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Chapter

Pre-treatment Technologies to Enhance Anaerobic Digestion

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Abstract

Sustainable energy production is the major priority in the world due to global warming, climate change, and fossil fuels depletion. Anaerobic digestion (AD) of sludge is the sustainable process producing the energy and minimizing the fossil fuel usage. However, conventional AD of sludge is not sustainable since it takes longer time for digestion which increases the energy input and greenhouse emissions. Therefore, pretreatment technologies have emerged to enhance methane production and thus the energy output from the AD process. In this chapter, pre-treatment technologies adopted mainly physical, chemical, thermal, and other advanced processes to enhance methane production in the last decade are elaborated. In addition, energy balance of the process and the feasibility of the pre-treatment technologies and their current status are discussed.

Keywords: waste activated sludge, pre-treatment, anaerobic digestion, methane, sustainability

1. Introduction

The production of inevitable waste activated sludge (WAS) as a by-product during the biological wastewater treatment demands for sustainable treatment options that assist in the proper utilization of sludge before its disposal. The composition of sludge mainly includes microbial cells and organic components such as proteins, carbohydrates, and lipids. Understanding the properties of the sludge may help in processing it to produce beneficial products or as a feedstock for bioenergy generation.

Anaerobic digestion (AD) for sludge stabilization is operated in about 38% of the total treatment plants, whereas only 6% of plants employ aerobic digestion and composting [1]. AD of sludge produces biogas, which mainly contains methane and carbon dioxide. AD of sludge is a sustainable process as it recovers energy from the biogas and replaces fossil fuel usage and minimizes GHG emissions. However, the efficacy of these processes is limited by the presence of complex structural components, extracellular polymeric substances, and rate-limiting cell lysis in WAS [2]. Moreover, the effects of hydrolytic enzymes are reduced in WAS as their penetration inside the bacterial cell is hindered by the cell walls, making the degradation of intracellular organic compounds tedious. Thus, it increases the digestion time and energy required for digestion processes. Hence, to overcome these drawbacks, pre-treatment technologies are adopted to break the cells and to liberate the cell constituents.

Various physical, chemical, and biological pre-treatment methods have been reported in the literature which is used individually or in combination for pre-treatment of WAS. These include treatment by hydrolysis, ultrasound, enzymatic lysis, acidification, alkaline hydrolysis, alkaline-thermal, and thermal-H_2O_2, microwave alkaline, and others [3]. The use of appropriate pre-treatment strategy can enhance the degradation and disintegration of both extracellular and intracellular substances reducing the retention time needed by biological digestion processes [4].

This chapter presents literature about the different methods of pre-treatment that have been used for enhancing the AD. Moreover, the factors affecting their operational efficiency have also been discussed. Furthermore, a brief account of the large-scale feasibility and economic aspects of the overall pre-treatment processes is discussed.

2. Pretreatment technologies

The pre-treatment technologies enable the cells constituents easily available for the microorganisms to produce the biogas. Various pre-treatment technologies (mainly mechanical, chemical, biological, and physio-chemical) and their effect on enhancing the AD and methane production presented in the literature during last decade are discussed here.

2.1 Mechanical pre-treatment

2.1.1 The process involved and mode of action

Mechanical pre-treatment disintegrates and/or grinds solid particles of the substrates, thus releasing cell compounds and increasing the specific surface area. The increased surface area provides better contact among substrate and anaerobic bacteria, which enhances the AD process [5]. A larger particle radius exhibits lower chemical oxygen demand (COD) degradation and a lower methane production rate [6]. Likewise, the particle size is inversely proportional to the maximum substrate utilization rate of the anaerobic microbes [7]. Therefore, mechanical pre-treatments such as sonication, liquid shear, collision, a high-pressure homogenizer and liquefaction are conducted to reduce the substrate particle size. During sonication the electrical energy from the source is converted to mechanical vibration which then converts to cavitation. The shear forces exerted as a result of cavitation cause WAS floc dispersion and further cell disintegration releasing organic macromolecules that are further degraded into short-chain compounds [8]. The irradiation intensity, time, and temperature-induced, as a result, can impart a cumulative effect enhancing the sludge degradation [9]. The main effect of ultrasonic pre-treatment is particle size reduction at low frequency (20–40 kHz) sound waves [10]. High-frequency sound waves also cause the formation of radicals such as HO^-, H^+, which results in oxidation of solid substances [11]. Besides ultrasonic irradiation, cavitation can also be produced by venturi meter tubes under controlled conditions of liquid flow [12].

High-pressure homogenization (HPH) pre-treatment involves the use of 30 and 150 MPa pressure for 3–30 min to pressurize the heterogeneous sludge components. The homogenization occurs due to shear, that is, when the pressurized sludge is released to impact on a ring [13]. The formed cavitation induces internal energy, which disrupts the cell membranes [14]. Both electroporation and liquefaction pre-treatments cause cellular structure damage, thus the effect on the AD process is similar to maceration [15]. Barjebruch and Kopplow treated surplus sludge with

HPH at 600 bars and showed that the filaments were completely disintegrated [14]. Increased 25% volatile solids reduction was observed in AD for HPH pretreated sewage sludge [16]. This improvement was induced by increased soluble protein, lipid, and carbohydrate concentration.

The advantages of mechanical pre-treatment include no odor generation, an easy implementation; better dewaterability of the final anaerobic residue, and moderate energy consumption. Disadvantages include no significant effect on pathogen removal and the possibility of equipment clogging or scaling [17].

