AN EXECUTIVE REVIEW OF SLUDGE PRETREATMENT TECHNIQUES

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ABSTRACT

Anaerobic digestion of sludge has been an efficient and sustainable technology for sludge treatment but the low microbial conversion rate of its first stage requires sludge pretreatment, such as biological (aerobic, anaerobic conditions), thermal, mechanical (ultrasonication, lysis-centrifuge, liquid shear, grinding), and chemical (oxidation, alkali, acidic pretreatment, *etc.*) techniques. This work aims at presenting a review and a short comparison of these common sludge pretreatment techniques, serving the selection of the most suitable technique for lab scale research and for subsequent actual application.

Keywords: anaerobic digestion; waste activated sludge; sludge pretreatment; biological pretreatment; thermal pretreatment;

1. INTRODUCTION

Sludge treatment aims at removing organic materials and water, consequently reduces the volume and mass of sludge and degradable materials, and then odors and pathogens. Incineration, ocean discharge, land application and composting are the common sludge treatments used over the years but no longer sustainable due to the economic difficulties and their negative impacts on environment. Therefore, *anaerobic digestion* (*AD*) of sludge has applied as the efficient and sustainable technology for sludge treatment thanks to mass reduction, odor removal, pathogen decrease, less energy use, and energy recovery in form of methane.

However, the low rate of microbial conversion in the hydrolysis stage (the first stage of *AD* process) requires *the pretreatment of sludge* that ruptures the cell wall and facilitates the release of intracellular matter into the aqueous phase to accelerate biodegradability and to enhance the *AD*. Figure 1 shows the process flowchart of sludge processing steps.

There are some very popular techniques used for sludge disintegration such as biological, thermal, mechanical, and chemical pretreatments. The objective of this work is to present an executive review and a short comparison of common sludge pretreatments, serving the selection of the most suitable technique for lab scale research and for subsequent actual application.

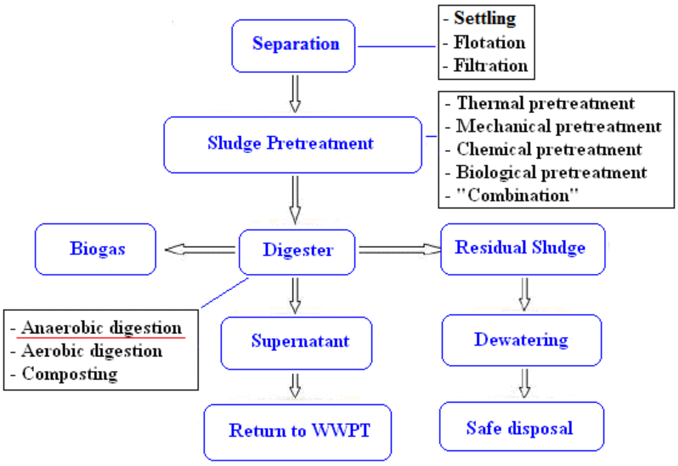

Figure 1. Process flowchart of sludge processing steps [1].

2. SLUDGE TYPE

It was proven that sludge characteristics and microbial kinetics of sludge degradation are the most important parameters influencing the *AD* performance. Five main categories of sludge considered for *AD* are presented as follows: (a) organic fraction of municipal solid waste, (b) organic waste from the food industry, (c) energy crops or agricultural harvesting residues, (d) manure, and (e) sludge from wastewater treatment plants (*WWTP*) [2]. Figure 2, presenting the collection of pretreatment techniques and sludge types, shows sludge from *WWTP* to be the most common object for studying on pretreatment applications and divided into 3 main sludge types as described in figure 3.

Primary sludge is produced through the mechanical wastewater treatment process. It occurs after the screen and the grit chamber and includes untreated wastewater contaminations. The sludge amassing at the bottom of the primary clarifier is also called primary sludge. It is decay-able and must be stabilized before being disposed off. The composition of this sludge depends on the characteristics of the catchment area. Primary sludge is easily biodegradable since it consists of more easily digestible carbohydrates and fats (faeces, vegetables, fruits, textiles, paper, *etc.*). Biogas therefore is produced more easily from primary sludge but the methane proportion in the gas is small.

Activated sludge comes from the secondary wastewater treatment. In the secondary treatment, different types of bacteria and microorganisms consume oxygen to live, grow and multiply to biodegrade the organic matter. The resulting sludge from this process is called activated sludge, consisting largely of biological mass, mainly protein (30%), carbohydrate (40%) and lipids (30%) in particulate form [3]. Normally, a part of the activated sludge is returned back to the system called returned activated sludge and the remaining is removed at the bottom of secondary clarifier called excess sludge, or secondary sludge, or waste activated sludge (*WAS*). Overall, the sludge is the same properties but different name regarding to their usage. Activated sludge contains large amount of pathogens and causes odor problem, thus it has to be stabilized. Besides, activated sludge is more difficult to digest than primary sludge and identified as a low biodegradability sludge, which explains the interest in *WAS* pretreatment applications.

Digested sludge is the residual product after anaerobic digestion of primary and activated sludge. The digested sludge is reduced in mass, less odorous, and safer in the aspect of pathogens and more easily dewatered than the primary and activated sludge.

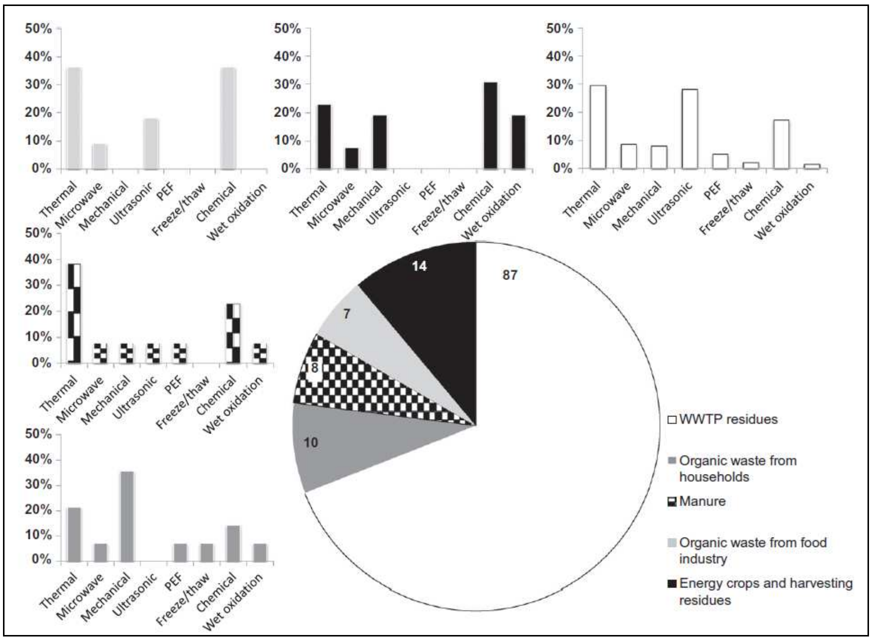

Figure 2. Collection of pretreatment techniques and sludge types [2]. The pie-chart corresponds to the number of times each sludge type occurs in combination with a pretreatment. The bar-charts present the distribution among the different pretreatments for each type of sludge.

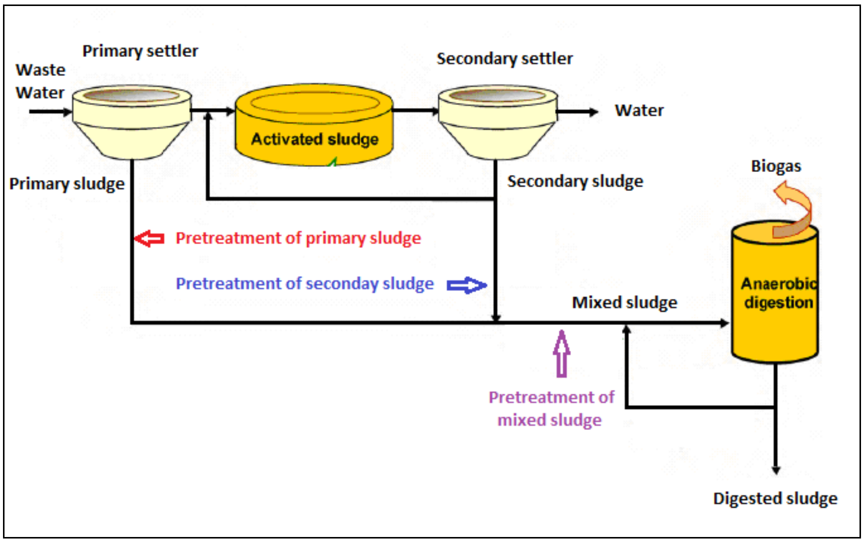

Figure 3. Sludge sources from classical wastewater treatment plants [4].

3. MAIN EFFECTS OF PRETREATMENTS ON SLUDGE

According to Carlsson *et al.* [2], the main effects of pretreatments on sludge could be listed as (i) particle size reduction, (ii) solubilisation, (iii) biodegradability enhancement, (iv) formation of refractory compounds and (v) loss of organic material.

Particle size reduction has been used to describe the effect of pretreatment on sludge (the increase in sludge surface area), but challenged by difficulties in quantifying the shape of particles, and any effects on increased inner surface as on increased particle porosity without overall particle size modification remains unaccounted for by this factor. Therefore, this parameter may misrepresent the effect of pretreatment on the actual surface area for some materials, such as fibrous materials subjected to shear forces, which may be damaged, increase in their surface area without decrease in their particle size. Moreover, this parameter may be only based on the distribution of particles remaining after pretreatment without accounting for the solubilised material.

Solubilisation has been analysed and calculated by various ways, most commonly based on chemical oxygen demand (*COD*) measurements (before and after pretreatment) followed by total solids (*TS*), volatile solids (*VS*) or organic compositions (proteins, carbohydrates, and lipids). Generally, these soluble concentrations after pretreatment are compared to either the (total, particulate, or soluble) concentrations or the ‘‘maximum hydrolysable’’ concentrations of the raw sludge. However, the definition of soluble fraction is not always specified: soluble fraction has been either measured directly in the supernatant after centrifugation (without filtration) or separated from total sample or from supernatant after centrifugation by filtration using different membrane filters (materials and pore sizes).

