and dielectric; gives a high yield of biogas; cost-effective and energy-efficient heating. Apart from this, the MW is a compact equipment with fast positioning. Heat loss is minimized in microwave heating as the heat passes to the substrate without heating the vessel and allows substrate overheating (as the boiling point does not limit the maximum temperature). MW pre-treatment disadvantages are the uneven and non-uniform distribution of MW power in the heterogeneous substrate like OFMSW, forming standing waves leading to local overheating (Aguilar-Reynosa et al., 2017). The MW pre-treatment for OFMSW was not reported as effective as conventional thermal pre-treatment for substrate solubilization and net biogas recovery. No significant relationship could be established between OFMSW solubilization and biogas yield. Therefore, more investigations are needed on MW thermal pre-treatment's practicality in terms of energy input and its transformation to substrate solubilization and subsequent biogas recovery.

5.3 Thermal-chemical

In thermo-chemical pre-treatment, strong alkalis, acids, and oxidants and thermal treatment are used to solubilize the organic matter in substrates and enhance the biogas recovery and VS removal in anaerobic digestion.

5.3.1. Thermal-alkali

When alkali pre-treatment is coupled with thermal treatment, the two important functions are solvation and saponification of lignin-carbohydrate bonds, which enlarges the surface areas and de-crystallizes the OFMSW. Solvation removes the lignin, acetyl groups, uronic acid of hemicellulose, breaks the lignin structure, and disrupts the bonds between the lignin and other components. It leads to swelling of the substrate, increasing its surface area, leading to easy accessibility of organic matter to anaerobes. Moreover, the substrate consumes some of the alkali, causing a balance in pH (Ariunbataar et al., 2014a; Xu et al., 2019). Of the many chemicals used for thermo-chemical pre-treatment, NaOH disintegrates the complex biomass structure, hence making it vulnerable to microbial enzymatic degradation. Bala et al. (2019) studied the thermal (180ºC, 60 min.) and thermo-chemical (3g/L NaOH, 180ºC, 60 min.) pre-treatment of OFMSW. They observed a 6.87% and 11.60% increase in sCOD, respectively, and a 54% increase in biogas production (thermo-alkali) over control. Abudi et al. (2016)

observed a 559 mL/g VS_{added} biogas yield and 79.8% VS removal when OFMSW was co-digested with thermo-chemically pre-treated WAS (90ºC, pH 11 for 10 h) and chemically pre-treated rice straw (RS) (3% H_2O_2 w/w) at an OFMSW:WAS: RS ratio of 3:0.5:0.5. Wang et al. (2009) optimized the alkaline-hydrothermal pre-treatment of OFMSW at 170ºC (1 h) with 4g NaOH/100g solid dosage. They observed a 50% higher biogas yield over control. Guelfo et al. (2011) optimized the best conditions for thermo-chemical pre-treatment of OFMSW for organic matter solubilization at 180ºC and 3g/L NaOH dosage. They reported that the sCOD was increased by 246% compared to the control.

5.3.2. Thermal-acid

The hybrid thermal-acid pre-treatment hydrolyses the hemicellulose to monosaccharides, increasing the cell wall's volume, size of pores, and enzymatic attack on the cellulose. It leads to the dissolution of lignin to a greater extent (Ariunbataar et al., 2014a, Xu et al., 2019). Vavouraki et al. (2013) optimized the combined effect of H_2SO_4, HCl, NaOH, H_2SO_3 at 50ºC, 75ºC, and 120ºC at a residence time of 30-120 minutes. They reported that the thermo-chemical pre-treatment with 1.12%-1.17% HCl at 100ºC increased the concentration of soluble sugars by 120% (due to mono-sugars glucose and fructose) over control. Nevertheless, higher COD solubilization does not mean higher conversion to biogas yield due to the formation of phenolic or furanic compounds (furfurals and hydroxymethylfurfural, HMF), recalcitrant formation from Maillard reactions, affecting biogas recovery. Ma et al. (2011) reported a 14% decrease in methane yield (over control) for thermal-acidic pre-treated OFMSW (120ºC and HCl until pH 2) despite achieving a higher degree of COD solubilization (32%). Thus, substrate solubilization in terms of COD, proteins, and sugars cannot accurately reflect the potential of thermal/hybrid thermal pre-treatments. Therefore, substrate pre-treatment followed by anaerobic digestibility of pre-treated substrate together can evaluate the actual effectiveness of thermal pre-treatment of OFMSW. The hybrid weak acid (per-oxide)- low-temperature pre-treatment (85ºC) could be an excellent option to achieve higher process performance (Shahriari et al., 2012).