2.1.2 Nanobubbles

Nanobubbles are spherical liquid structures containing gas which are stable and efficient when possessing typical overall diameters in the nanometer range (less than 10^3 nm). The presence of negative charge on nanobubbles is observed when present in pure water over a wide pH range. Nanobubbles stabilized their structures because of the same charge repulsion that occurred between adjacent nanobubbles [18]. However, some reports suggest hydrophobic attraction between negatively charged surfaces of nanobubbles and these contradictory reports could be attributed to the differences in nanobubble generation techniques, surface tension, or varying molecular arrangement at the gas-liquid interface [19]. Nanobubbles with diameters of approximately 13 nm have been well-engineered as spherical water packages with gas for food safety applications whose efficiency is well established based on bubble surface stability and the electrostatic charges present on the bubble surface [20]. Besides possessing high stability, nanobubbles in liquid systems also show a high mass transfer rate and enhanced solubility in gas [21]. Nanobubbles with a varying range of diameters have been engineered by different methods such as constant purging of octafluoropropane gas into an ultrasonicated solution of mixed surfactant which creates bubbles ranging in 400–700 nm mean diameter [22]. Palladium electrode with ultrasonication has been used to form nanobubbles of 300–500 nm diameter [23]. Nanobubbles form reactive free radicals as they collapse due to the presence of ions in groups at the gas-liquid interface [24]. The ability of nanobubbles to form reactive free radicals makes them potent applicants in the field of pre-treatment of wastewater components. In submerged systems, nanobubbles formed by the use of air or nitrogen are known to enhance the activity of aerobic and anaerobic microorganisms that improve the waste degradation efficiency and overall water quality [25]. According to the studies the higher negative charges were observed on sludge components on the addition of nitrogen gas nanobubble water the degradation of carbohydrates and proteins get increased along with methane production, that is, 29% more than that of control [25].

2.1.3 Hydrodynamic cavitation

Cavitation is a process of cavity bubble formation which burst within the liquid to create intense pressure spots and shock waves. These factors create localized energy and turbulence which causes an impact on adjacent particles and also mixing of insoluble substances like oil and water to form emulsion [26]. This mechanism is favorable in cases where AD of sludge is hindered due to the presence of lipid-containing substances which are insoluble in water. Their insolubility causes adversity in their interaction with hydrolytic bacteria which decreases the efficiency of the overall hydrolysis process. Applying localized energy supplies insignificant amounts to small elements of the liquid volume resulting in an increase of internal energy of the liquid elements to that point which causes phase change from liquid to gas and the formation of bubbles filled with vapor and gases. Following, when

the bubbles leave the high energy zones, they violently implode and disappear. The localized energy could be provided by a laser beam or a stream of heavy elementary particles such as protons by molecular or optical cavitation process based on the source of applied energy [27]. Hydrodynamic cavitation was frequently been proved as a more energy-efficient method compared to other cavitation techniques [28]. Hydrodynamic cavitation for pre-treatment of sludge where cavitation was generated by using a venturi cavitation system in which bubbles are created in venturi throat (constriction) has been used. The system achieved better energy efficiency than high-speed homogenizer in terms of soluble COD/kJ WAS and also the authors observed linear relationship between total solid concentration and the increased insoluble COD for WAS indicating towards better cavitation formation at high concentration of total solids [29]. In another study, the degradation of WAS was analyzed using a novel rotation generator of hydrodynamic cavitation at pilot scale [30]. Cavitation (as a pre-treatment) of WAS resulting in an increment in soluble COD from 45 to 602 mg/L along with a 12.7% increase in biogas production due to improved AD of the pretreated WAS [30].

2.2 Thermal pre-treatment

Thermal pre-treatment of WAS has been classified as low-temperature pre-treatment (<100°C) and high-temperature thermal pre-treatment at 100–210°C [31]. High-temperature pre-treatment cause disintegration of solids in sludge, removal of pathogens at low sludge retention time and leads to biogas production but it also has been reported that exposure to high temperature causes the formation of new chemical bonds which results in agglomeration of substances present in the sludge [32]. Both the thermal pre-treatment approaches have been reported to degrade volatile solids and produce biogas, however, the efficiency of high-temperature thermal pre-treatment for solids reduction and biogas formation has been known to be comparatively higher [11]. In thermal pre-treatment strategies, temperature and time of application are the main operational parameters which decide the success of the treatment process. When effectively applied, this pre-treatment can cause the disintegration of cell membranes accompanied by the solubilization of organic compounds [33]. During WAS pre-treatment, cell wall disruption and hydrolysis due to temperature generally occur when the temperature is in the range of 160–180°C at a pressure ranging from 600 to 2500 kPa for about an hour [32].

Microwaves generate heat by causing the movement of dipoles in polar molecules, realigning them, and producing thermal effects [34]. They cause both thermal and non-thermal effects (degradation of polymeric structure) on sludge, improving bio-gas production and reducing volatile solids. However, microwave generation requires higher energy consumption when compared to conventional thermal pre-treatment. The increase in temperature is associated with an increase in biodegradability while a higher concentration of solids present in WAS inversely affects the degree of pen-etration of microwaves to the sample [35].