Biodegradability often represents the amount of material that can be biologically converted into methane by *AD*, thus it includes the concept of bioavailability [2]. Under pretreatment, mechanical or physical-chemical effects cause sludge disintegration, solubilisation and/or chemical transformation; consequently sludge biodegradability could be changed. The exposure of biodegradable matters previously unavailable to microorganisms and the alteration of the composition of hardly degradable compounds lead to an increase in biodegradability. Biodegradability is commonly evaluated through biochemical methane potential (*BMP*) tests (known as an approximate indicator) and expressed as accumulated methane volume produced per unit of *TS, VS* or *COD* input. It is important to note that inoculum quality and testing duration for *BMP* tests significantly affect the total biodegradability and also the biodegradability enhancement.

The correlations between biodegradability enhancement and particle size reduction and solubilisation are ambiguous: positive (strongly correlated), lacking, or even negative. As mentioned, the efficiency of a pretreatment heavily depends on sludge type and characteristics, where the solubilised material is inherently easily biodegradable, the effect on biodegradability enhancement may be limited. In some cases, that sludge biodegradability decreases after pretreatment may be caused by the **formation of refractory/toxic compounds** and **removal of organic material.** For examples, lignocellulosic biomass pretreatment results in the formation of furfural, hydrolymethylfurfural (*HMF*), and soluble phenolic compounds, or Maillard reactions of sludge containing proteins and carbohydrates results in the formation of melanoidines, or removal of organic material results in a net decrease of organic material available for methane production.

4. BIOLOGICAL PRETREATMENT TECHNIQUES

Biological pretreatments have a wide range of processes that comprise of both aerobic and anaerobic processes, and can be applied in the excess sludge destruction process, or biological pretreatment prior to *AD*. This technique disintegrates sludge with enzymes (external enzymes, enzyme catalyzed reactions and autolysis processes for cracking cell wall compounds) or without enzymes [5].

Aerobic or anaerobic digestion of *WAS* is often slow due to the rate limiting cell lysis step. Several systems combining biological and physical-chemical treatments have been studied in order to improve the aerobic/anaerobic biodegradation [6]. Yamaguchi *et al.* [7] suggested a two-step pretreatment system with a biological reactor consisting of sludge degrading microorganisms. First step was alkali pretreatment that increased the pH above 9. Consequently, sludge was introduced into biological degradation reactor where sludge was further degraded to simple molecules and pH became appropriate for further digestion.

4.1. Aerobic pretreatment

In order to improve the degradation of recalcitrant organic matter, aerobic pretreatments have been applied because there are materials that can be degraded under aerobic, not anaerobic conditions [8].

Aerobic hyper-thermophilic pretreatment: Hyper-thermophilic aerobic microbes are protease-excreting bacteria, presented in untreated sludge, and can survive under anaerobic mesophilic conditions. The potential for increase in performance thus is inherent in sludge itself [9]. An increase of 50% in biogas production was observed using a hyper-thermophilic aerobic reactor as the first stage of a dual process (with *AD* as the second stage) [10].

Another term is *co-treatment process*, aiming at enhancement of the main *AD* processes by altering physical or chemical properties, improvement of degradability (subsequently enhance gas production and anaerobic digester performance), allowance of process intensification with faster kinetics (provide the same performance in a smaller digester and decrease hydraulic retention time - *HRT*) [4].

Aerobic thermophilic co-treatment: The process includes two different stages: a biological wastewater treatment and a thermophilic aerobic digestion of the resulting sludge. A part of returned sludge from the wastewater treatment step is injected into a thermophilic aerobic sludge digester (*TASD*) to be solubilized by thermophilic aerobic bacteria. The solubilized sludge is then returned to the aeration tank in the wastewater treatment step for its further degradation. Destruction of 75 % organic solids from waste activated sludge was obtained at full scale (65 °C, *HRT* of 2.8 day) [11].

Aerobic hyper-thermophilic co-treatment (Figure 4): A combination of a Mesophilic Anaerobic Digesters - *MAD* (*HRT* of 21 and 42 days) and hyper-Thermophilic Aerobic Reactor - *TAR* (65 °C, *HRT* of 1 day) increased the intrinsic biodegradability between 20 and 40 % [12]. The *MAD/TAR* model increased *COD* release by 30 % for *HRT* of 42 days. However, this amount of *COD* was oxidized in the aerobic stage, and consequently the methane production yield was not improved. Besides, the degraded *COD* with 21 days *HRT* for the *MAD/TAR* mode was equal to that with 42 days *HRT* for conventional *MAD*, which indicates that the *MAD/TAR* reduces the *HRT* or digester volume by half. An increase in soluble mineral fraction release (from 6 % to 10 %) was also observed [12].

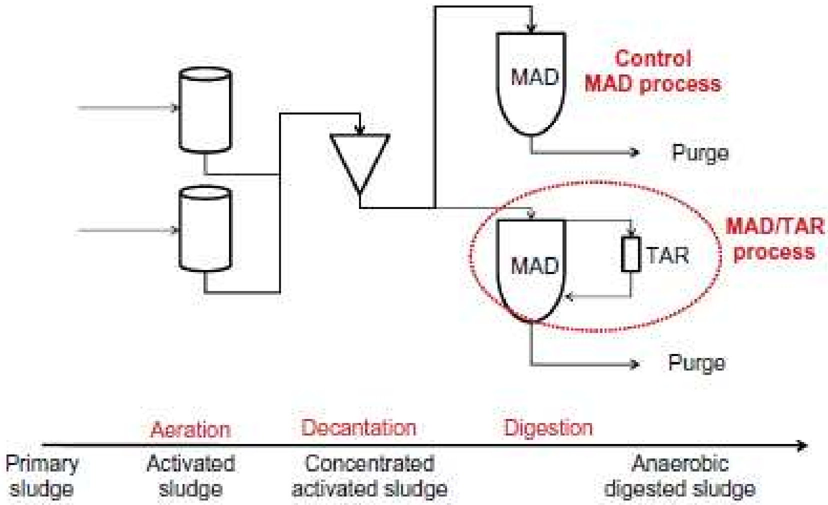

Figure 4. Aerobic hyper-thermophilic co-treatment [12].

An industrial process combined with the aerated sludge process, Biolysis® E, is being commercialized by Ondeo-Degremont (Suez), resulted in 40 – 80 % reduction of excess sludge production, without deteriorating the wastewater quality [13]. Thickened sludge is introduced in a thermophilic reactor where enzymes (proteases, amylases, lipases) are produced by specific microorganisms (Bacillus stearothermophillus).

4.2. Anaerobic Digestion

Anaerobic digestion is a favored stabilization method compared to aerobic digestion, due to its lower cost, lower energy input, and moderate performance, especially for stabilization [14]. The *AD* of sludge is a complex and slow process requiring high retention time to convert degradable organic compounds to CH_4 and CO_2 (a renewable energy source helping replace fossil fuels) in the absence of oxygen through four stages, namely, (1) Hydrolysis, (2) Acidogenesis, (3) Acetogenesis, and (4) Methanogenesis (figure 5). There are three different groups of bacteria in this process. (1) *Hydrolytic and acidogenic bacteria* hydrolyze the complex substrates (carbohydrates, lipids, proteins, etc.) to dissolved monomers (sugars, fatty acids, amino acids, etc.) and further to CO_2, H_2, organic acids and alcohols. (2) *Acetogens* include *Hydrogen producing acetogens* converting the simple monomers and fatty acids to acetate, H_2, and CO_2 and *Homoacetogens* converting H_2 and CO_2 to acetate. (3) *Methanogenic bacteria* utilize the H_2, CO_2 and acetate to produce CH_4 and CO_2.

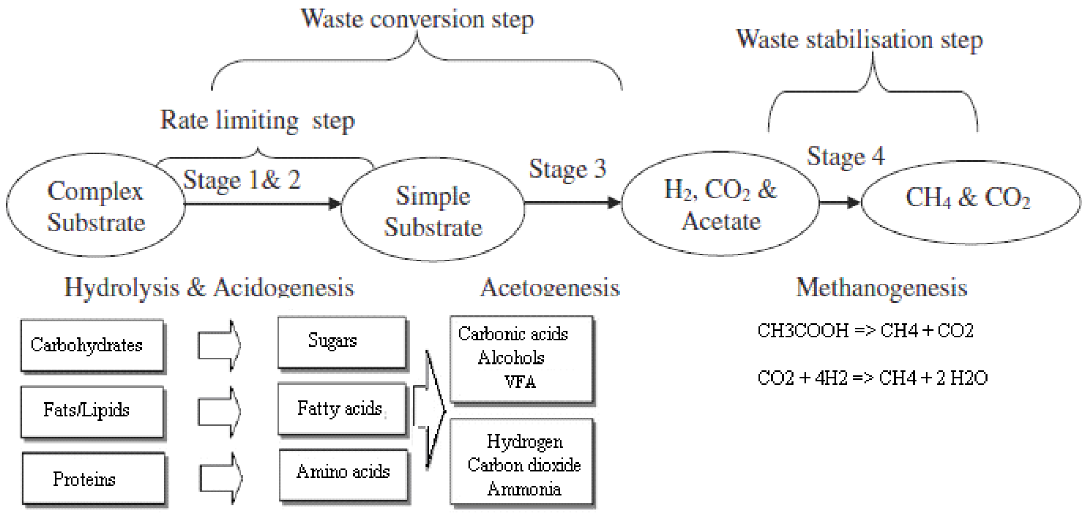

Figure 5. The main stages in anaerobic digestion process [15].

Since methane formers (last group of microorganisms in mechanism) are quite sensitive to environmental conditions, *AD* process requires strictly control of environmental conditions during operation. Factors affecting anaerobic digestion process are presented in *table 1*.

Table 1. Factors in anaerobic digestion [16].