5.3.3. Hydrothermal carbonization (HTC)

Hydrothermal carbonization (HTC) is another type of thermo-chemical pre-treatment, carried out at temperature ranges of 180-250ºC, the reaction time of 0.5-8 hours, and vapor pressure of 10-50 bar. Lucian et al. (2020) studied the effect of HTC treatment at 180ºC-1hr., 220ºC-3 hr., and 250ºC-6 hr. The specific methane yield from the soluble fraction of the HTC was the highest (205 mL CH4/ gCOD) at 180ºC-1 h when compared with 166 mL CH4/gCOD at 250ºC-6 h. Similarly, the methane yield from the hydrochar slurry was about 350 mL/gCOD at 180ºC-1 h compared to 50 mL/gCOD at 250ºC-6 h. The high methane production at 180ºC-1 h was due to the high biodegradability of hydrochars at a low temperature-reaction time and the availability of high soluble proteins and sugars concentrations. Moreover, the biochar can be a carrier for microbes, thus having sufficient active biomass. Also, hydrochar is rich in oxygen functional groups. Thus the VFAs get easily converted to methane, and ammonia inhibition is reduced (Lucian et al. (2020).

5.3.4. Recalcitrant formation

The thermo-chemical pre-treatment at high temperatures and chemical dosage also leads to recalcitrant production like furfurals and Hydroxymethylfurfural (HMF) as inhibitory products. Shahriari et al. (2012) studied the effect of microwave (115ºC, 145ºC, and 175º C) and thermo-chemical (0.38 gH_2O_2/gTS and 0.66 gH_2O_2/gTS, 85ºC) pre-treatment of OFMSW on AD process performance. At 115ºC and 145ºC, a 4-7% improvement in biogas production was observed over control, whereas at 175ºC, a decrease in biogas production owing to the formation of refractory compounds has been observed. The thermo-chemical pre-treatment of OFMSW showed a 7% improvement in cumulative biogas production (CBP) compared to control. Hamzawi et al. (1998) observed a decrease in biogas yield by 5% compared to control when OFMSW and SS mix (25:75) was pre-treated at 130ºC with 185 meq/L NaOH dosage. Ma et al. (2011) carried out a comparative study on pre-treatment of OFMSW using two different approaches of thermal (120ºC, 1 bar pressure) and thermo-chemical (120ºC and pH 2) pre-treatment. The highest COD solubilization of 32% was obtained for thermo-chemical pre-treatment, followed by thermal pre-treatment (19% COD solubilization). However, a 14% decrease in methane production was observed during thermo-chemical pre-treatment and only a 3% increment in methane production for thermally pre-treated substrate, i.e., due to the formation of refractory compounds like carboxylic acids, furans, and phenolic compounds. The thermo-chemical pre-treatment enhances the substrate solubilization to a certain degree, but this does not confirm the improved methane production, i.e., due to the formation of inhibitory compounds, challenging to degrade molecules, and resulting toxicity by chemicals.

Thermo-chemical pre-treatment of OFMSW causes low toxicity, thermal stability, improved electro-chemical stability, and low hydrophobicity. During thermo-chemical pre-treatment of OFMSW, the inhibitory concentrations of cations like Na^+, K^+, Mg^{2+}, Ca^{2+} affect the AD as the chemicals are mostly added in salt form. Kim et al. (2000) reported that more than 5g/L of sodium during the pre-treatment of OFMSW showed a decreased biogas production. The toxicity of sodium is more towards propionic acid consuming bacteria as compared to the VFA degrading bacteria. Bashir et al. (2004) stated that potassium inhibition starts at a concentration of 400 mg/L, although the tolerance concentration of anaerobic microbes is up to 8000 mg/L. High levels of calcium ions cause scaling of reactors and biomass, reduces the buffering capacity, and decreases the methane yield. In contrast, magnesium ions cause disaggregation of methanogens and inhibition in acetate conversion. 200 mg Ca/L and

