Journal of Hazardous Materials 183 (2010) 1–15

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Review

Pretreatment methods to improve sludge anaerobic degradability: A review

H. Carrère[a,*], C. Dumas[a], A. Battimelli[a], D.J. Batstone[b], J.P. Delgenès[a], J.P. Steyer[a], I. Ferrer[a,c]

[a] INRA, UR50, Laboratoire de Biotechnologie de l'Environnement, Avenue des Etangs, F-11100 Narbonne, France
[b] Advanced Water Management Centre, The University of Queensland, Australia
[c] Environmental Engineering Division, Department of Hydraulic, Maritime and Environmental Engineering, Technical University of Catalonia (UPC), c/Jordi Girona 1-3, Building D1, E-08034 Barcelona, Spain

ARTICLE INFO

Article history:
Received 25 March 2010
Received in revised form 29 June 2010
Accepted 30 June 2010
Available online 7 July 2010

Keywords:
Biosolids
Biogas
Methane
Activated sludge
Pretreatment
Renewable energy

ABSTRACT

This paper presents a review of the main sludge treatment techniques used as a pretreatment to anaerobic digestion. These processes include biological (largely thermal phased anaerobic), thermal hydrolysis, mechanical (such as ultrasound, high pressure and lysis), chemical with oxidation (mainly ozonation), and alkali treatments. The first three are the most widespread. Emphasis is put on their impact on the resulting sludge properties, on the potential biogas (renewable energy) production and on their application at industrial scale. Thermal biological provides a moderate performance increase over mesophilic digestion, with moderate energetic input. Mechanical treatment methods are comparable, and provide moderate performance improvements with moderate electrical input. Thermal hydrolysis provides substantial performance increases, with a substantial consumption of thermal energy. It is likely that low impact pretreatment methods such as mechanical and thermal phased improve speed of degradation, while high impact methods such as thermal hydrolysis or oxidation improve both speed and extent of degradation. While increased nutrient release can be a substantial cost in enhanced sludge destruction, it also offers opportunities to recover nutrients from a concentrated water stream as mineral fertiliser.

Contents

1. Introduction 2
2. Digestion of waste activated sludge 3
3. Biological pretreatment methods 3
4. Thermal hydrolysis (>100 °C) 4
5. Mechanical treatment 6
   5.1. Ultrasonic treatment 6
   5.2. Lysis-centrifuge 7
   5.3. Liquid shear 7
      5.3.1. Collision plate 7
      5.3.2. High pressure homogeniser 8
   5.4. Grinding 8
6. Chemical treatment 8
   6.1. Oxidation 8
   6.2. Alkali treatments 9
7. Comparison of treatment methods 9
8. Energy aspects 11
9. Nutrients issue 11
   9.1. Release of nutrients 11
   9.2. Nutrients removal or recovery 12
10. Conclusions 12
   References 12

* Corresponding author. Tel.: +33 4 68 42 51 68; fax: +33 4 68 42 51 60.
E-mail addresses: carrere@supagro.inra.fr (H. Carrère), dumasc@supagro.inra.fr (C. Dumas), battimel@supagro.inra.fr (A. Battimelli), damienb@awmc.uq.edu.au (D.J. Batstone), delgenes@supagro.inra.fr (J.P. Delgenès), steyer@supagro.inra.fr (J.P. Steyer), ivet.ferrer@upc.edu (I. Ferrer).