Physical factors	**Chemical factors**
Temperature	pH
Hydraulic Retention Time	Volatile Acids
Solids Retention Time	Alkalinity
Solids loading	Nutrients
Volatile Solids Loading	Toxic compounds
Mixing	Trace elements
Solids Concentration	
Sludge Type	

Temperature: It is a main factor for monitoring anaerobic digester. Microorganisms normally grow faster at higher temperature leading to digest much organic matters. The organic substances therefore can be decomposed and more biogas was produced, even faster by thermophilic AD (50 – 60 °C) than by mesophilic condition (30 – 38 °C). Because of more energy consumption for temperature control, very sensitive of methanogenic bacteria to temperature variation (< 0.5 °C), and comparable biogas yield to mesophilic, thermophilic is not economical. Mesophilic thus has been selected and operated at 35 - 37 °C. Besides, the two-stage *AD* with thermophilic and mesophilic digestion and proper retention time gave the best results [17, 18].

Table 2. Comparison of mesophilic and thermophilic conditions.

Parameter	**Mesophilic**	**Thermophilic**
Temperature	20 – 45°C	> 45°C
Residence Time	15 – 30 days	10 – 20 days
Benefits	- More robust and tolerance process - Less sensitive to the temperature change (within 2°C) - Less energy consumption due to low temperature supplied	- High gas production - Faster throughput - Short residence time - Small digester volume - High organic loading rate
Limitations	- Low gas production rate - Large digester volume - Long residence time	- Need effective control - Very sensitive to temperature change (<0.5 °C) - High energy consumption

Hydraulic Retention Time (*HRT*) & Solids Retention Time (*SRT*): *HRT* represents the time spent in a reactor of a water molecule. *SRT* represents the ratio of mass of solids in the reactor to mass of solids wasted daily. For a single stage or high rate conventional anaerobic digester (with no recycle), *HRT* is equal to *SRT*. ***SRT = V/Q*** where *V* is working volume of the reactor (mL), *Q* is sludge flow or loading rate (mL/day). According to Vesilind [19], typical *SRT* value for mesophilic *AD* lies between 10-20 days. Meanwhile, digestion at 35°C requires minimum *SRT* of 4 days [20]. Therefore, general approach is determining the minimum *SRT* by using growth rate of microorganisms and choosing afterwards a larger *SRT* value to be on the safe side [21]. Longer retention time leads to the decrease in specific gas production [22]. In other word, higher effects on methane production were achieved with short *HRT* of *AD* (an increment in *VS* removal by 12% and 88% compared to that of the control corresponding to 7 days and 2 days of *HRT*, respectively) [23], indicating an acceleration of *AD* as the main effect of pretreatment.

Organic Loading Rate (*OLR*): The *SRT*, *HRT*, volume, and solids concentration determine the solids loading to the digester, including the amount of feed sludge that microorganisms must stabilize and the time for stabilizing this sludge. Microorganisms growth and stabilization rate are main factors that determine the maximum loading rates. Due to degradable properties, biologically volatile solid (*VS*) reduction (depending on sludge type digested, temperature, and *OLR*) is commonly used to assess the performance of anaerobic digestion processes. It is well known that the *OLR* is one of the most important factors to control *AD* systems: $\boldsymbol{OLR = C_{in} * V_{in} / V}$ where C_{in} is influent *VS* concentration, V_{in} is influent feeding volume per day and *V* is working volume of the reactor. Typical range of *OLR* is 1.0 – 5.0 kg_{COD}/m^3*d [24], or 0.64 – 1.60 kg_{VSS}/m^3*d for low rate and 2.40 – 6.40 kg_{VSS}/m^3*d for high rate digesters [25]. An important advantage of *AD* is the ability of stabilizing stronger organic loads; higher efficiencies therefore are expected when increasing *OLR* [26].

Mixing plays an important role in *AD* by preventing the settlement and the formation of scum, providing effective contact between food and microorganisms, and facilitating the release of biogas. Mixing is necessary for preventing temperature grading and stratification that limit the digestion performance. Ineffective mixing reduces the active volume of a reactor, consequently *SRT* decreases and washout becomes a potential problem.

pH, Volatile Acids, and Alkalinity: These three factors and their effects on *AD* are interdependent, hence should be considered together. pH drop is the major risk due to faster growth rate of acetogenic bacteria and the increase in volatile acids concentration (*VFAs*). *VFAs* are important intermediary compounds in the metabolic pathway of methane fermentation. In high concentrations, *VFAs* cause microbial stress and finally lead to failure of the digester [27-29]. The main acids are acetate, propionate, and n-butyrate [30]. The ratio of propionic acid to acetic acid can also be used as an indicator of digester imbalance. The acetic acid level in excess of 800 mg/L or a propionic acid to acetic acid ratio greater than 1.4 indicated digester failure [31]. Besides, alkalinity plays an important role of neutralizing VFAs in order to maintain the optimum pH range of 6.8 - 7.2 for methanogenesis that is extremely sensitive to both high and low pH methane-forming microorganisms. Some optimum values or ranges could be listed such as pH 6.4 - 7.5 [32], pH 6.5 - 8 [33, 34], pH 6.5 - 7.2 [35], pH 7 - 8 [36], and pH 6.5 – 7.6 [37], *etc.*

Nutrient: Sufficient amount of nutrients such as nitrogen and phosphorus are required for an efficient *AD* due to production of microbial cell. The amount of each nutrient required is directly proportional to the amount of microorganisms grown. Overall, the optimum C/N ratio for *AD* is about 20 - 30.

Toxicity: The *AD* is sensitive to certain compounds including sulfides, volatile acids, heavy metals, calcium, sodium, potassium, dissolved oxygen, ammonia and chlorinated organic compounds [38]. The inhibitory concentration of a substance depends on many variations, including pH, organic loading, temperature, hydraulic loading, the presence of other materials, and the ratio of the toxic substance concentration to the biomass concentration.

As mentioned, biological pretreatment aims at intensification by enhancing the hydrolysis step in an additional stage prior to the main digestion process. The most common type is *temperature phased anaerobic digestion* at either thermophilic (55 °C) or hyper-thermophilic (60 – 70 °C) conditions.

Anaerobic thermophilic pretreatment (Figure 6): There are some modes, such as short pretreatment prior to mesophilic digestion (Two-Stage, Thermophilic-*MAD*) [17, 18, 39], single-stage digesters [40]. Thermophilic conditions generally increased organic solids destruction rate, subsequently increased hydrolytic activity. An increase of 25 % on methane production and solids destruction (for primary sludge) was observed under thermophilic compared to that under mesophilic pretreatment (*HRT* of 2 days) prior to *MAD* (*HRT* of 13–14 days) [41]. Ge *et al.* [41] indicated that the performance improvement was due to an increase in hydrolysis coefficient rather than an increase in inherent biodegradability.

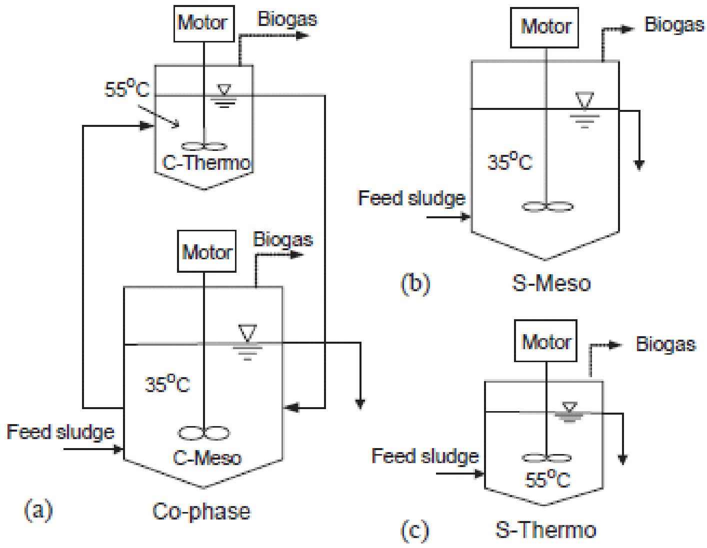

Figure 6. Anaerobic thermophilic pretreatment: (a) The temperature co-phase *AD* system; (b) The single-stage *MAD*; (c) The thermophilic AD processes [17].

Anaerobic hyper-thermophilic pretreatment: Increased temperature biochemical pretreatment enhances pathogen destruction [42 - 44], and hydrolysis rates as well. Higher temperatures might reduce the effectiveness and increase energy costs. With anaerobic hyper-thermophilic pretreatment (70 °C), the increased biodegradable *COD* content was 15 – 50 % depending on sludge characteristics: primary sludge [45], secondary sludge [46 - 48] or mixed sludge [49, 50].

One of the most significant elements, related to environment and finance, is energy. In general, energy utilized should match the energy produced by increased biogas production. Energy consumption in anaerobic digesters is electrical and thermal. *Electrical requirements* are mainly feed and mixing (about 0.1 – 0.2 kWh/m^{3}d) [24, 51]. *Heating requirements* are thermal capacity along with about 10 % or 20 % losses in mesophilic or in thermophilic, respectively [24]. Generally, mesophilic and thermophilic pretreatments produce an adequate thermal energy and an excess of electrical energy. Only thermophilic systems in cold climate or with poorly degradable feeds are difficult to produce sufficient energy for self-heating [52].

5. MECHANICAL PRETREATMENT TECHNIQUES

Among mechanical pretreatments, secondary sludge ultrasonic pretreatment has been focused with a large number of scientific researches. Other mechanical pretreatments, such as centrifugation, grinding, high-pressure pretreatment, have been applied to large particle size materials (energy crops/harvesting residues and organic waste from households, *etc.*) [2].

5.1. Lysis-centrifuge, Grinding, and Liquid shear techniques

Lysis-centrifuge operates directly on the thickened sludge stream in a dewatering centrifuge [53]. It is then suspended again with the liquid stream. The increase of biogas production is 15 – 26 %. This technique has been conducted in some *WWTP* as a pretreatment for *AD*: Liberec (100,000 person equivalent (PE), Czech Republic), Furstenfeldbruck (70,000 PE) and Aachen-Soers (650,000 PE) in Germany [54].

Grinding (by stirred ball mills) is more effective on digested sludge (increase of batch biogas production by 60 %) and on *WAS* from an extended aeration process (24 % increase) than on activated sludge with a higher *SRT* (7 % increase) [23, 55].