720 mg Mg/L are determined as the optimum concentrations of calcium and magnesium ions in thermo-chemical pre-treatment of OFMSW (Ariunbataar et al., 2014a; Schimdt et al.,1993). It can be concluded from this section that thermo-alkali pre-treatment is more effective than acid pre-treatment in terms of substrate solubilization and biogas yield. The integrated alkali-thermal pre-treatment could make the combined process energy efficient and helps to maintain the alkaline medium inside the digester. However, the key disadvantage of chemically added thermal pre-treatment could be excess chemical cost owe to pH adjustment requirement for anaerobic digestion.

Table 3. Effect of thermal pre-treatment on substrate solubilization and biogas production

Substrate	Pre-treatment condition	Solubilisation effect	Biogas/methane yield	Reference
Conventional Thermal Pre-treatment				
OFMSW-Leachate	65ºC, 60 min	-	–7% higher biogas yield	Amiri et al., 2017
OFMSW	80ºC for 90 min	-	–647 $mLCH_4/gVS_{added}$, 52% increase over control	Ariunbaatar et al., 2014b
FW	70ºC		- 91% of FW transformed to 91% bio-hythane: 8% H_2 and 83% CH_4	Kim et al., 2000b
FW	70ºC, 2 h	-	–3% higher methane generation	Wang et al., 2006
FW	70º, 70 min 90ºC, 70 min		- 25% higher biogas yield - 29% higher biogas yield over control	Li and Jin, 2015
OFMSW	120ºC for 30 min	62% VS removal; 50% COD removal	–870 mL/gVS_{added} –4.67% higher biogas production.	Deepanraj et al., 2017
OFMSW	120ºC	-	–24% increase over control	Ma et al., 2011
FW	120ºC, 50 min	-	–899 $mLCH_4/gVS_{added}$, 48% higher CH_4 than control	Li and Jin, 2015
FW	150ºC, 1 h	-	–11.9 % higher methane generation	Wang et al., 2006

OFMSW	160ºC, 6.2 bar	22% increase in NH_4-N; 16% increase in sCOD	–445 mL CH_4/g VS_{added} –11% lower CH_4 production than control	Tampio et al., 2014
OFMSW	175ºC, 60 min	+114- 312 % sugars solubilization, +204-185% Proteins solubilizations	–7.9 and 11.7 % decrease in biogas production over control	Liu et al., 2012
OFMSW	175ºC-200ºC, 40 bar, 60 min	55-70% COD solubilization	–3% higher biogas production over control	Schieder et al., 2000
Microwave Pre-treatment				
OFMSW	145ºC at a ramp rate 2.7ºC/min 175 ºC	sCOD increased from 15% to 26%	–7% increase at 145 ºC –No increase at 175ºC	Shahriari et al., 2013
OFMSW-WAS	135ºC for 1 min holding time @ 25ºC/min	104% COD solubilisation	–1760 mL/gVS_{added}	Ara et al., 2104
OFMSW	Microwave (96 ºC, 4 min.) and Autoclave (134ºC, 2 bar, 15 min.) pre-treatments	219% COD solubilisation	–8.5% higher CH_4 yield in MW pre-treatment over control –4.4% higher CH_4 yield in autoclave pre-treatment over control	Pecorini et al., 2016
OFMSW	175ºC for 1 min, 7.8 ºC /min	82% sugar solubilisation, 78% protein solubilisation 88% humic acid solubilisation	–340 mL/gVS_{added} –14% increase over control	Marin et al., 2010
OFMSW	6946 kJ/kg TS specific energy	sCOD of 11000 mg/L	-No enhancement in H_2 yield due to formation of recalcitrant	Bundhoo et al., 2017
OFMSW	298kJ/kg TS of microwave pre-treatment	32% VS reduction	- No enhancement in H_2 yield due to formation of recalcitrant	Bundhoo et al., 2017

OFMSW	350 watt for 15 min	39% COD solubilisation 35% VS reduction	−271 mL/g $VS_{removed}$	Savoo and Mudhoo, 2018
Pre-treated OFMSW and non-pre-treated SS	100ºC	-	−316 mL/gVS_{added} (+6% higher over control)	Zhang et al., 2016