2.2.1 High-temperature treatment

High-temperature thermal pre-treatment is performed at temperature >100°C. The heat exchangers or direct steam injection are used to supply the steam at the desired temperature [36]. The pressure is developed as a result of steam and high temperature which is abruptly released causing a sudden pressure drop. This sudden drop in pressure along with application time and temperature comprises the major parameters necessary for efficient solubilization and subsequent methane production in AD [37]. In many cases, it has been observed that when the temperature is raised

above 190°C, recalcitrant and inhibitory compounds, that is, ammonia is released which adversely affect the process [38]. Moreover, at a temperature above 180°C carbohydrates can react with protein amino terminals resulting in pyrolysis of sludge organic matter and formation non-biodegradable compounds [39]. In the case of sludge pre-treatment by high temperature, a range of 150–180°C at 600–2500 kPa @ 30 min to an hour is optimal because when the temperature is further increased, methane production is reduced due to formation of inhibitory products due to Maillard reaction. [38]. If the temperature is not in the required range, certain biomolecules are partially or incompletely degraded, e.g. proteins solubilize during sludge pre-treatment at 175°C but are not completely degraded to ammonia [40]. Some of the advantages of high-temperature thermal pre-treatment include the reduction in viscosity of sludge which in turn eases handling and transport costs [41]. Besides the reduction in viscosity, high-temperature pre-treatment at 134°C causes an increase in specific charge on sludge components as a result of colloids and extracellular polymer substance (EPS) release [42].

2.2.2 Low-temperature treatment

Low-temperature thermal pre-treatment of WAS deals with the application of <100°C temperature for a few minutes to several hours [31]. At temperatures ranging from 60 to 70°C, particle size reduction and solubilization of organic components occur [43]. Low-temperature thermal hydrolysis of sludge causes solubilization of organic matter and increase in activity of thermophilic bacteria activating the release of hydrolytic enzymes in sludge [44]. Also, rheological properties of sludge and concentration of methane in biogas during AD are positively influenced by thermal pre-treatment of sludge at low temperature [45]. The relation between pre-treatment temperature and the time of application is a very crucial factor that affects the WAS biodegradation rate [46]. It has been reported that deflocculating or reduction in the size of particles is observed when the temperature is applied in the range of 50–95°C resulting in an increased surface area which in turn increases the rate of hydrolysis in WAS [47]. The type of sludge being pretreated by thermal exposure also affects the efficiency of temperature treatment. At 70°C, the total percentage of volatile suspended solids removed from WAS was reported to be 17% which for primary non-stabilized raw sludge was only 28% indicating towards its low biodegradability [48].

2.3 Chemical pre-treatment

2.3.1 Procedure and mode of action

In chemical pre-treatment methods alkali, acid or advanced oxidation methods are used to disintegrate the organic sludge components and disrupt microbial cells (**Table 1**). AD generally requires an adjustment of the pH by increasing alkalinity, thus alkali pre-treatment is the preferred chemical method [87]. The increase in pH of WAS due to alkali pre-treatment causes many effects on sludge components which include saponification of lipid bilayer and protein denaturation in the cell membrane, solubilizing EPS by ionization of its carboxyl and amino groups and hydrolysis of sludge organic substances [99]. In the literature it was stated that excessive reagent doses can inhibit the anaerobic microbes and AD, which makes it important to control the amount and type of reagent used along with the pH desired [100]. Besides treatment with alkali and acids, oxidation processes like ozonation are also employed to increase sludge hydrolysis and biogas production rate. An advanced oxidation process like ozonation depends upon the oxidation