Liquid shear (such as *Collision plate* and *High-pressure homogenizer*) depends on high liquid flows thanks to a high-pressure system to disrupt mechanically cells and flocs. For *collision plate,* sludge is pressurized to 30 – 50 bar by a high-pressure pump and jetted to the collision plate through a nozzle. This process (a rapid depressurization with high flow velocities of 30 – 100 m/s) has only been applied at laboratory scale and decreases *HRT* (from 14 to 6 day) without affecting *AD* performance [56 - 57]. For *high-pressure homogenizer*, sludge is pressurized up to 900 bar then goes through a homogenization valve under strong depressurization [58]. This process has been tested at full-scale for *AD*. A part of digested sludge was treated at 150 bar and returned to the digester, leading to an increase of 30 % in biogas production and a reduction of 23 % in sludge volume [59], but a decrease in sludge dewaterability [60]. Several other (de)pressurization-based processes are commercially available, such as The Crown® process (Biogest company), with operation at 12 bar in several full-scale implementations [61], Cellruptor or Rapid non-equilibrium decompression, RnD® process (Ecosolids) [62], and Microsludge® process (Paradigm Environmental Technologie Inc), applied in Los Angeles *WWTP* [63]. For RnD® process, that sludge is pressurized higher than 1 bar allows a gas (soluble in sludge stream) to go through cell walls due to its rapid rate of diffusion. The gasified sludge stream is then depressurized (a rapid non-equilibrium decompression), subsequently causes extremely high shear rates and cell rupture, consequent particle size reduction, the interstitial water release, and biogas production increase (0.3 – 0.816 m^3/kg_{VS}) [62]. For Microsludge® process, chemicals are applied first (pH 11 or pH 2) to weaken sludge cell walls. A high-pressure homogenizer at 830 bar then causes cell disruption. Pretreated *WAS* is introduced in a digester together with primary sludge, with a ratio 68/32 (w/w). The degradation of mixed sludge is increased by 50 – 57 % [63].

5.2. Ultrasonic pretreatment technique

The mechanisms of ultrasonic sludge disintegration are (a) Hydro-mechanical shear forces created by cavitation, (b) Oxidizing effect of $^{\cdot}OH$, $^{\cdot}H$, $^{\cdot}N$, and $^{\cdot}O$ produced under the ultrasound radiation, (c) Thermal decomposition of volatile hydrophobic substances in the sludge, and (d) Increase in temperature during ultrasonic activated sludge disintegration. It was proved that sludge disintegration is mainly caused by hydro-mechanical shear forces and by the oxidizing effect of $^{\cdot}OH$, but mostly in the former process [15, 64]. The ambient conditions of the reaction system can significantly affect the intensity of cavitation; consequently affect the efficiency (rate and/or yield) of reaction. Different conditions resulted in different effectiveness of sludge ultrasonic pretreatment. Main parameters effecting cavitation include ultrasonic frequency, power input, intensity, and specific energy input (*ES*), temperature, hydrostatic pressure, stirrer type and speed, and sludge characteristics (sludge type, pH, total solid content *TS*, etc.).

As cited by Pilli *et al*. [15]**,** *ultrasonic irradiation (US)* is a feasible and promising mechanical disruption technique for sludge disintegration and microorganisms' lyses according to the treatment time and power, equating to specific energy input. Several positive characteristics of this method are efficient sludge disintegration [15], improvement in biodegradability and biosolids quality [3], increase in biogas/methane production [65 - 67], no chemical additives [68], less sludge retention time [69], and sludge reduction [67].

Ultrasonic pretreatment is very effective in **particle size reduction of sludge**. The mean particle size reduction increases with the increase in *US density* [15], 60 % and 73 % at 2 W/mL and 4 W/mL, respectively [68], or 61 %, 74 %, and 82 % corresponding to 0.18 W/mL, 0.33 W/mL, and 0.52 W/mL, respectively [70], indicating that sludge disintegration efficiency also increases at higher *US* densities. In addition, sludge particle size reduces very fast owing to the increase in *US duration* [69 - 72], especially in the initial period of ultrasonic process, and much faster than *COD* release in the aqueous phase. On the other hand, although this reduction accelerates the hydrolysis stage of sludge *AD* and enhances degradation of organic matters, the findings of Le *et al*. [72] indicated this parameter not to be convenient for process optimization.

Under *US*, **sludge mass reduction** is happened and usually measured by the decrease in the suspended solids (*SS*), *VS*, *TS*, or total dissolved solid (*TDS*) concentrations. During *US* (0–30 min), *SS* reduction, and *VS* reduction increase were almost linear with *US* duration, indicating the continuous and stable sludge floc disintegration, mass reduction, and cell lysis [80]. Besides, the solubilisation of *TS* (S_{TS}) increased linearly following an increase in *ES* (from 3600 to 108000 kJ/kg_{TS}) and reached 14.65 % at ES_{max}. Meanwhile, the *VS* solubilisation (S_{VS}) initially fast increased in the *ES* range of 0 - 31500 kJ/kg_{TS} (reached 15.8 %) and then slowed down at higher *ES* values (reached 23 % at ES_{max}) [81]. In terms of sludge disintegration, S_{VS} was proportionally more important than S_{TS} [81, 82]. Moreover, Feng *et al*. [74] found the *TDS* also increased (2.9 - 45.8 %) with an increase in *ES* (500–26000 kJ/kg_{TS}).

In terms of **sludge dewaterability, t**he capillary suction time (*CST*) and the specific resistance to filtration (*SRF*) tests are both commonly used to estimate. In one hand, the enhancement level of dewaterability depends on *ES*, *US* duration, and sludge volume [33]. The *CST* of sludge decreased at lower P_{US} and *US* duration because the flocs did not reduce their sizes, but with an increase in *US* duration at the same P_{US}, the *CST* value increased [71]. Na *et al*. [76] found that an increase in *US* doses (0-above 2000 kJ/L) leaded to a decrease in *CST* (from 53s to under 10s), implying ultrasonic treatment of *WAS* improved the dewaterability. According to Li *et al*. [84], sludge dewaterability will increase when the degree of sludge disintegration (DD_{COD}) is 2 – 5 % because floc structure has a limited change at DD_{COD} of less than 2 %, the number of fine particles in bound water increases at DD_{COD} of 6 – 7 %, and sludge particle size significantly decreases at DD_{COD} of more than 7 %. In the other hand, sludge dewaterability decreased gradually with an increase in *US* duration [73, 83, 85], *US* density [15, 73]), *ES* [83, 86], cell lysis and release of biopolymers from extracellular polymeric substances (*EPS*) and bacteria into aqueous phase [15, 85], and a decrease in free water of the sludge [85].

The settleability of sludge is inversely proportional to the degree of sludge disintegration under *US*. Sludge settleability changed with an increase in *ES* (increased after the first hour but decreased thereafter), in which the optimum *ES* for improving *WAS* settleability was 1000 kJ/kg_{TS} [74]. *WAS* settleability was improved at *ES* of less than 1000 kJ/kg_{TS} because of the slight flocs disruption; on the contrary, the settleability deteriorated at *ES* of more than 5000 kJ/kg_{TS} [74] due to the complete breakdown of flocs and increase in *EPS* concentration in the liquid phase. However, Chu *et al*. [73] indicated that ultrasonic treatment has no effect on sludge settleability that contradicts recent research results about the changes in particle size and floc structure [74, 76].

The turbidity of sludge increased due to the increase in *ES* and particle size reduction during disintegration [75]. The supernatant turbidity of pretreated sludge decreased at *ES* of less than 5000 kJ/kg_{TS}. However, it increased significantly at *ES* greater than 5000 kJ/kg_{TS} due to the release of micro-particles from sludge flocs into supernatant, which settle very slowly [74]. Therefore, the minimum *ES* required to disrupt sludge flocs and/or to release large amounts of organic matters was 1000 kJ/kg_{TS} [71, 74].

US has considerable effect on **microbial disruption** which leads to the changes of floc density, particle size, turbidity, settling velocity, and filterability, but still unclear about the efficiency of the disruption [15]. According to Dewil *et al.* (2006) cited by Pilli *et al.* [15], *US* pretreatment reduces average size of flocs and creates the bulk of separate cells and short filaments pieces (Actinomyces). In addition, the flocs and cell wall will be completely broken down with the increase in *US* duration [73, 87]: after 60 min of sonication [73]. However, Feng *et al.* [74] found that even at high level of *ES* (26000 kJ/kg_{TS}), neither the floc structure nor the microbial cells were totally disintegrated (because there was still a network of filamentous bacteria in the photomicrographs of the treated sludge).

Both cellular or extracellular matter and organic debris or *EPS* of sludge are disintegrated by *US*, leading to the solubilisation of solid matters and the increase in organic matters/*EPS* concentrations in aqueous phase, consequent the increase in ***SCOD* of sludge** [75, 80, 86, 88, 89], **protein, polysaccharide, *DNA*, Ca^{2+}, and Mg^{2+} levels** [85, 86], and *AD* performance [90]. The increase in proteins slowed down after longer *US* duration while polysaccharide and *DNA* concentrations dropped after 20 min of sonication [86]. Among those components, the level of released protein was the highest in the aqueous phase of sonicated sludge. This predominance of proteins may be due to large quantities of exoenzymes in the floc: the ratio of protein to polysaccharide was about 5.4 [74].

Besides, **Organic nitrogen and ammonia** concentrations in sludge samples increased owing to the increase in *ES* and *TS* content of *WAS* [65, 74, 91]. The bacterial cells were disintegrated and the intracellular organic nitrogen was released in the aqueous phase, which was subsequently hydrolyzed to ammonia, resulting in the increase in ammonia-N concentration [91].