Thermo-chemical Pre-treatment

FW	70°C for 60 min KOH, pH = 10 at 70°C, 60 min		- Methane yield of 500 mlCH4/gVS_{added}, - No increase for pre-treatment	Chamchoi et al., 2011
OFMSW-WAS-Rice straw	AcoD with WAS (pre-treated at 90ºC at pH 11 for 10 h) and rice straw(RS) (pre-treated with 3% H_2O_2 w/w)	-	−559 mL/g VS_{added} −79.8% VS removal	Abudi et al., 2016
FW	0.4 N NaOH and autoclaved at 120°C for 30 min	Enhanced VS and COD solubilization	−360.7 mLCH_4/g $VS_{removed}$ – + 33% methane yield	Naran et al., 2016
OFMSW	130ºC , 185 meq/L NaOH., AcoD with SS	-	−5% decrease in biogas yield	Hamzawi et al., 1998
OFMSW	170ºC at 4g NaOH/100g solid	COD concentration of 13,936 mg/L	−164 mL/g VS_{added} −50% higher biogas yield	Wang et al., 2009
OFMSW	3g/L NaOH, 180ºC, 60 min	11.6% increase in sCOD; 2.36% reduction in VS%	−54% increase over control	Bala et al., 2019
OFMSW	180ºC and 3g/L NaOH	sCOD increased by 246%	–	Guelfo et al., 2011
OFMSW	0.66 g_{H2O2}/g_{TS} and 85ºC for 4 min	9.1% COD solubilisation 1.5% VS solubilisation	−496 mL/gVS_{added} biogas −5.8% improvement	Shahriari et al., 2012

OFMSW	120ºC and HCl until pH 2	32% COD solubilisation	−14% decrease in methane yield	Ma et al., 2011
OFMSW	1.12%-1.17% HCl at 100ºC	Increase in soluble sugars by 120%	-	Vavouraki et al., 2013
OFMSW	1% HCl at 60 ºC , 90 min	95% higher soluble sugar than non-pre-treated waste	-	Kerimak Öner, 2018
			–	

6. Thermal hydrolysis process: Lab to Field

The thermal hydrolysis process (THP) is a biomass pre-treatment process that has been commercially established for the pre-treatment of dewatered sludge and food waste before anaerobic digestion. It produces Class A biosolids, rendering a more digestible residue and improved dewatering characteristics, reducing its viscosity, enabling the digesters to reduce their volume requirements as they could operate at a much higher total solids content. The CambiTHP process worked at an applied pressure of 6 bar, 165ºC temperature, and a reaction time of 30 min (Figure 3).