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
Physical approach						
Activated sludge (40.8 g TS/kg)	MW	Power = 800 W Duration = 3.5 min Energy = 336 kJ/kg TS	Increase of SCOD: 214%	AD, semi-continuous, 37°C, SRT 20 d, 42 d	+50% biogas production, +66.6% DS removal	[49]
Activated sludge (23 g TS/L)	Ultrasonication	Frequency = 24 kHz Power = 300 W Energy = ~5000 kJ/ kg TS	DDcod: 9%	AD, semi-continuous, 37°C, HRT 20 d, 80 d	+35% methane yield, 0.86 energy ratio	[50]
WAS	High-pressure thermal hydrolysis	Temp = 150°C Pressure = 3 bars Duration = 30 min	36% of active, Heterotrophs converted to readily biodegradable COD and 64%	Aerobic digestion	Increase in total mass = 21%	[51]
WAS	MW	Power = 600 W Temp = 85°C Duration = 2 min	COD solubilization up to 8.5%	NR[*]	NR	[52]
WAS	MW-Alkali	Power = 600W Duration = 2 min	COD solubilization increased up to 46%	Aerobic digestion	Soluble COD reduction = 93% VSS reduction = 63%	[52]
Mix primary and secondary sludge	Ultrasound	Power = 480 W Duration = 30 min	Dissolution of chemical oxygen demand up to 44.4%	Thermophilic aerobic digestion	VSS removal efficiency = 55% RT = 3 d	[53]
Activated sludge	Electro kinetic disintegration	Power = 19 kV Frequency = 110 Hz Duration = 1.5 s	Increase of SCOD/TCOD: 4.5 times, increase of exocellular polymers: 6.5 times	AD, Batch, 35°C, 20–30 d	+2.5 times higher biogas production	[54]
Thickened sludge	MW	Power = 1250 W Frequency = 2450 MHz Intensity = 100%	Increase of SCOD/TCOD from 0.06 to 0.2	AD, Semi-continuous TPAD	+106% biogas production, the maximum VS removal: 53.1%	[55]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
WAS (14.2±0.7 g TS/kg)	MW	Energy = 14,000 kJ/kg TS	Increase of SCOD/TCOD from 2 to 21%	Aerobic digestion, Batch, 35°C, 35 d	+570.7% biogas production	[56]
Meat processing Wastewater sludge	Alkaline-MW	Temp = 140°C Duration = 30min, pH = 13	Sludge disintegrate on the degree increased up to 54.9.VS solubilization increased up to 42.5%	Anaerobic digestion	Increase in biogas production = 44.5%	[57]
Excess sewage sludge	Ultrasonic-Fenton	Ultrasonic density = 720 W/L Duration = 20 min Fe2 + dosage = 0.4 g/L H$_2$O$_2$ dosage = 0.50 g/L Duration = 20 min	Soluble COD increased up to 2.1 fold	NR	NR	[58]
Thickened sludge (43.6 g TS/kg)	Ultrasonication	Power = 100 W Duration = 8 min Energy = 96 kJ/kg TS	Increase of SCOD: 1741%	AD, semi-continuous, 37°C, HRT 20 d, 67 d	+27% biogas production	[59]
Thickened sludge (43.6 g TS/kg)	MW	Frequency = 2.45 GHz Power = 800 W Duration = 1 min Energy=96kJ/kg TS	Increase of SCOD: 117%	AD, Semi-continuous, 37°C, HRT 20 d, 67 d	+ 20%biogas production	[59]
Primary sludge	Electro kinetic disintegration	Energy = 33 kWh/m^3	Accumulation of acetate increased by 2.6-fold	AD, MEC, anode potential: –0.3 V vs Ag/AgCl	+2.4-fold current density (~3.1 A/m^2)	[60]
Activated sludge	Electro kinetic disintegration	Energy = ~34kWh/m^3	Increase of SCOD: 220%	AD, CSTRs, 37 ± 1°C, SRT 20 d	+33% methane production, +18% TCOD removal, –40% digester size	[61]
WAS	Visible-photocatalysis	NR	Increase COD degradation up to 61.1%	AD	Up to 7866.7 mmol H$_2$/L-sludge of hydrogen production was achieved	[62]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
Mix waste activated and digested sludge	UV Photocatalysis	Catalyst = TiO$_2$ Duration = 4 h Temperature = 35°C UV intensity = 0.7 mW/cm^2	Soluble COD concentration increased from 1087.2 to 1451.6 mg/L for 8 h pre-treatment	AD	Methane production=1266.7 mL/L sludge, VS reduction = 67.4%, total COD reduction= 60.5%	[63]
Mixed sludge (132 ± 1 g TS/kg)	Ultrasonication	Power = 150 W Duration = 45 min	Increase of TOC: 81.5%, increase of TN: 50.0%	AD, Batch, 35°C, OLR 0.9 ± 0.1 kg VS/m^3 d	+95% methane yield	[64]
Dewatered sludge (15–20% TS)	Thermal hydrolysis	Full-scale CAMBI™ Temperature = 160°C Pressure = 6 bar	SS removal: 20–30%, increase of SCOD/TCOD from 0.04 to 0.4	AD, semi-continuous, 42 and 55°C, HRT 1–6 d, 142 d	+2–5 times in VFAs yield, +4–6 times in VFA production rate	[65]
Textile dying sludge	Ultrasonic-Fenton	Ultrasonic density= 0.14W/ml pH < 3.0	The floc structures disruption, increased from 1.48 to 6.96%	NR	NR	[66]
Concentrated sludge (40 g/L)	High-pressure homogenization (HPH)	Pressure = 150 bar, Flow rate = 2.7 m^3/h	NR	AD, full-scale, 36–38°C	+30% biogas production, +23% sludge reduction	[67]
Dewatered activated sludge (16%TS)	Thermal hydrolysis	Pilot-scale CAMBI™ Temperature = 65°C Pressure = 6 bar Duration = 20 min	Increase of VS removal from 26% to 42% (+62%)	AD, pilot-scale, treated sludge: primary sludge (80%:20%),3°C, SRT 20d	+2.3 times increase in SLR, +30–40%biogas production, improved dewaterability	[68]
Secondary sludge (30.00 g TS/L)	Thermal hydrolysis	Temperature = 134–140°C Pressure = 3.4 bar Duration = 30 min	NR	AD, batch, 35°C, HRT 30 d	+40.2% methane production, +12.6% VS removal, +6.8% digestated reduction	[69]
Secondary sludge (31.4 g TS/L)	Ultrasonication	Frequency = 20 kHz Power = 750 W Energy = 5742 kJ/kg TS	Increase of SCOD/TCOD from 0.02 to 0.10	AD, batch, 35°C, 30 d	+16.9% VS removal, +7.89 × 10-6 kWh/g energy output, 1.0 energy ratio	[69]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
Dairy activated sludge (11.66 g TS/L)	MW	Frequency = 2450 MHz Power = 900W Power = 12 min Energy = 1814 kJ/L	Increase of SCOD: 19%	AD, semi-continuous, 37°C, SRT 15 d, 170 d	+57% biogas production, +64% VS removal	[70]
WAS (35.5 ± 0.7 g TS/L)	Ultrasonication	Energy = 3380 kJ/kg TS	DDcod: 21%	AD TPAD-BMP assay (55°C→35°C)	+42% methane production, +13% VS removal	[71]
Mixed sludge	Electro kinetic disintegration	NR	Increase of SCOD: 160%, increase of DOC: 120%	AD, full-scale WWTP	+40% biogas production, biosolids requiring disposal reduced by 30%	[72]
WAS	Low thermal	Temp = 70 and 90°C Duration = 180 min	Sludge disintegration the rate was increased up to 25%	Anaerobic batch digestion	Increase in methane production = 21%	[45]