0304-3894/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jhazmat.2010.06.129

Nomenclature

AhT	aerobic hyper-thermophilic
BNPR	biological nitrogen and phosphorus removal
COD	chemical oxygen demand
CST	capillary suction time
CSTR	continuous stirred tank reactor
d_b	balls diameter
EPS	extracellular polymeric substances
Es	specific energy
HRT	hydraulic retention time
PE	person equivalent
PS	primary sludge
rpm	round per minute
SRT	solids retention time
TNK	total Kjeldahl nitrogen
TP	total phosphorus
TPAD	temperature phased anaerobic digestion
TS	total solids
TSS	total suspended solids
UASB	upflow anaerobic sludge blanket
v_b	balls velocity
VS	volatile solids
VSS	volatile suspended solids
w/w	weight/weight
WAS	waste activated sludge
WWTP	wastewater treatment plant
X_I	inert fraction
X_P	particulate fraction

1. Introduction

In the field of sludge treatment, the terms pretreatment, cotreatment, disintegration and hydrolysis usually refer to processes which are combined with the main biological sludge treatment process. The objectives of the overall treatment train is to remove organic material and water, hence reducing volume and mass, remove degradable material, which prevents subsequent odours and pathogen vectors, and remove pathogens [1]. Anaerobic digestion is a favoured stabilisation method compared to aerobic digestion, due to its lower cost, lower energy footprint, and moderate performance, especially for stabilisation [2]. Cotreatment processes aim at enhancing the main anaerobic digestion processes by altering physical or chemical properties. The two basic properties that determine sludge behaviour are degradation rate (often defined by a 1st order coefficient), and extent, or conversely, inert fraction [3]. Cotreatment processes may change either property, and can be located in a number of places in the treatment plant (Fig. 1). In the case of main treatment plant enhancement, the main process is manipulated to provide improved degradability. Changes in either kinetic degradation rate or degradability will enhance gas production and anaerobic digester performance. Improving rate can also allow process intensification, with the faster kinetics allowing for the same performance in a smaller digester, and thus decreasing hydraulic retention time (HRT).

Figure 1 shows potential locations for cotreatment in a classical urban wastewater treatment plant. When combined with wastewater treatment processes, cotreatment may be implemented either directly in the aeration tank (T1) or in the sludge recirculation loop, after thickening (T2). Cotreatment in the main water line may aim at either total volume minimisation, or at producing a more degradable material [4]. Direct material pretreatment preceding sludge anaerobic digestion, may be either on primary sludge (T3), to excess waste activated sludge (T4) or to the mix of primary and waste activated sludge (T5). However, since primary sludge is already