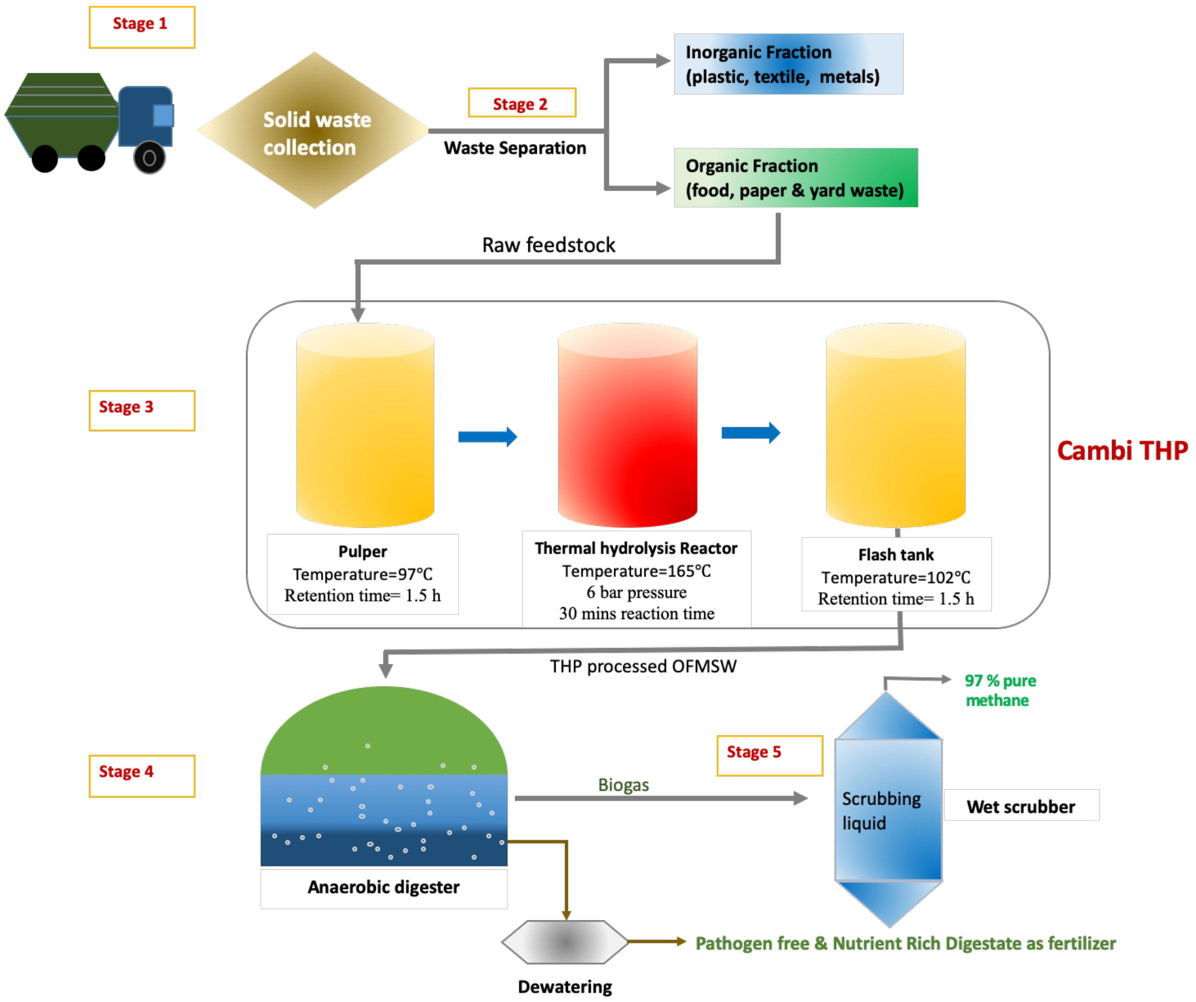

Fig 3. Various treatment stages of CambiTHP® process

(S1) Solid waste collection (S2) Waste separation (S3) Thermal hydrolysis of Organic fraction (S4) Anaerobic digestion of THP processes waste (S5) Biogas purification for end usage.

In the Cambi THP process, the steam injection is discontinuous (based on timers and number of reactors), has a flash steam heat recovery system, works at a solid feed of 16% TS and the standard size in multiples of 20 tons per day. Cambi THP system required less steam, as the steam recycles from the flash tank to the pulper when fed at solid content < 25% TS (Abu Orf et al., 2012). High disintegration of cells takes place, and organic solids get dissolved into the water at high temperatures. The complex structure of proteins and carbohydrates gets reduced to the single monomer of saccharides and amino acids, which acidify to short-chain fatty acids during anaerobic digestion. In AD, these fatty acids convert to biogas, leading to enhance biogas yield and improved digestate dewaterability. The feedstock is well sterilized, due to which any risk of pathogens contamination is eliminated.

In a lab scale work, Svensson et al. (2017) pre-treated (Steam explosion) the FW at 170 ºC for 30 min and reported a methane yield of 543 mL/ gVS_{added}, which was 6.1% higher than the non-pretreated FW. In another lab study, Svensson et al. (2018a) pre-treated (175ºC for 30 min) the centrifuged cake collected from two different full-scale digesters treating OFMSW and sewage sludge, and observed a methane yield of 415 mL/ gVS_{added} (+12% increase) and a notable reduction of COD (74%) and VS (72%). Moreover, improved dewatering of digestate leads to a 60% reduction in final wet cake mass over control. Thermal hydrolysis (steam explosion) of FW at 135 ºC for 20 min resulted in 601 mL/ gVS_{added} methane yield and 71% COD reduction (Svensson et al., 2018b). The thermal hydrolysis process has been successfully applied for source-separated OFMSW and co-digestion with sewage sludge at Lillehammer, Oslo, and Verdal (Barber, 2016). At the Verdal plant, 16,000 tonnes/y (wet) food waste has been processed with 9000 tonnes/y (wet) sludge. A biogas yield of 534 mL/g VS_{added} and 65% VS reduction has been achieved (Panter, 2011). In Lillehammer, 14,000 tonnes/y of OFMSW (70-82% food waste, <7% garden waste, <7% paper waste, <12% nappies) are processed, and