WAS	Mechanical	Mixing = 5000 rpm Duration = 10 min	Sludge disintegration the rate was increased up to 1.5 %	NR	NR	[45]
WAS	Ultrasonic-acid	Ultrasonic density= 10W/mL Duration = 10 min pH = 2.0	Sludge disintegration increased up to 40%	NR	NR	[73]
WAS	Electro kinetic disintegration	Energy = 10kWh/m^3	Increase of SCOD/TCOD to 10%, increase of SCOD from 20 to & $gt;1000 mg/L	AD, batch, 25–30 d	+100% methane production	[74]
Sewage sludge	Thermal	Temp = 120°C Pressure = 2 atm Duration = 15 min	Soluble carbon concentration, nitrogen, and phosphorus increased by 165, 16, and 24%,	AD	Increase in methane production = 29%	[75]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
WAS	MW—Alkali (NaOH)	Power = 900 W Temp = 95˚C pH = 12	Sludge solubilization increased from 0.5 (raw) to 52.5%	Mesophilic aerobic digestion	COD degradation =81.1% VSS degradation =72.4% VSS RT = 20 d	[76]
Mixed sludge	High-pressure homogenization	Pressure = 12,000 psi Catalyst = 0.009 g NaOH/g TS	SCOD/TCOD: & 2 gt; 4.0	AD, 2TPAD, SRT 14 d, OLR 1.24±0.05 g VS/L d	0.61–1.32 L CH4/L d methane production, 43–64% VS removal, pathogen removal, net energy output	[77]
WAS	Free nitrous acid-heat pre-treatment	Nitrous acid = 0.52–1.11 mg N/L Temp = 70°C	sCOD increased and found between 0.16 and 0.28 mg sCOD/mg VS	AD	Methane production increased by = 17–26%	[78]
Dewatered sludge (16.7 ± 0.5% TS	Thermal hydrolysis	Temperature = 140–160°C Duration = 60– 90 min	Increase of DDcod from 4.5 to 34.7–42.5% (+6.7–8.4 times)	AD, batch, 37°C,28d	+~16.5% biogas production, reduction of SRT from 18–20 d to 12–14 d	[79]
Primary sludge	MW- Ultrasound	Power = 800 W Frequency = 2450 MHz, Duration = 3min US density = 0.4 W/ml US intensity = 150 W Duration = 6 min	Increase in disintegration of flocs and extracellular polymeric substances	AD	Methane production = 11.9 ml/g tCOD	[80]
Mix aerobic thicken sewage sludge	NaOH Ultrasonic	NaOH dosage = 100 g/kg Duration = 30 min Ultrasonic energy = 7500 kJ\Kg	Soluble COD increased from 275 to 6797 mg/L	Aerobic digestion	Increase in organic matter degradation = 50.7%	[81]
Sewage sludge (23 g TS/L)	High-pressure homogenization (HPH)	Pressure = 50MPa Cycles = 2	SCOD: 2167 mg/L, DDcod: 7.7%	AD, batch, 35°C, 7 d	+115% biogas production, +41.17% VS removal, +61.89% TCOD removal	[82]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
Chemical approach						
Maize canning sludge	Ozonation	Ozone intensity = ~0.18 g O_3/g D	Increase of BOD5/COD from 26 to 93% (+2.58 times)	AD, batch,30°C, 30 d	Increase of biogas production from 1.037 to 9.52 cm^3/g COD d(+8.2 times)	[83]
WAS	Ozonation	Ozone intensity = 10 mg O_3/g TSS 20 cycles Duration = 30 s/cycle	DDcod:18%, VSS reduction:18%	AD, batch, F/I0.8, 35°C, 20 d	+800% specific biogas production, +1.6 folds VSS reduction	[84]
Activated sludge (5%TS)	Acidic treatment	8.75 mLHCl/kg wet sludge pH = 2	Four and Six times increase of soluble carbohydrate sand proteins, respectively	AD, semi-continuous, 35°C, HRT 12 d	+14.3% methane yield, –40% polymer dose for dewatering	[85]
Activated sludge	Ozonation	Ozone intensity = 0.09g O3/g MLSS, pH = 11	COD solubilization: 40%, TS reduction: 30%	AD, lab-scale AS-MBR, 120 d	Solids degradation: 37%	[86]
Sewage sludge	Alkaline treatment	0.1 mol NaOH/L	Increase of DDcod from 22.3 to 26.9%	AD, batch (BMP), 21 d	+26.4% organic removal, +1.5% biogas yield; delay of AD start up due to residual NaOH	[87]
Pulp and paper sludge	Alkaline treatment	8 g NaOH/100 g TS	Increase of SCOD:83%, 56–192% higher Sv	AD, batch, 37°C, 42 d	1040 mg acetate/L, +83% methane yield (0.32 m^3CH_4/kg S removed); sodium toxicity at 16 g NaOH/100 g TS	[88]
Secondary waste water sludge	Peroxide/oxidation	60 g H_2O_2/kg TS, 0.07 g Fe^{2+}/g H_2O_2 pH = 3	Reduction of SS:21%, reduction of VSS:25%, increase of SCOD from 0.82 to 7.8 g/ L	AD, lab-scale, 35°C, 30 d	Increase of methane production from 430 to 496 m^3 CH_4/Mg VS degraded, +3.1 time increased net energy, reduced GHG emissions (0.128 Mg CO_2/Mg of TDS)	[89]
WAS	Alkali	NaOH dosage = 157 g/ kg TS	Pre-treatments reduced the viscosity of the sludge	AD	Increase in methane production = 34%	[90]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	Condition	Outcome	Method	Outcome	
Activated sludge (10.2 mg TS/L)	Peroxide/oxidation	4 g Fe3+/°/kg TS, 40 g H_2O_2/kg TS pH = 3 1 h	DDcod:23.6%, (Fe2+), DDcod:16.7% (Fe°)	AD, BMP, 35°C, 60 d	+30.2%biogas and +38.0% methane production for Fe2+, +24.4% biogas and +26.8% methane production for FeO	[91]
WAS	Fenton	Catalyst iron dosage = 4 g/kg TS H_2O_2 dosage = 40 g/kgTS pH = 3 Duration = 60 min	Sludge disintegration increased up to 23.6%	AD	Total methane production increased = 26.9%	[73]
Activated sludge (10.6 ± 0.1 g TS/L)	Alkaline treatment	pH = 9–11(4 mol/L NaOH) Duration = 24 h	NR	AD, batch,37.0 ± 0.1°C, 25 d	+10.7–13.1% TSS removal, +6.5– 12.8% VSS removal, +7.2–15.4% biogas yield, improved dewaterability	[92]
Anaerobically digested sludge	Acidic treatment	Temperature = 170°C pH = 5–6 (H_2SO_4) Duration = 1 h	NR	AD, continuous, 35°C, HRT 20 d	+2–2.5 times VSS removal, +14–21% methane production, 22–23% better dewaterability	[93]
Activated sludge	Alkaline treatment	Temperature = 130°C pH = 10 (KOH)	DDcod: around 60%	AD, continuous, 35°C, HRT 20 d	+36.4% COD removal, +33% TS removal, +74% biogas production	[94]
WAS	Free nitrous acid-heat pre-treatment	Nitrous acid = 0.52–1.11 mg N/L Temperature = 70 °C	sCOD increased and found between 0.16 and 0.28 mg sCOD/mg VS	AD	Methane production increased by = 17–26%	[78]
Sewage sludge	Ozonation	Ozone intensity = 0.1 g O_3/g COD	Oxidization of organics: 38%, solubilization of organics: 29	AD, batch, 33°C, 30 d	+1.8 times methane yield, +2.2 times production rate, decreased dewaterability	[95]
Activated sludge (11.7 ± 2.3 g TS/L)	Alkaline treatment	8 g NaOH/m_3 wet sludge (pH 8)	SCOD/TCOD: 1.99%	AD, CSTR, 55°C, HRT 21 d	+9.7% TS removal, +11.5% VS removal, +18.1% COD removal, 84.22–78.24 mL/d for biogas (–7.1%)	[96]