The breakdown of bacterial cell walls because *US* can be evaluated based on Oxygen Utilization/Uptake Rate (*OUR*). In general, sludge microbial activity decreased when DD_{COD} increased during ultrasonic sludge treatment [84]. The survival ratio (ratio of viable bacteria density levels after *US* to those of original sample) of the heterotrophic bacteria decreased owing to the increase in *US* duration [73]. Zhang *et al.* [80] suggested the hypothesis as follows: sludge disintegration and cell lysis occurred continuously during sonication but *sludge inactivation* occurred mainly in the second stage (10–30 min) [80]. Inactivation of sludge (biomass inactivation) depends on *US* duration. It occurred after 10 min of sonication [80] and after 20 min of sonication using low *US* density [73], which indicated that *US* density is also a parameter affecting on inactivation of sludge. Besides, Li *et al*. [84] indicated two main stages of ultrasonic sludge pretreatment process: (i) sludge flocs were changed and disintegrated at first, and then (ii) the exposed cells were disrupted. *In the first stage*, some organic matters in the flocs were dissolved and *SCOD* increased slightly. At the same time, *SOUR* was increased due to the enhancement of oxygen and nutrients consumption. *In the second stage*, some cells were exposed and damaged by ultrasonic cavitation, leading to the release in intracellular organic matters, the further increase in *SCOD*, and the significant decrease in *SOUR*. Due to the heterogeneity of sludge and the differences in the external resistances of many types of zoogloea and bacteria, activation and inactivation took effects at the same time and the comprehensive effectiveness was under the influence of various ultrasonic parameters.

6. THERMAL PRETREATMENT

While the carbohydrates and the lipids of the sludge are easily degradable, the proteins are safe from the enzymatic hydrolysis by the cell wall. Heat provided during thermal pretreatment destroys the chemical bonds of the cell wall and membrane, thus makes the proteins accessible for biological degradation [1]. In addition, this pretreatment allows a high level of solubilisation, modification in sludge characteristics (increase in dewaterability and viscosity reduction), and reduction of pathogens. Two main temperature brackets, either higher or lower than 150 °C and high enough pressures to prevent evaporation, can be considered for economic or efficiency point of views.

In terms of pretreatment conditions, most studies have reported 160 – 180 ^{o}C of temperature, 600 to 2500 kPa of pressure associated to these temperatures, and 30-60 min of pretreatment time to be optimum values [92]. However, temperature has more impact on sludge solubilisation than duration of pretreatment [6, 93, 94]. On the other hand, thermal pretreatments at moderate temperature (70 °C) may last several days because the main mechanism in such case is assumed to be enzymatic hydrolysis [46, 49].

*For heating equipments***,** thermal pretreatment can be carried out either with direct steam/vapor injection [95, 96], or autoclave or microwave heating (electric heating) [97], or water bath heating [98]. Some industrial processes (conducted at 150 – 180 °C during 30 – 60 min by vapour injection) have been commercialized. For example, *Cambi*, at *HIAS WWTP* of Hamar-Norway from 1995 for 90,000 *PE*, results in an increase in the electric production by 20 % [95]. *BioTHELYS®* has been implemented at the urban *WWTP* of Saumur for 62,000 *PE* and 1400 ton *TS*/year of sludge since 2006, resulted in 46% of sludge volume reduction; or at Château Gontier for 38,000 *PE* and 1000 ton *TS*/year of sludge [96]. Some positive results from more than 10 installations were an increase in biogas production and reduction of organic matter around 60 %, a reduction of sludge volume, an average increase in digester capacity with organic loading of 5.6 kg *VS*/m^{3}day [99]. The interests of sludge thickening before thermal pretreatment and the recovery of heat from hot streams in order to reduce energy requirements have been underlined [100].

Some advantages of thermal pretreatment could be listed as follows*:* to degrade sludge gel structure, reduce sludge viscosity, improve sludge dewaterability after treatment at 150 – 180 °C [101-103], increase hydrolysis rates [97, 104, 105], decrease HRT [106], guarantee sludge sanitation, limit energy input [95], solubilize partial of sludge, enhance *AD* [101, 107, 108], and increase methane production. The increase of methane production is related to sludge *SCOD* by linear correlations [109]. Conversely, Dwyer *et al.* [110] found that elevating temperature above 150 °C increased solubilisation, but did not increase methane conversion. Moreover, pretreatments at excessively high temperatures, higher than 170 – 190 °C, lead to the decrease in sludge biodegradability in spite of achieving high solubilisation efficiencies. This is usually attributed to the so-called Maillard reactions [110], involving carbohydrates and amino acids in the formation of melanoidins, which are difficult or impossible to degrade [108]. Melanoidins also increase the color from the anaerobic digester, subsequently increase color in the final effluent [110]. In general, thermal pretreatment of *WAS* can considerably increase methane production with respect to *MAD* but a lesser extent was obtained when combining to thermophilic *AD* (thermophilic digestion is already more efficient at *VSS* reduction and methane production as compared with mesophilic digestion, hence reduces benefits of pretreatment) [1].

On the other hand, *disadvantages of thermal pretreatment* are to increase largely soluble inert fraction and final effluent color [110], as well as ammonia inhibition in the main digester due to increased performance [111].

According to Carlsson *et al*. [2], **freeze/thaw pretreatment**, whose mechanism relies on freezing sludge from between -10 and -80 °C with thawing afterwards, has been applied to a much lesser extent than other thermal pretreatments.

7. CHEMICAL PRETREATMENT

Chemical pretreatments mainly consist of oxidative treatments and acids/alkalis addition and may be conducted with increased temperatures (known as thermo-chemical technique).

7.1. Oxidation

Wet oxidation has been applied to sewage sludge, with the solubilised fraction subsequently treated in a *UASB* reactor [104, 112]. Besides, **Fenton catalyzed oxidation** (0.067 $g_{Fe(II)}/g_{H2O2}$, and 60 g_{H2O2}/kg_{TS}) also decreased sludge resistance to dewatering in terms of *CST*, but did not have a positive effect on sludge dewatering performance on a belt press simulation [113]. **Hydrogen peroxide (H_2O_2)** has also been used as an oxidant [114, 115]. The *COD* removal during *AD* was enhanced by 2 g_{H2O2}/g_{VSS} at 90 °C, but not at 37 °C [114]. Moreover, *post-treatment* (90 °C, 2 g_{H2O2}/g_{VSS}, 30 days of *SRT*) on the recirculation loop, treating 20 % of the sludge stream, was more efficient than a configuration with *pretreatment* (90 °C, 2 g_{H2O2}/g_{VSS}, 30 days of *SRT*). However, the process consisting of one anaerobic digester (15 days of *SRT*), high temperature oxidation (90 °C, 2 g_{H2O2}/g_{VSS}) and a second digester (15 days of *SRT*) led to the highest removal of fecal coliform (figure 7) [114].

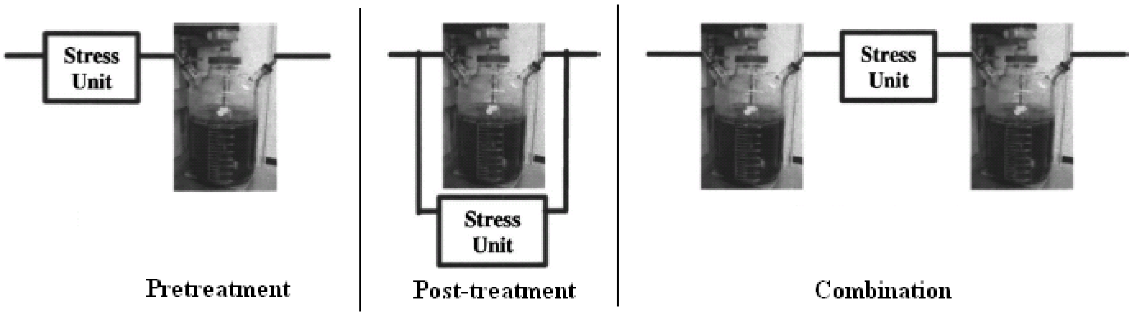

Figure 7. Oxidation pretreatment using hydrogen peroxide oxidant [114].

The most cost-effective and widely used chemical pretreatment technique with the highest disintegration capability is **ozonation,** [116], and an attractive pretreatment procedure for solid hydrolysis prior to aerobic/anaerobic digestion [6]. Ozone is a strong cell-lytic agent, which can kill microorganisms in activated sludge and further oxidize the organic substances released from the cells [117 - 118]. Following ozonation, the characteristics of the sludge are greatly changed. The floc is broken, generating a large number of microparticles dispersed in the supernatant apart from soluble organic substances [119]. Sludge disintegrated by ozonation is therefore well described by the sequential decomposition processes of floc disintegration, solubilisation, and mineralization. In other hand, nitrogen and SS concentrations in the effluent slightly increased although it remained under authorized limits.

Ozonation treatment has two opposite effects: (1) degradation of molecules and cell structures that are undegradable for methanogen will increase biogas production; (2) oxidation of organic molecules that are degradable for methanogen will decrease biogas production [120]. Saktaywin *et al*. [117] found around 60 % of *SCOD* generated due to ozonation to be biodegradable at the early stage of ozonation, while the remaining soluble organic matter was refractory. According to Weemaes *et al*. [121], the biogas production increased by 80% at $0.1g_{O3}/g_{COD}$ of ozonation; higher ozone doses, although still positive, were found to have a less pronounced effect. The biodegradation was also found to increase with ozone dose up to 0.2 g_{O3}/g_{SS} but further increase in ozone dose did not improve the biodegradation [122]. Ozone dose therefore heavily affects sludge biodegradation.

Sludge ozonation was first used in combination with activated sludge process for wastewater treatment [123, 124]. Chu *et al*. [119] have recently proposed a review of concerned studies (figure 8). Ozonation has also been combined with *AD* as a pretreatment [121,122,125] or post-treatment and recycling back to the anaerobic digester [126, 127]. Better performance and lower ozone consumption in the case of post-treatment and recycling in the digester were achieved [126]. The Japanese Kurita company, Ondeo-Degremont (Suez): Biolysis® O process [128] have commercialized this process and about 30 installations have been implemented [121].