70% VS removal has been reported (Sargalski et al., 2007). Cambi has installed six food waste-sewage sludge co-digestion plants in China, Norway, South Korea, and Sweden. The Luoqi project in Chongquing, China, is the largest facility that has been constructed so far. The variable mixing ratios of FW and sludge have been pre-treated and co-digested. In Chongqing (China), sewage sludge is pre-treated by the THP process, then mixed with food waste before feed to an anaerobic digester. In Anyang (South Korea), pre-treated food waste is mixed with THP treated sludge and co-digested (Sahu, 2019).

7. Techno-economic and environmental feasibility

Until now, the economic feasibility analysis of thermal pre-treatment methods for OFMSW processing at lab, pilot, or full scales are limited in the literature. Ma et al. (2011) carried out the cost-benefit analysis of acid, alkali pre-treatments alone and in combination with thermal pre-treatment to improve the anaerobic digestibility of FW. They observed the best condition of 18 US$/ ton FW with acid pre-treatment. Although the thermal pre-treatment methods were observed energy-intensive, the surplus biogas recovery can level up the additional expenses, thus make the process profitable, i.e., net profit of around +0.6 US$ for thermal and »5 US$/ton FW for thermo-acid pre-treatments can be achieved. On the other hand, Ariunbaatar et al. (2014b) reported a profit of 9.0-16.0 US$ /ton FW could be achieved from net energy recovery after thermal hydrolysis of FW at 80 ºC for 1.5 h. However, thermal pre-treatment at 120 ºC could yield a profit of 0.5 US$/ ton FW. Yang et al. (2010) proposed that thermal pre-treatment significantly improved the biogas yield, and the surplus biogas recovered can be utilized to reduce the cost thru an efficient heat exchanger. The use of thermal pre-treatment expedites heat recapturing via cool down the pretreated feedstock from thermal pre-treatment to the digestion temperature. Pecorini et al. (2016) carried out the specific energy assessment of microwaved and autoclaved OFMSW and considered energy demand, net energy recovery in terms of biogas and heat, and profit of the pre-treatments. No energy profits were achieved for both pre-treatments, however, MW (-1324 kJ/kgVS) showed a better energetic response than autoclave (-2658 kJ/kgVS) pre-treatment. Fan et al. (2018) compared the energy input, carbon footprints, and the process enhancement in terms of biogas yield for conventional thermal and microwave pre-treatment processes. They reported that despite achieving the enhanced biogas production, the carbon footprints (60- 4218 kg CO2/ ton waste) and energy demand by MW pre-treatment are relatively higher than thermal hydrolysis (carbon footprint: 59–420 kg CO2/ ton waste). In conventional thermal pretreatment, the energy input and % process enhancement for biogas yield are ranging from 0.15 to 0.59 kWh/L, -3.4% to +31.5%, and 112 to 800 kWh/ton, -5 to +15.4%, respectively. The large variations in the biogas improvement could result from distinctive characteristics of OFMSW and operational conditions. For MW pre-treatment, the energy input and % process enhancement for biogas yield are ranging from 114 to 8040 kWh/ton, +4 to +39.3%, respectively. Earlier review works suggested that net paybacks (monetary and energy balances), carbon emission footprints, local situations like workforce availability and cost, collection and transportation cost, treatment capacity, extra mixing and pumping requirement, energy costs, taxes and tariffs, land worth, marketplace, cost of value-added products recovered, and residue disposal should be taken into account to realize the true economic potential and practicality of the technology (Ariunbaatar et al., 2014a; Cesaro and Belgiorno, 2014). The energy, economic and environmental feasibility of a thermal pre-treatment process can be enhanced through the incorporation of the usage of renewable energy (e.g., solar), waste segregation at source, co-digestion approach, and avoidance of high-temperature thermal pre-treatment of carbohydrate and protein-rich substrate (Forster-Carneiro et al., 2008; Ariunbaatar et al., 2014b; Fan et al., 2018). Due to the lack of scientific studies, no conclusive techno-economic and environmental assessment for thermal pre-treatment of OFMSW could be made in this review.