Type of sludge	Pre-treatment method			Biological coupling		Ref.
	Method	**Condition**	**Outcome**	**Method**	**Outcome**	
Activated sludge (13.9 ± 0.2 g TS/L)	Peroxide/oxidation	50 mg H_2O_2/g TS, 7 mg Fe/g T Sin sludge pH = 2.0 Duration = 30 min	Increase of SCOD from 8 ± 1 in control to 103 ± 7 mg/g TS (+11.9 times)	AD, BMP, 37 ± 1°C, 23 d	+10% methane production, +13% methane potential but no significant effect on hydrolysis rate	[78]
Biological approach						
WAS	Bacterial enzymaticpre-treatment	Strains = *Bacillus jerish* EDTA dosage = 0.2 g/g SS	Extracellular polymeric substance decrease to 40 mg/L	AD	Suspended solids reduction = 48.5% COD solubilization = 47.3%	[97]
Thicken sewage sludge	Bioleaching-Fenton	Bioleaching = 2 days H_2O_2 dosage= 0.12 mol/L Fe2+ dosage = 0.036 mol/L Duration = 60 min	Volatile solids reduction up to 36.93%. Sludge resistance to filtration was 3.43 × 108 s2/g. Increased dewater ability by 4%	NR	NR	[98]

*NR, not reported; AD, anaerobic digestion; RT, retention time; MW, microwave; WAS, waste activated sludge.

Table 1.
Methods for activated sludge treatment.

reaction of hydroxyl radicals with organic compounds present in WAS. Hydroxyl radicals are highly reactive species and may cause complete mineralization of WAS after oxidation [101]. Ozone forms free radicals on reacting with water and causes hydrolysis of organic matter in WAS enhancing its biodegradability [102]. Chemical pre-treatment is not suitable for easily biodegradable substances containing high amounts of carbohydrates, due to their accelerated degradation and subsequent accumulation of volatile fatty acids, which leads to failure of the methanogenesis [103].

2.3.2 Acid pre-treatment

Acid pre-treatment is done to disintegrate the polymeric structures and cells in WAS which is achieved by the use of reagents such as HCl, H_2SO_4, H_3PO_4, and HNO_2. The pH during the acid pre-treatment ranges from 1 to 5.5. During acid pre-treatment, flocculation is observed near isoelectric point as the lowering of pH causes reaction between hydrogen ions and the ionized carboxyl groups rendering them in unionized forms resulting in the formation of aggregates [104]. Strong acidic pre-treatment may result in the production of inhibitory by-products, such as furfural and hydroxyl-methylfurfural [105]. Hence, strong acidic pre-treatment is avoided and pre-treatment with dilute acids is coupled with thermal methods. Other disadvantages associated with acid pre-treatment include the loss of fermentable sugar due to the increased degradation of complex substrates, a high cost of acids, and the additional cost for neutralizing the acidic conditions before the AD process [106].