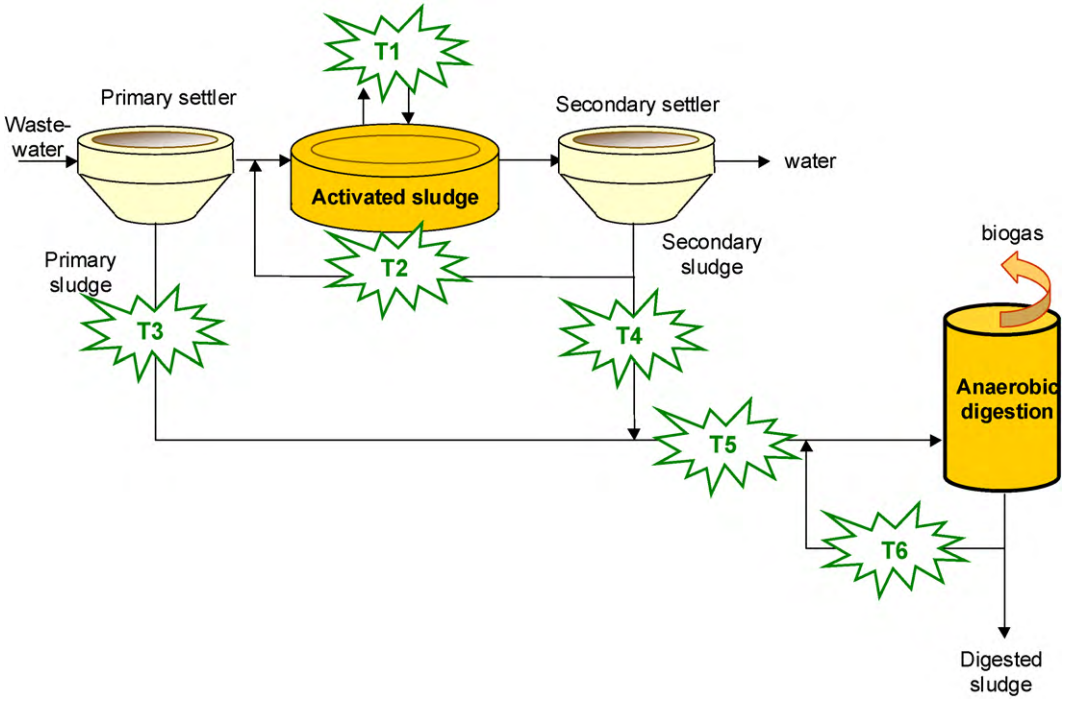

Fig. 1. Potential location for sludge cotreatments in a classical wastewater treatment plant. T1: Cotreatment on activated sludge process. T2: Cotreatment on the activated sludge recirculation loop. T3: Pretreatment of primary sludge before anaerobic digestion. T4: Pretreatment of waste activated sludge before anaerobic digestion. T5: Pretreatment of mixed sludge before anaerobic digestion. T6: Cotreatment on the anaerobic digester recirculation loop.

readily degradable, pretreatment may be less effective [5]. Waste activated sludge however, has relatively low degradability, especially at long sludge ages [6]. In addition, waste activated sludge is generally hydrolysis limited, and can be enhanced by improved rates [7]. Thus, activated sludge pretreatment (T4) is often used in preference to primary sludge pretreatment (T3), except for specific circumstances discussed later. Pretreatment of mixed sludge (T5) may be useful when the treatment also leads to sludge sanitation (for example thermal treatment). Finally, cotreatment can be implemented in the recirculation loop of the digester (T6). This is where degradability of inert or slowly degradable material is to be enhanced, as the digester has already removed readily biodegradable material. This is particularly interesting when cotreatment costs are proportional to the level of organics (for example in the case of sludge ozonation, where the ozone dose is expressed as gram of ozone per gram of total or organic solids in the sludge). A disadvantage is that any active anaerobic biomass will normally be killed by the pretreatment method, and therefore, high circulation rates cannot be used.

Configurations T1 or T2 normally minimise sludge production by either increased sludge age, or increased aerobic destruction of organics. This leads to additional CO_2 emissions, and energy costs. The other configurations are linked with anaerobic digestion. Enabling this technology reduces greenhouse gas emissions and costs compared to aerobic treatment. For this reason, this paper is mainly focussed on the combination of cotreatments with anaerobic digestion (configurations T3 to T6). These pretreatment methods have improved recently in popularity due to a number of factors, including: (i) a trend towards lower nitrogen limits, which is driving up sludge ages and decreasing degradability of activated sludge streams [6], and (ii) increased final handling costs (especially for final destruction options like incineration), and (iii) increased legislative requirements for stabilisation performance and pathogen removal. There is therefore an increased need to review and analyse the different pretreatment options in terms of mechanism, costs, and performance. Here, we review the major classes of biological, thermal, mechanical and chemical pretreatment methods, and evaluate the likely future of pretreatment prior to sludge stabilisation.

2. Digestion of waste activated sludge

For the reasons described above, this paper focuses on the enhancement of anaerobic digestion of waste activated sludge.

The microbial activity (secretion and lysis of cells) during aeration processes in wastewater treatment plants results in a microbial matrix (flocs) composed of microbial and exopolymeric substances [8]. These microbial-originated extracellular polymeric substances (EPS) are a complex mixture of biopolymers comprising polysaccharides, proteins [9–11], nucleic acids, uronic acids, humic substances [12], and lipids, amongst others. EPS is relatively recalcitrant to anaerobic and aerobic digestion by nature. Indeed, various studies have reported EPS as 30–50% biodegradable [13–15]. Specific compounds in EPS are known to be recalcitrant to both aerobic and anaerobic activity [16]. Degradability of activated sludge depends on a number of properties, including whether or not there is a primary settler, the level of inerts coming from the upstream catchment, temperature, aerobic/anoxic fraction, and in particular, the aerobic sludge age [6,17]. Actually, the X_P and X_I fractions in the activated sludge models [18] can generally be used to predict aerobic degradability. In a conventional mesophilic anaerobic digestion process with a HRT of 20 days, conversion of organics to gas is typically 25–60% [19,20], with the lower performance on long-sludge age activated sludges, and higher performance on primary sludges. The goal of pretreatment is to improve this either by increasing the rate, or the inherent degradability of the material. The first is done by increasing rate of the process, which is generally rate-limiting [13,21].