8. Discussion and Future Perspective

Thermal pre-treatment has been proved to be one of the widely applied methods for enhancing the substrates solubilization and improve AD process performance. The pre-treatment temperature and reaction time have a significant effect on substrate solubilization and overall process performance. Thermophilic and temperature phased anaerobic digestion (TPAD) offers the best conditions for the treatment of OFMSW. Both processes show effective and stable performance at low HRTs and high OLRs and achieved higher methane yield than mesophilic digestion. Nevertheless, all these processes were encountered a typical VS removal of 60%, high HRT (25-30 days), and low OLR of < 7.0 kg VS/ m^3. d. Low thermal pre-treatment (<100°C) is not effective for enhancing substrate solubilization and biogas production. However, thermal pre-treatment at 120ºC showed a notable increase in biogas generation over control. Higher temperature (>150 ºC) pre-treatment with longer reaction time triggered the Maillard reaction and recalcitrant formation, which inhibit the process performance and reduce the biogas yield. The addition of conductive materials like ferric (Fe^{3+}) salts before thermal pre-treatment of organic substrate can lead to a pathway whereby the recalcitrant formation can be mitigated (Gahlot et al., 2020). The Maillard reaction intermediates can get oxidized either by direct oxidation or by Fenton-like reactions, thereby reducing recalcitrant formation. Earlier research has reported the role of direct interspecies electron transfer (DIET), which allows the direct flow of electrons from one cell to the other cell bypassing the need for hydrogen to transform organic matter into methane (Gahlot et al., 2020). Although carbon-based conductive materials like granular activated carbon (GAC), biochar, carbon nanotubes, etc., have proven to be efficient in enhancing the biogas production of organic wastes, their role in mitigating the toxic effects of recalcitrant in thermally pre-treated organic waste and OFMSW is yet to be validated. MW thermal pre-treatment for OFMSW was not reported as effective as conventional thermal pre-treatment for substrate solubilization and net biogas recovery. For MW pre-treatment, no significant relationship could be established between OFMSW solubilization and biogas yield. More investigations are needed on MW thermal pre-treatment's practicality in terms of energy input and its transformation to substrate solubilization and subsequent biogas recovery. Thermo-alkali pre-treatment is more effective than acid pre-treatment in terms of substrate solubilization and biogas yield. The integrated thermal-alkali pre-treatment could make the combined process energy-efficient and maintain the digester's alkaline medium. However, chemically added thermal pre-treatment's main disadvantage could be excess chemical cost owe to pH adjustment requirement for anaerobic digestion. The substrate solubilization (COD, protein, and carbohydrates) followed by anaerobic biodegradation and process yield in terms of solubilized substrate conversion rate to net methane recovery should be considered to realize the true potential of the thermal pre-treatment method. Commercially, Cambi thermal hydrolysis process (CambiTHP) has gained worldwide applicability, which operates at 160℃ temperature, 6 bar pressure, and 30 min reaction time. The anaerobic co-digestion of THP processed mixed OFMSW and sewage sludge achieved high biogas yield and VS removal and produce Class A biosolids of excellent dewatering and fertilizer value. Thermal pre-treatment variables such as the temperature used, the time of pre-treatment, and the heat type applied are essential to identify the best strategy to enhance AD performance and make the process economical.

9. Conclusions

Thermal pre-treatment enhanced the solubilization and anaerobic digestibility of OFMSW. Thermophilic and temperature phased anaerobic digestion (TPAD) processes shows effective and stable performance at low HRT and high OLR and achieved higher methane yield than mesophilic digestion. Thermal pre-treatment at 120 ºC improves the net biogas yield. High-temperature, >150°C, result in decreased biogas yield due to recalcitrant formation. Recalcitrant formation and inhibition can be mitigated with conductive material mediated thermal pre-treatment and anaerobic digestion. COD solubilization cannot accurately reflect thermal pre-treatments' potential. Thus, pre-treatment followed by anaerobic digestibility of pretreated substrate together can evaluate the actual effectiveness of thermal pre-treatment.

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