2.3.3 Alkali pre-treatment

Alkali treatment is relatively effective in sludge solubilization, within the order of efficacy being highest for NaOH followed by KOH, $Mg(OH)_2$ and $Ca(OH)_2$ [107]. However, too high concentrations of Na^+ or K^+ may cause subsequent inhibition of AD [107]. The increase in pH of WAS due to alkali pre-treatment causes many effects on sludge components which include saponification of lipid bilayer and protein denaturation in the cell membrane, solubilizing EPS by the ionization of its carboxyl and amino groups and hydrolysis of sludge organic substances [99].

An alkali pre-treatment study demonstrated that the best-performing alkali for WAS is NaOH. The results of this study indicated an increase by 39.8, 36.6, 15.3, and 10.8% of the soluble COD (mg/L) for WAS by using NaOH, KOH, $Ca(OH)_2$, and $Mg(OH)_2$, respectively [108]. Using 8% of NaOH, an increase in the methane yield by 81% was observed for pulp and paper sludge [88]. Moreover, these pre-treatment methods were further studied with the pH range between 4 and 11 [109]. The results indicated that acidic pre-treatment was less effective than the alkali pre-treatment method for soluble COD in short-chain fatty acids from excess sludge. The main disadvantage of this pre-treatment includes additional pH adjustment need of this pre-treatment for AD which increases operational cost and also increases environmental concerns due to additional chemical agents.

2.3.4 Oxidation

The COD removal during AD was enhanced through oxidation at 90°C with 2 gH_2O_2/g VSS (volatile suspended solids) but not by the oxidation at 37°C [110]. Moreover, post-treatment on the recirculation loop, treating 20% of the sludge stream, was more efficient than a configuration with pre-treatment. However, the process consisting of one anaerobic digester, high-temperature oxidation and a

second digester led to the highest removal of fecal coliforms [110]. Fenton reaction involves the decomposition of hydrogen peroxide in the presence of ferrous ions as the catalyst to form hydroxyl radicals [111]. The hydroxyl radicals thus formed are highly reactive free radical species that oxidize organic matter in sludge further enhancing WAS biodegradability and dewatering [112]. Besides catalyst and hydrogen peroxide, pH during the reaction is also a very crucial parameter to be maintained during Fenton oxidation as the catalytic activity of ferrous ions is lost at pH > 4 [113]. Hence, an effective Fenton oxidation involves adjustment to acidic pH values, oxidation, neutralization, and separation of by-products [114]. Use of Fenton catalyzed oxidation (0.067 g Fe(II)/g H_2O_2, and 60 g H_2O_2/kg TS) decreased sludge resistance to dewatering in terms of capillary suction time, but did not have a positive effect on sludge dewatering performance on a belt press simulation [115].

2.3.5 Ozonation

Ozonation depends upon the oxidation reaction of hydroxyl radicals with organic compounds present in WAS. Ozone forms free radicals on reacting with water and causes hydrolysis of organic matter in WAS enhancing its biodegradability [102]. The pH of the system is reduced after ozonation because ozone degrades higher molecular weight organic compounds into simpler acidic compounds like carboxylic acid [11]. Ozone is a strong oxidant, which disintegrates itself into radicals and reacts with organic substrates [116] in two ways; the direct reaction depends on the structure of the reactant, whereas the indirect reaction is based on the hydroxyl radicals. As a result, the recalcitrant compounds become more biodegradable and accessible to the anaerobic bacteria [117]. Prior to ozone treatment, the methane production was observed to be 440.3 ml CH_4/g VS and after applying ozone doses of 0.034 g O_3/g TS, 0.068 g O_3/g TS, 0.101 g O_3/g TS, and 0.202 g O_3/g TS increased by 35.2, 46.4, 32.9, and 22.2%, respectively [118]. Several ozonation pre-treatment systems are commercially available in the market. They include the Aspal SLUDGE™ and Praxair® Lyso™. The former offers high dewaterability and low energy consumption, and the latter achieves 80% sludge reduction and 75% reduction in ozone use with increasing dewaterability of sludge [112].

2.3.6 Temperature phased AD (TPAD)

Temperature phased AD (TPAD) occurs in two phases. In the primary hydrolytic/ acidogenic phase, 45–70°C temperatures for 2–6 d is applied whereas the second phase is the methanogenic or acetogenic phase for which temperature favorable to thermophilic microorganisms is provided for 14–30 d. The effects of Maillard reaction have not been reported during TPAD which may be due to increased activity of hydrolytic enzymes or defense created by microorganisms through enzymes suppressing the effects of Maillard reaction products [119]. An additional acidification step decreases the amount of polyelectrolyte required to dewater the digestate since poor dewaterability is observed in the acidogenic effluent.

3. Other different pre-treatment methods

3.1 Thermochemical pre-treatment

Integration of pre-treatment methods has also been studied for sludge stabilization to further improve AD and biogas production. A combination of thermal and chemical pre-treatment methods is known to improve the degradation of volatile

solids and biogas production [108]. Thermal-alkaline pre-treatment of sludge was reported to cause floc disintegration, cell disruption and reduction in organic sludge components with high increase in sludge pH to 13 [120]. The authors also reported about 100 times increase in SCOD of sludge as compared to raw untreated sludge [120]. The improvement in reduction of volatile solids as a result of thermochemical pre-treatment enhanced two times high reduction in volatile solids than that in control when sodium hydroxide was combined with thermal pre-treatment at 121°C [108]. In a similar study, 72% enhancement in volatile solids removal and biogas production was observed when sludge was pre-treated at 170°C and pH 12 [94]. The improved content of soluble COD after thermochemical pre-treatment plays role in increasing the efficiency of AD with biogas production increased to 52.78% [2]. Chemical pre-treatment of carbohydrates and proteins can increase their hydrolysis into sugars and amino acids, respectively, and these later products react with each other through Maillard reaction at high temperature resulting in high molecular weight polymers like melanoidins. In another study, a high 78% biogas production with 60% methane was obtained after thermochemical pre-treatment at a lower temperature of 70°C [121].