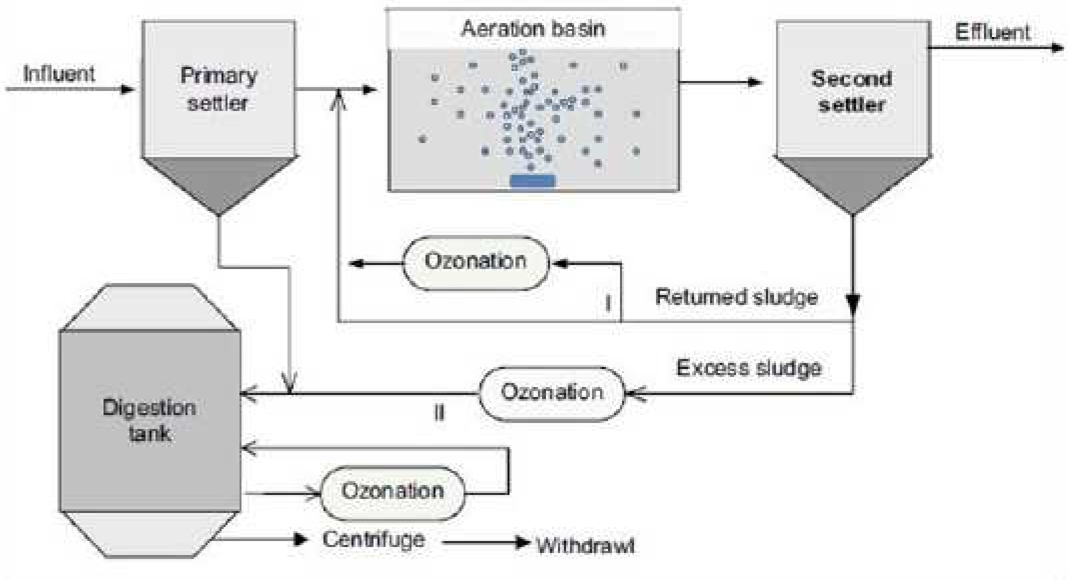

Figure 8. Application of ozonation for sludge disintegration [119].

7.2. Alkali treatments

According to Pilli *et al.* [15], the effects of sonication parameters and sludge properties on solubilisation of *COD* can be rated as follows: sludge pH > sludge concentration > ultrasonic intensity > ultrasonic density. This suggests that pH adjustment to a suitable value prior to *US* pretreatment is an important step.

Alkaline pretreatment enhanced sludge solubilisation, anaerobic biodegradability, as well as methane production [33, 115]. Besides, the combination of alkaline and *US* gave better performances of *TS* solubilisation as compared to both thermo-acidic and ultrasonic-acidic pretreatments [130]. Moreover, the combined alkaline-ultrasonic pretreatment released more

COD in solution than the individual pretreatments, due to the complementary effects of hydroxyl anion reactions (solubilizing extracellular polymeric matrix) and mechanical shear force (disrupting flocs and cells). Some synergetic effects were even noticed [131].

The chemicals used for increasing the pH of sludge also affect *WAS* solubilisation and their efficacy is as follows: NaOH > KOH > $Mg(OH)_2$ and $Ca(OH)_2$ [33, 132]. Ca^{2+} as well as Mg^{2+} are key substances connecting cells with extra-cellular polymeric substances (EPS). As a result, their presence may enhance the reflocculation of dissolved organic polymers [132], which leads to a decrease in soluble *COD*. On the other hand, overconcentration of Na^+ (or K^+) was reported to cause subsequent inhibition of *AD* [4].

Chiu *et al*. [133] investigated the hydrolysis rate of alkaline, ultrasonic, chemical-ultrasonic and simultaneous ultrasonic and alkaline pretreatment on *WAS* (1% of TS contend at ambient temperature). Three sets of experiments were designed and conducted: (i) pretreated with 40 meq/L NaOH for 24 h, (ii) pretreated with 40 meq/L NaOH for 24 h followed by US for 24 sec/mL, and (iii) simultaneous ultrasonic (14.4 sec/mL) and chemical (40 meq/L NaOH) pretreatment. The authors indicated the initial hydrolysis rate of the third approach was the highest (211.9 mg/L*min). Moreover, this approach could shorten the *WAS* pretreatment time and resulted in a prolific production of *SCOD*. The second approach was more effective in *SCOD* release and soluble organic nitrogen compared to the first one but to be closed to the third one.

Jin *et al*. [132] investigated the effects of combined alkaline and *US* pretreatment of sludge on *AD*. *SCOD* was used as an indicator to evaluate the efficiency of different combinations in pretreatment stage as well as in the subsequent *AD*. *SCOD* levels for combined pretreatment were higher than those for sole ultrasonic or sole alkaline pretreatment. Low NaOH dosage (100 g/kg dry solid), short duration of NaOH treatment (30 min), and low ultrasonic specific energy (7500 kJ/kg dry solid) were proved to be suitable for sludge disintegration. In the subsequent *AD*, the degradation efficiency of organic matter was increased from 38.0% to 50.7 %, which was much higher than that with ultrasonic (42.5%) or with NaOH pretreatment (43.5 %) at the same retention time.

Bunrith [134] compared effects of different (*US*, chemical, and combined) pretreatment techniques on *WAS* disintegration and subsequent *AD* (10, 15, and 25 days of *SRT*). The optimum chemical dose was found at 50 mg_{NaOH}/g_{TS} at short holding time of 6 min since *SCOD* increase started slowing down when higher dose was applied. Chemical-ultrasonic pretreatment, the most effective technique on sludge disintegration, released more *SCOD* at high chemical dose and energy input. The higher efficiency of chemical-ultrasonic is due to the combination effects of hydro-mechanical shear force and OH- radical reaction. Pretreatments enhanced the subsequent anaerobic digestibility of *WAS* with significant high *TS* and *VS* destruction, and biogas production, but no methane improvement in the biogas. The hydrolysis rate for chemical-ultrasonicated sludge was higher than that for ultrasonicated and unpretreated sludge; subsequently the degradation rate was faster than others, which eventually reduce the digester volume for same digestion efficiency. Besides, energy requirement for mixer was found the highest followed by heat loss for maintaining the temperature of the digester. In addition, energy obtained from methane gas from all digesters was sufficient for either heating sludge to meshophilic temperature or supplying to ultrasonic unit at 25 days of *SRT*, but not enough to compensate both energy used for heating sludge and ultrasonic unit. Economic analysis revealed that only control digester at 25 days of *SRT* was economically viable since the income and expense was almost the same. At the same *SRT*, the income of ultrasonic and chemical-ultrasonic digester was less than 30 % compared to expense

Kim *et al*. [131] studied the effects of alkaline (pH 8 - 13), ultrasonic (3750 – 45,000 kJ/kg_{TS}), and combined (alkaline + ultrasonic) pretreatments on sewage sludge disintegration. The authors found that in individual pretreatments, the solubilisation (*SCOD/TCOD*) increase was limited (50 %); however, it reached 70 % in combined method, indicating that high pH levels of sludge played a critical role in enhancing the subsequent *US* pretreatment efficiency. Besides, sludge disintegration (with respect to the variation of pH and *ES*) proportionally increased following the increase in pH (from 8 to 13), but decreased gradually when *ES* values were more than 20,000 kJ/kg_{TS}. Besides, the pretreated sludge (pH 9 + *ES* of 7500 kJ/kg_{TS}) was fed to a 3 L of anaerobic sequencing batch reactor after 70 days of control operation. CH_4 production yield significantly increased from 81.9 ± 4.5 $mL_{CH4}/g_{CODadded}$ to 127.3 ± 5.0 $mL_{CH4}/g_{CODadded}$ by pretreatment. However, about 20 % higher soluble N concentration found in the reactor after anaerobic digestion would be an additional burden in the subsequent nitrogen removal system.

7.3. Acidic Pretreatment

Acidic pretreatment is a rare chemical pretreatment method and is applied by the addition of acid to lower the pH of the sludge.

Sludge cells could be disintegrated by acidic pretreatment [5, 94, 135]. According to Neyens *et al.* [94], the net negative charges on the surface of sludge particles kept them apart. When the pH was decreased down to 2.6 – 3.6, the negative charge on the surface became neutral, the repulsive force between particles consequently decreased down to minimum, and physical stability (such as easy dewatering and flocculation) could be observed. At pH 3, sludge volume could be decreased up to 75 % by dewatering and soluble solids could be increased due to solubilisation of intracellular solids. pH 3 was therefore decided to be the most appropriate pH for acidic pretreatment [94].Chen *et al.* [135] showed that at pH 2.5, the viscosity of pretreated sludge was smaller than that of unpretreated sludge, but the settleability was better. These results indicate that acidic pretreatment favours dewaterability and a physically stable sludge.

Meuner *et al.* [136 showed that rapid hydrolysis of *VSS* through sulphuric acid treatment, due to rapid mineralization of organic portion of sludge. Consequently, the amount of excess sludge was minimized. pH 1.5, 2, 2.5 and 3 were analyzed and maximum *VSS* reduction was observed in the lowest investigated pH value, which consumed the highest amount of acid as expected.

Woodard and Wukash [137] pointed that at room temperature, 4 g_{H2SO4}/g_{SS} consumed a significant amount of SS during 5 minutes of holding time. The reduction of *SS* was 61% mostly independent from the initial solids concentration and temperature. The only parameter significantly effected the solubilisation was found to be the acid dose**.**

Acidic pretreatment was thought to accelerate the hydrolysis step by breaking up the cell walls, mineralization of microbial cells, improve dewaterability, and improve the overall performance of subsequent anaerobic digestion. On the other hand, according to Weemaes and Verstraete [92], only few successful results for acidic pretreatment at ambient temperature were reported. Elevated temperatures create aggressive reaction conditions and enhance the effects of pretreatment. Another negative aspect of acidic pretreatment is the requirement of neutralization for subsequent biological application.

Apul [138] indicated that acidic pretreatment (pH 1.5, 2.5, and 4.5 with 20 min of holding time, in which pH 1.5 seemed to be the best condition in terms of cell disintegration and solubilisation) had a very low performance compared to ultrasonic pretreatment for enhancing the solubility of sludge. Primary requirement of a pretreatment is the effectiveness of solubilisation prior to digestion; however, acidic pretreatment was not capable of dissolving organic matter effectively.

Combining acidic and mild-sonication pretreatment technique (acidic-ultrasonic pretreatment) was expected to disturb the floc structures and to release organic matters into liquid phase and consequently, decrease the overall consumption of energy and chemical. Additionally, the physical characteristics (such as dewaterability and turbidity) of the pretreated sludge were expected to be much better compared to sole ultrasonic pretreatment. However, the lower the pH value, the worse the solubilization was due to the antagonistic effect of acid on ultrasonic pretreatment. Briefly, the efficacy (in terms of solubilisation of organics) of combination of acidic and *US* pretreatments was better than that of sole acidic pretreatment but worse than that of sole mild-ultrasonic pretreatment [138].