3. Biological pretreatment methods

Biological treatment encompasses a broad range of processes that can include both aerobic and anaerobic processes. These can include in-process excess sludge destruction (configurations T1 and T2 in Fig. 1), or biological pretreatment prior to anaerobic digestion (configurations T3 to T6 in Fig. 1). This section focuses on the latter for the reasons described above.

Biological pretreatment aims at intensification by enhancing the hydrolysis process in an additional stage prior to the main digestion process. The most common type is temperature phased anaerobic digestion (TPAD), which uses a higher stage at either thermophilic (around 55 °C) or hyper-thermophilic (between 60 and 70 °C) conditions, anaerobic and aerobic.

Thermophilic processes and particularly thermophilic hydrolytic activity of bacterial populations have been investigated 80 years ago, mainly at a temperature of 55 °C [22]. There have been a number of configurations tested, including short pretreatment prior to mesophilic digestion [23], dual digesters: thermophilic and mesophilic [24], single stage digesters [25,26] and recently, temperature co-phase processes [27,28]. Thermophilic conditions generally result in an increase of the organic solids destruction rate, attributed to increased hydrolytic activity. Ge et al. [29] evaluated thermophilic against mesophilic pretreatment (HRT of 2 days) prior to mesophilic anaerobic digestion (HRT of 13–14 days) for primary sludge. An increase of 25% on the methane production and solids destruction was observed. Model based analysis indicated that the improved performance was due to an increased hydrolysis coefficient rather than an increase in inherent biodegradability [29]. Elevating temperature above 55 °C did not provide additional benefits. Bioaugmentation by specific thermophilic hydrolytic anaerobic bacteria has been attempted, but not successful [30].

Table 1 summarises biochemical pretreatment methods (Table 1). Elevated temperature biochemical pretreatment allows increased pathogen destruction [31–33], and generally, an increase in hydrolysis rates 70 °C. Higher temperature can reduce the effect, and increase energy costs. With anaerobic hyper-thermophilic pretreatment (70 °C), the increase of biodegradable COD content observed was in the range of 15–50% depending on the characteristics of the sludge: primary sludge [34], secondary sludge [35–37] or a mixture of both [38,39] (Table 1).

In order to improve the degradation of recalcitrant organic matter, aerobic treatments has also been evaluated [40], as there are materials that can be degraded under aerobic, not available under anaerobic conditions [41]. Hyper-thermophilic aerobic treatment is also an option (Table 1). Destruction of 75% organic solids from excess waste activated sludge was obtained at full scale, by combining a conventional municipal activated sludge process with a thermophilic aerobic sludge digester (65 °C, HRT of 2.8 days) [42]. Hyper-thermophilic aerobic microbes were identified as belonging to *Bacillus* with a predominance of *Geobacillus stearothermophilus* [43]. They are protease-excreting bacteria, present in untreated sludge, and can survive under anaerobic mesophilic conditions. Therefore, the potential for increased performance is inherent in the sludge itself [44]. An increase of 50% in biogas production was observed using a hyper-thermophilic aerobic reactor as the first stage of a dual process (with an anaerobic digester as the second stage) [43]. Recently, a combined aerobic hyper-thermophilic (AhT) process (65 °C, HRT of 1 day) coupled to conventional mesophilic digester (HRT of 21 and 42 days) was shown to increase the