Microwave-alkaline pre-treatment is another integrated technology for sludge pre-treatment which improves the efficiency of AD. Microwave irradiation coupled with alkaline pre-treatment of sludge improved the volatile solids reduction by 35% and methane formation by 53% as compared to control [122].

4. Feasibility of a full-scale application

With an ever-increasing concern for the environment, different pre-treatment methods can enhance the AD performance. Nevertheless, the high capital cost, high consumption of energy, required chemicals, and sophisticated operating conditions (maintenance, odor control, etc.) are the major factor hindering their full-scale application [123]. There are only a few examples of the thermal hydrolysis process (THP) that have been applied at full-scale such as the Cambi, Porteous, and Zimpro process and thermochemical pre-treatment methods such as Synox, Protox, and Krepro. It should be noted that these methods are all applied for WWTP sludge. Concerning the organic fraction of municipal solid wastes, only a few mechanical pre-treatment methods such as Cambi THP and AD with a pre-hydrolysis stage (two-stage AD) have been applied at a full scale.

4.1 Energy balance

The required energy depends on the desired pre-treatment temperature. If it is above 100°C, most of the energy is utilized in water vaporization, thus making it less desirable [124]. Microwave heating provides direct heating from the inside and therefore unlike conventional heating strategies, negligible or no heat losses are reported [125]. However, neither microwave nor ultrasound were found to be energy-intensive for pretreating mixed sludge, as the enhanced methane yields were not enough to compensate for the required energy [126]. The total biogas obtained after thermal pre-treatment of sludge is relatively higher than other methods and thus costs could be compensated by utilizing the extra biogas through an efficient heat exchanger [127]. A better energy balance was estimated while treatment of organic fraction of municipal solid waste in two-stage AD systems where the authors observed higher energy potential to be associated with not the first stage hydrogen production but to the higher performance in the methanogenic reactor [128]. The energy efficiency of a two-stage AD reactor for sewage sludge in which excess energy of 2.17 kJ/d was obtained was higher when compared to a single-stage

system [129]. It was further concluded that the energy balance can be enhanced by 18.5% if the two-stage AD process is optimized.

4.2 Economic feasibility

Estimated net profit of various pre-treatments (low-temperature thermal pre-treatment not included) to enhance the biogas production of food waste obtained the best result (10–15 euro/ton FW) with less energy-intensive methods (acid and freeze-thaw) [130]. The estimation of the economic feasibility of pre-treatment methods based on a full-scale application has only been reported for WWTP sludge. The operational and maintenance cost of a full-scale AD (3300 m^3) treating 380 m^3 sludge per day based on the application of focused-pulsed pre-treatment technology could generate a benefit of 540,000 USD per year [72]. An approximate cost estimate associated with pre-treatment methods was suggested in research which included capital, operational, and maintenance costs between 70 and 150 US \$/ton sludge [131]. In another cost estimate study, comparative costs for sludge pre-treatment methods were calculated to improve the process of AD of sludge where the authors estimated costs associated with microwave pre-treatment, conventional thermal pre-treatment, ultrasound, and chemical pre-treatment methods as 0.0162, 0.0187, 0.0264, and 0.0358 US\$/m^3, respectively [125]. The comparative cost analysis suggested microwave and conventional pre-treatment methods to be cheaper than ultrasonic and chemical pre-treatments.

The amount of sludge for pre-treatment is also an important factor to consider when estimating the pre-treatment cost. Pre-treatment strategies such as ultrasound can prove to be energetically acceptable for a large scale application if 6 kWh energy value is considered for each cubic meter of sludge [124]. If higher energy is required, biological pre-treatment such as adding hydrolytic bacteria could be a cheaper option [132]. The extent of net economic benefit also depends upon other factors besides the type of pre-treatment method and quality and quantity of sludge. Other parameters to be considered include treatment capacity, availability of labor, the cost associated with collection and transport, taxes and tariffs, energy prices, price of land selected for setup, costs for additional mixing and pumping requirements, the market value of end product as well as waste and residue disposal [133].

5. Conclusion

The pre-treatment methods have the potential to solubilize complex sludge components which include the organic matter, EPS, and the microbial cell wall which in turn makes the progression of subsequent biological degradation treatments easier. AD is the sustainable process widely employed for bioenergy generation from the WAS. Further pre-treatment enhances the methane percentage in the biogas thus the process is energy efficient and sustainable. To attain a clear understanding of the mechanism behind each method, the focus should be given to the conversion strategy and structural alterations that occur in complex WAS components upon the application of each pretreatment technology. Many physical, chemical and biological pretreatment methods have been mentioned in the literature that has been used individually but each of them owns certain disadvantages. These limitations range from high energy requirement of microwaves to excess degradation and fermentable sugars loss in acid pre-treatment. Using two pre-treatment methods in combination has known to overcome these problems, reaping the efficiency of both methods simultaneously. Thus combing pre-treatment process and AD will lead to sustainable process for sludge management.

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