8. COMPARISON OF PRETREATMENT TECHNIQUES

Sludge pretreatment has been dominated by thermal and ultrasonic techniques, followed by chemical pretreatment. The novel techniques of microwave irradiation and pulsed electric fields are at a rather early stage of development [2].

According to Carrère *et al.* [4], the basis of comparison of pretreatment techniques can be divided into a number of different components: Sludge types [4], Treatment effectiveness [2, 4] (Particle-size reduction; solubilisation; biodegradability – rate or extent), Cost of treatment [2, 4] (energy cost, and secondary costs from nutrient release or generation of by-products), Chemical consumption [4] (particularly for oxidative or chemical treatment), pretreatment mechanisms [4].

Bougrier *et al.* [139] compared the effect of *US*, thermal, and ozonation pretreatments on activated sludge prior to batch *MAD* (figure 9). In terms of solubilisation, thermal pretreatment was the most efficient, led to a strong decrease in apparent viscosity, a strong increase in filterability, and an increase in particle diameter. Sonication led to a decrease in particles size, apparent viscosity, and filterability. Ozonation also led to a decrease in apparent viscosity and filterability, but had no effect on particle size. In terms of anaerobic biodegradability**,** all three pretreatments improved biogas production. The enhancement by ozonation (0.10 and 0.16 g_{O3}/g_{TS}; 246 – 272 mL_{CH4}/g_{CODin} against 221 mL_{CH4}/g_{CODin} of the raw sludge) was lower than that by sonication (6250 and 9350 kJ/kg_{TS}; 325 – 334 mL_{CH4}/g_{CODin}) and thermal hydrolysis (170 or 190 ^{o}C; 325 – 334 mL_{CH4}/g_{CODin}). That low enhancement of biogas production by ozonation could be due to inhibitory conditions (to much ozone remained in the soluble phase), the formation of refractory compounds, an unwell-adapted inoculum, or ozone consumption by the reduction of sludge compounds, or due to the initial biodegradability percentage of raw sludge [139]. Meanwhile, *US* pretreatment provided minimal solubilisation of sludge and particle size reduction, but improved biodegradability of the particulate fraction. Thermal pretreatment increased solubilisation, but did not enhance degradability of residual particulates [139]. To sum up, *US* allowed a weak solubilisation of *COD* and a high biodegradability, ozonation allowed a weak solubilisation and a weak biodegradability, and thermal pretreatment allowed a strong solubilisation and a strong biodegradability. Therefore, sonication mainly focused on particles accessibility, whereas, thermal pretreatment mainly focused on compounds solubilisation [139].

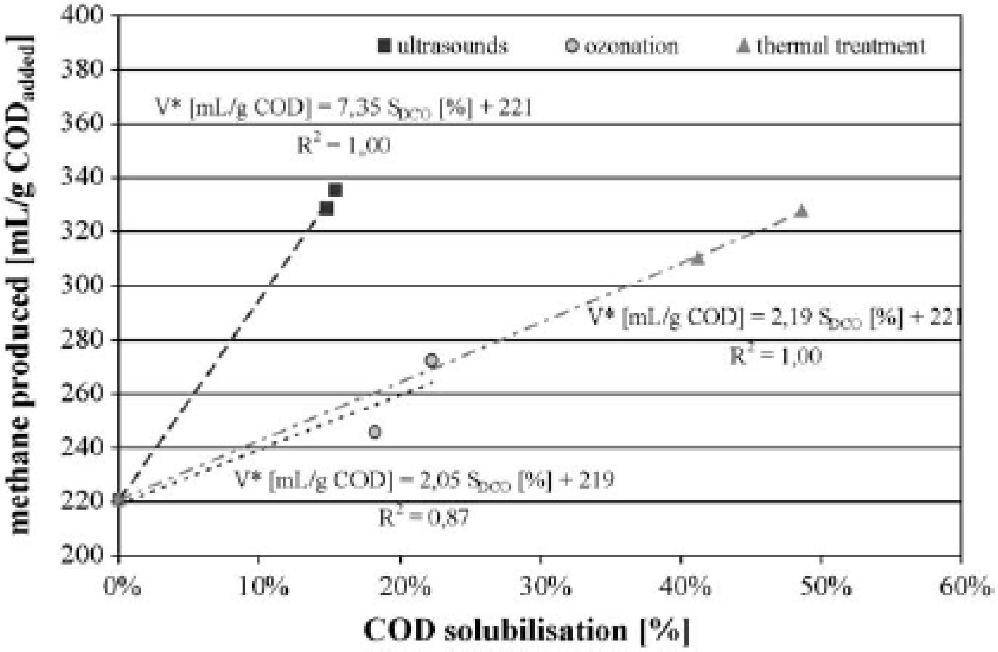

Figure 9. Methane production *vs. SCOD* for different pretreatment techniques [139].

Salsabil *et al*. [6] compared thermal (40, 60, and 90°C within 90 min, 120 °C within 15 min, 1bar), ozonation (0.1g_{O3}/g_{TS}), and sonication (200,000 kJ/kg_{TS}) pretreatments on *TSS* and *VSS* solubilisation, subsequent on batch *AD*. It could be inferred from table 3 that solubilisation could depend on the pretreatment ability rather than on the *ES* to break the flocs (mechanical or chemical effect). Moreover, pretreatments could improve *TSS* reduction and considerably reduce the digestion length afterwards (table 4). The global *TSS* reduction improvement (after pretreatment + digestion) increased with an increase in *TSS* solubilisation (after pretreatment only) whatever the kind of treatment (under both aerobic and anaerobic conditions) (figure 10). *TSS* solubilisation is therefore an interesting parameter to predict sludge reduction improvement [6]. In terms of economic efficiency, based on the exploitation costs with the laboratory scale devices (low energetic performances), Salsabil *et al.* [6] showed the application of a pretreatment before *AD* always led to the cost reduction compare to the control: 44 %, 25 %, and 8 % for sonication, high thermal treatment (90° C and autoclave) and ozonation, and low thermal treatment, respectively.

Table 3. TSS and VSS solubilisation under pretreatments [6].

Solubilisation (%)	*US*	Thermal pretreatment			Ozonation	Autoclave at 121°C
		90°C	*60°C*	*40°C*		
	200,000 kJ/kg_{TS}	558,620 kJ/kg_{TS}	216,000 kJ/kg_{TS}	144,000 kJ/kg_{TS}	46,285 kJ/kg_{TS}	665,024 kJ/kg_{TS}
TSS	46.5	15.8	8.8	5	15	4.2
VSS	55	22.1	11.7	6.5	19.2	4.8

Table 4. Pretreatment *vs.* digestion with respect of sludge *TSS* reduction [6].

	Control	US	Thermal pretreatment			Ozonation	Autoclave at 121°C
			90°C	*60°C*	*40°C*		
Aerobic conditions							
TSS reduction (%)	57-59	76	68	65	62.5	71	69
Part of pretreatment (%)	0	61	23	13.5	8	21	5.5
Part of digestion (%)	100	39	77	86.5	92	79	94.5
Anaerobic conditions							
TSS reduction (%)	66-72	86.2	76.5	73	69.5	78.5	76.9
Part of pretreatment (%)	0	53.5	19.8	12	7.2	19.1	4.4
Part of digestion (%)	100	46.5	80.2	88	92.8	80.9	95.6

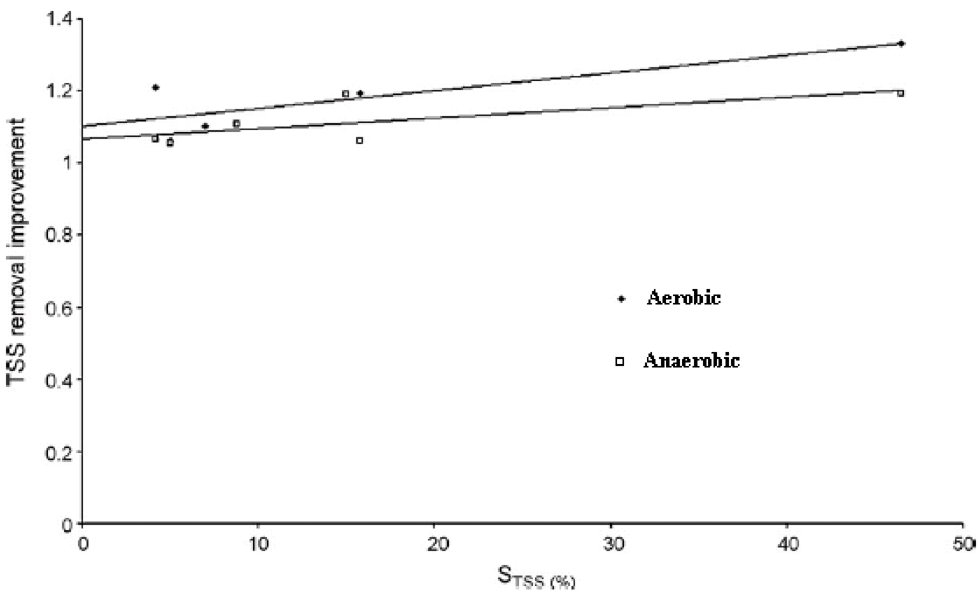

Figure 10. TSS reduction improvement as a function of *TSS* solubilisation [6].

Kim *et al.* [33] compared thermal (121 °C), chemical (7 g/L NaOH), ultrasonic (42 kHz, 120 min) and thermo-chemical (121 °C, 7g/L_{NaOH}) pretreatments prior to batch *AD*. The results were thermal pretreatment (3390 L_{CH4}/m^3_{WAS}) > thermo-chemical pretreatment (3367 L_{CH4}/m^3_{WAS}) > ultrasonication (3007 L_{CH4}/m^3_{WAS}) > chemical pretreatment (2827 L_{CH4}/m^3_{WAS}) > raw sludge (2507 L_{CH4}/m^3_{WAS}).