Table 1
Biological pretreatment methods.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge	Microaerobic, 60–70 °C, 1 day	Batch, 10 days 37 °C	Increase of biogas production from 200[a] to 300 mL g^{-1} VS_{in} (+50%)	[43]
Activated sludge	Microaerobic 65 °C, 1 day	CSTR, HRT: 21 and 42 days 35 °C	Increase of COD removal (+30%) No methane production increase	[45]
Activated sludge	70 °C 7 days	Batch 37 °C	Increase of CH_4 production from 8.30[a] to 10.45 mmol g^{-1} VS_{in} (+26%)	[35]
Activated sludge	70 °C 7 days	Batch 55 °C	CH_4 production of 10.9 mmol g^{-1} VS_{in} (no influence)	
Primary sludge	70 °C 4 days	Batch 37 °C	Increase of CH_4 production from 21.2[a] to 24.7 mmol g^{-1} VS_{in} (+16%)	
Primary sludge	70 °C 7 days	Batch 55 °C	Increase of CH_4 production from 13.7[a] to 25.5 mmol g^{-1} VS_{in} (+86%)	
Activated sludge	70 °C 2 days	CSTR, HRT: 13 days (15 days without pretreatment) 55 °C	Increase of CH_4 production from 40[a] to 55 mL L^{-1} d^{-1} (+28%)	[47]
Primary sludge	70 °C 2 days	CSTR, HRT: 13 days (15 days without pretreatment) 55 °C	Increase of CH_4 production from 146[a] to 162 mL day^{-1} (+11%)	
Activated sludge	70 °C 9 h	Batch 55 °C	Increase of biogas production +58%	[36]
Mixed sludge	70 °C 9, 24, 48 h	CSTR, HRT: 10 days 55 °C	Increase of CH_4 production from 0.15[a] to 0.18 mL g^{-1} VS_{in} (+20%) Increase of energy production (+60–100%)	[38,39]
Primary sludge	70 °C 2 days	CSTR, HRT: 13 days (15 days without pretreatment) 55 °C	Increase of CH_4 production from 13.6[a] to 20.1 mmol g^{-1} VS_{in} (+48%)	[34]
Primary sludge	50–65 °C 2 days	CSTR HRT: 13–14 days 35 °C	Increase of CH_4 production (+25%) compared to 35 °C pretreatment	[29]

[a] Performance of anaerobic digestion without pretreatment.

sludge intrinsic biodegradability between 20 and 40% [45]. The AhT cotreatment allowed to increase COD removal by 30% for an overall process retention time of 42 days. Nevertheless, this COD was oxidised in the aerobic stage, and therefore the methane production yield was not improved. Compared to conventional mesophilic digester the same quantity of COD was degraded with AhT treatment at 21 days HRT than without AhT treatment at 42 days HRT. Therefore, the AhT treatment enables to reduce the HRT or digester volume by half. Increase in the release of soluble mineral fraction (from 6% to 10%) was also observed [45].

An industrial process combined with the aerated sludge process, Biolysis® E, is being commercialised by Ondeo-Degremont (Suez) [46]. Thickened sludge is introduced in a thermophilic reactor where enzymes (proteases, amylases, lipases) are produced by specific microorganisms (*Bacillus stearothermophillus*). According to the company, this process allows from 40% to 80% reduction of excess sludge production, without deteriorating the wastewater quality.

4. Thermal hydrolysis (>100 °C)

Thermal hydrolysis was first applied to improve sludge dewaterability [48]. It allows degradation of the sludge gel structure and release of linked water. This improves sludge dewaterability after treatment at 150 °C [66] or 180 °C [67]. Thermal hydrolysis leads to partial solubilisation of sludge, which enhances anaerobic digestion, as can be seen in numerous studies on thermal hydrolysis for pretreatment of anaerobic digestion [48,50,68] reported in Table 2. Most studies report an optimal temperature in the range of 160–180 °C and treatment times from 30 to 60 min (Table 2). Pressure associated to these temperatures may vary from 600 to 2500 kPa [69]. However, treatment time is often shown to have little effect at this temperature range [70]. Dohanyos et al. [55] proposed a very fast thermal treatment at 170 °C, lasting only 60 s. On the other hand, thermal treatments at moderate temperature (70 °C) may last several days [35,38], because the main mechanism in such a case is assumed to be enzymatic hydrolysis as described in the previous section.