Barjenbruch and Kopplow [60] compared thermal pretreatment (80, 90, and 121°C for 60min in an autoclave), high-pressure homogenization (*HPH* 600 bar), and enzymatic pretreatment (enzyme *carbohydrase*) prior to continuous *AD* with 10 days of *HRT*. An increase in biogas production was observed in the following order: thermal pretreatment at 90 °C (>21%)

> thermal pretreatment at 121 °C (20 %) > *HPH* 600 (17 %) > thermal pretreatment at 80 °C (16 %) > enzymatic pretreatment (>13 %) (figure 11).

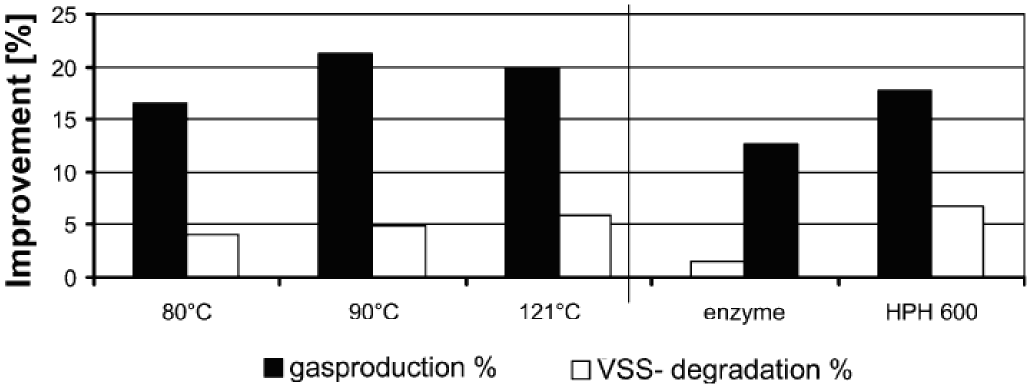

Figure 11. Improvement in anaerobic degradation compared to the control reactor [60].

Yang *et al.* [140] studied thermal pretreatment (200 °C) and wet air oxidation (200 °C, 20 MPa) followed by *AD* of the liquid fraction in a two-stage *UASB* reactor. Although some *COD* was oxidized to CO_2 during pretreatment, wet air oxidation led to better results than thermal treatment: 385 vs. 261 mL_{biogas}/g_{CODin}, 3084 vs. 2775 mL_{CH4}/kg_{WAS}, and a better filterability of the residue.

Muller *et al.* [141] considered a 250,000 *PE* virtual *WWTP* to compare stirred ball milling, ozonation, lysis-centrifuge, and sonication, provided several classifications of pretreatments. Energy demand: ozonation > sonication > stirred ball mill > lysis-centrifuge. Increase of sludge degradation: ozonation > stirred ball mill > sonication > lysis centrifuge. Increase in polymer demand for dewatering: ozonation > sonication > stirred ball mill > lysis-centrifuge. Increase in soluble COD and ammonia concentrations in supernatant after dewatering: ozonation > stirred ball mill > lysis centrifuge > sonication.

Carlsson *et al.* [2] concluded that *particle size reduction* (due to floc structure destruction) is the result of ultrasonic, other mechanical, low temperature thermal and in some cases high temperature thermal and chemical (ozone) pretreatments. However, thermal pretreatment also increases particle size by particle agglomeration due to the creation of chemical bonds under the high temperature [139]. According to Weemaes and Verstraete [92], 100 % cell disintegration can be reached under *US* if the *ES* is high enough. *Sludge solubilisation* (due to microbial cell disruption and *EPS* solubilisation) is caused by all pretreatment techniques. The findings by Appels *et al.*, (2010) cited by Carlsson *et al.* [2] showed that low temperature thermal pretreatment of *WAS* (80 – 90 ^{0}C) can solubilise proteins and carbohydrates, indicating that both cells (rich in proteins) and *EPS* (rich in carbohydrates) are solubilised. Moreover, sludge solubilisation has also increased linearly with temperature up to 200 °C [108]. *Biodegradability enhancement* benefits from most pretreatments, but by different mechanisms, and not in all cases. High temperature pretreatments cause *the formation of refractory substances.* For examples, formation of recalcitrant or even toxic *COD* may occur at temperatures above 165 °C and the *COD* that is solubilised between 140 and 165 °C is not degradable [110]. Besides, *a loss of organic material* has been observed from wet oxidation, high temperature thermal, and freeze/thaw techniques (table 5).

Table 5. Effects of different techniques on sludge pretreatment efficiency [2].

Pretreatment effect	Ultrasonic	Thermal		Microwave	Other mechanical
		(<100°C)	*(>100°C)*		
Particle size reduction	+	+	-/+	na	+
Solubilisation	0/+	+	+	+	+
Formation of refractory compounds	na	0	+	0	na
Biodegradability enhancement	0/+	+	0/+	0/+	+
Loss of organic material	na	na	+	na	na

Table 5. Effects of different techniques on sludge pretreatment efficiency [2] (*cont.*).

Pretreatment effect	Chemical (+/- thermal)			Electric pulses	Wet oxidation	Freeze/Thaw
	Ozone/ oxidative	*Alkaline*	*Acid*			
Particle size reduction	0/+	na	na	na	na	na
Solubilisation	+	+	na	+	+	+
Formation of refractory compounds	+	+	na	na	na	na
Biodegradability enhancement	0/+	-/+	na	+	-	na
Loss of organic material	na	na	na	na	+	+

+ : positive effect   0 : no effect   – : negative effect   na : no information available

The pretreatment effects on WAS have been also compared in terms of *pretreatment mechanisms, energy inputs, and sludge characteristics*. As presented in *table 4*, *the pretreatment mechanism* has been claimed to be more important than the energy input [6]. However, with the same pretreatment technique, effects are often improved following an increase in *energy input*, at least up to a certain level. Related to *sludge characteristics* (primary sludge, *WAS*, or mixed sludge), for examples, the effect of pretreatment on *WAS* depends on the initial biodegradability of the sludge, which in turn depends on the sludge age of the wastewater treatment process. As mentioned, the pretreatment is generally more efficient in enhancing biodegradability when applied to low initial biodegradability sludge, generally corresponding to a long sludge age, even though this might not be reflected on increased solubilisation [22, 108, 125].

Overall, the performance level of each pretreatment technique is reflected in the intensity of treatment. *Lower energy techniques*, mechanical (*US*, lysis-centrifuge, liquid shear, grinding) and biological pretreatments, mainly affect hydrolysis rate with a limited extent (20–30 % improved *VS* destruction). *High impact techniques* -thermal pretreatment and oxidation- have significant improvements of both speed and extent of degradation but with a substantial energy input (figure 12) [4].

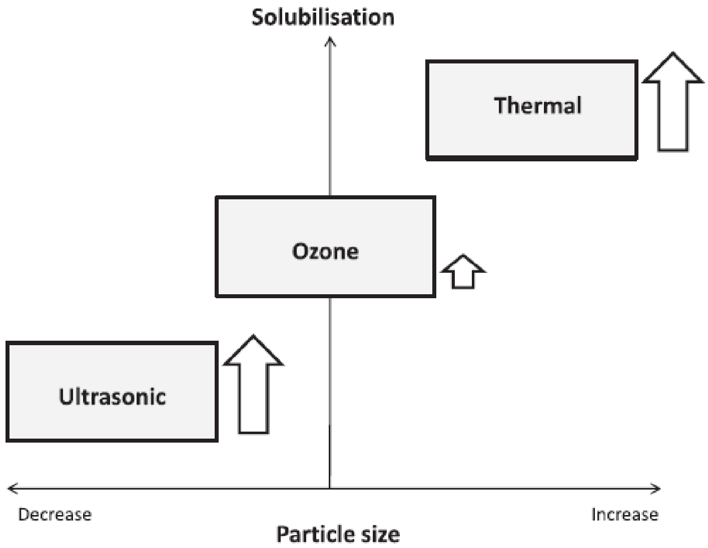

Figure 12. Qualitative pretreatment effects on *WAS* [4]. The arrows indicate the effect on biodegradability that was equally enhanced by *US* and thermal pretreatments and much less by ozonation.

9. CONCLUSIONS

Anaerobic digestion of sludge has been an efficient and sustainable technology for sludge treatment. However, the low rate of microbial conversion of its first stage requires the pretreatment of sludge, such as biological (aerobic, anaerobic conditions), thermal, mechanical (ultrasonication, lysis-centrifuge, liquid shear, grinding), and chemical (oxidation, alkali, acidic pretreatment, *etc.*) techniques.

In terms of efficient operation, pretreatments should be applied to *WAS* (rather than primary or mixed sludge) because the greatest improvement of hydrolysis could be achieved. The fact that pretreatments followed by anaerobic digestion were more effectively than aerobic digestion should be taken into account in actual application. Although the gas produced from pretreated and unpretreated sludge are almost the same at the end of *AD* process, the kinetics of gas production for pretreated sludge is improved, remarkably in the early period of monitoring. Moreover, pretreatments can result in higher methane production regardless of low or insignificantly increased *COD* solubilisation. On the other hand, apart from effects of pretreatment on sludge body disintegration, other counterproductive effects (colorization of effluent, nutrients release, *etc.*) should be taken into consideration.

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TÓM TẮT

TỔNG QUAN CÁC KĨ THUẬT TIỀN XỬ LÍ BÙN THẢI

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Phân hủy yếm khí là công nghệ xử lí bùn hiệu quả và bền vững. Tuy nhiên, tốc độ chuyển hóa chậm của vi sinh vật ở giai đoạn thủy phân đòi hỏi công đoạn tiền xử lí bùn thải thông qua các kỹ thuật như sinh học (hiếu khí, yếm khí), nhiệt, cơ học (siêu âm - *ultrasonication*, li tâm - *lysis-centrifuge*, cắt lỏng cao áp - *liquid shear*, nghiền - *grinding*), và hóa học (oxy hóa, kiềm, axit …). Bài báo nhằm mục tiêu tổng quan và so sánh các kĩ thuật tiền xử lí bùn thải, phục vụ lựa chọn các kĩ thuật thích hợp nhất cho nghiên cứu và ứng dụng thực tế.

Từ khóa: phân hủy yếm khí, bùn hoạt tính, tiền xử lí bùn thải, tiền xử lí sinh học, tiền xử lí nhiệt.