The increase of methane production has been linked to sludge COD solubilisation by linear correlations [71]. Conversely, Dwyer et al. [72] found that while increasing temperature above 150 °C increased solubilisation, no increase in methane conversion was observed. Treatments at excessively high temperatures (higher than 170–190 °C) lead to decreased sludge biodegradability in spite of achieving high solubilisation efficiencies. This is usually ascribed to the so called Maillard reactions [72], involving carbohydrates and amino acids in the formation of melanoidins, which are difficult or impossible to degrade [68]. Melanoidins also increase the colour from the anaerobic digester, which can increase colour in the final effluent [72]. The increase of methane production depends on the initial biodegradability of the sludge, with higher impacts on hardly biodegradable sludge [71] and better results on waste activated sludge than on primary sludge [73]. While thermal hydrolysis largely decouples degradability from sludge age, there is still a basic relationship at long sludge ages [3]. Thermal hydrolysis also results in increased hydrolysis rates [3,65,74] and HRT could be decreased down to 2.9 days by feeding the digester with the liquid fraction of pretreated sludge [57]. Additional advantages of

Table 2
Thermal pretreatment.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge	175 °C 30 min	CSTR, HRT: 15 days 35 °C	Increase of CH_4 production from 115 to 186 mL g^{-1} COD_{in} (+62%)	[48]
Primary sludge	175 °C 30 min	CSTR, HRT: 15 days 35 °C	CH_4 production of 252 mL g^{-1} COD_{in} (no influence)	
Mixed sludge	175 °C 30 min	CSTR, HRT: 15 days 35 °C	Increase of CH_4 production from 205[a] to 234 mL g^{-1} COD_{in} (+14%)	
Activated sludge	175 °C 60 min	Batch, 25 days 35 °C	Increase of COD conversion to CH_4 from 48% to 68% (+42%)	[49]
Activated sludge	175 °C 60 min	CSTR, HRT: 5 days 35 °C	Increase of gas production from 108[a] to 216 mL g^{-1} COD_{in} (+100%)	[13]
Activated sludge (industrial)	180 °C 60 min	Batch, 8 days 37 °C	Increase of CH_4 production (+90%)	[50]
Mixed sludge	165–180 °C 30–60 min	WWTP 90,000 PE HRT: 17 days	Increase of electricity production (+20%)	[51]
Activated sludge	160 °C	WWTP 45,000 PE CSTR, HRT: 15 days	Increase of biogas production (+60%)	[52]
Mixed sludge	121 °C 60 min	CSTR, HRT: 20 days 36 °C	Increase of biogas production from 350[a] to 420 mL g^{-1} soluble VS_{in} (+20%)	[53]
Activated sludge	121 °C 30 min	Batch, 7 days 37 °C	Increase of biogas production from 3657[a] to 4843 L m^{-3} $sludge_{in}$ (+32%)	[54]
Digested mixed sludge	170 °C 60 s, 0.8 MPa	Batch 20 days	Increase of biogas production (+49%)	[55]
Activated sludge	170 °C 60 min	Batch, 24 days 35 °C	Increase of biogas production (+45%)	[56]
Activated sludge	170 °C 60 min	CSTR, HRT: 20 days 35 °C	Increase of CH_4 production from 88[a] to 142 mL g^{-1} COD_{in} (+61%)	
Activated sludge	175 °C 40 min	Fixed film reactor, HRT: 2.9 days 37 °C	65% TSS reduction	[57]
Activated sludge	170 °C 30 min	Batch, 24 days 35 °C	Increase of CH_4 production from 221[a] to 333 mL g^{-1} COD_{in} (+76%)	[58]
Activated sludge	170 °C 30 min	CSTR, HRT: 20 days 35 °C	Increase of CH_4 production from 145[a] to 256 mL g^{-1} VS_{in} (+51%)	[59]
Mixed sludge	140 °C, 1 min 0.6 MPa	WWTP 100,000 PE two-stage digestion 55–53 °C	Increase of biogas production from 507[a] to 599 L kg^{-1} VS_{in} (+18%)	[60]
Activated sludge (extended aeration)	160 °C 30 min	WWTP 62,000 PE HRT: 15 days 35 °C	Increase of TS removal from 25%[a] to 45%	[61]
Activated sludge	170 °C 30 min, 7 bar	Batch	Increase of CH_4 production (+50%)	[62]
Activated sludge	170 °C 30 min, 7 bar	Continuous HRT: 12 days	Increase of biogas production (+40–50%) Increase of electricity production (+40%)	
Activated sludge (wastewater from a crude oil refinery)	200 °C 30 min, 20 MPa	Two-stage UASB, HRT: 3.8 days 35 °C digestion of liquid after pretreatment (batch 33 days without pretreament)	Increase of CH_4 production from 2419[a] to 3775 mL kg^{-1} WAS (+15%)	[63]
Activated sludge	Microwave 175 °C	Batch, 18 days 33 °C	Increase of CH_4 production (+31%)	[64]
Primary sludge	Microwave 35–90 °C	Batch, 18 days 33 °C	Increase of degradation rates No impact on ultimate methane production	[65]

[a] Performance of anaerobic digestion without pretreatment.

thermal treatments include sludge sanitation, reduction of sludge viscosity with subsequent enhancement of sludge handling, and no extra energy needs, since energy requirements can be covered by excess biogas production and energy balance is positive [51]. Disadvantages are largely increased soluble inert fraction and final effluent colour [72], increased ammonia inhibition in the main digester due to increased performance [75], and possibly, poorer centrifuge or press solids capture due to an increase in fines.

Some industrial processes such as Cambi [51] and *Bio*THELYS® [61] have been commercialised (Table 2). Both processes consist of a treatment at 150–180 °C during 30–60 min, by vapour injection. The first Cambi process was implemented in 1995 at HIAS wastewater treatment plant (WWTP) (90,000 person equivalent (PE)) of Hamar (Norway). An energy balance showed that thermal hydrolysis led to 20% increase of electricity production [51]. More than 10 installations are currently in operation and the main results obtained are [76]: (i) an increase in biogas production and

reduction of organic matter around 60%, (ii) a reduction of sludge volume with digested sludge cake total solids (TS) content higher than 30%, (iii) an increase of digester capacity with organic loading of 5–6 kg VS m^{-3} day^{-1}. In France, the *Bio*THELYS® process (Veolia Waters) was implemented in 2006 at the urban WWTP of Saumur (62,000 PE, 1400 t TS $year^{-1}$ of sludge from an extended aeration tank) and Château Gontier (38,000 PE, 1000 t TS $year^{-1}$ of sludge). The results from Saumur were an increase of TS removal from 25% to 45% and an increase of sludge cake TS content from 22% to 30%, corresponding to 46% reduction of sludge volume compared to classical digestion [61]. The interest of sludge thickening before thermal treatment as well as the recovery of heat from hot streams in order to reduce energy requirements has been underlined [77].

While direct steam injection is normally used in industrial processes, research is mainly conducted with autoclave or microwave heating. Mottet et al. [74] compared 165 °C pretreatment by steam injection and electric heating and found no significant difference. Eskicioglu et al. [78] compared microwave heating and conventional heating in a water bath. Whereas both treatments led to the same solubilisation results, microwave heating resulted in slightly higher methane potentials (+16% after 15 days of mesophilic digestion and 96 °C pretreatment). A thermal effect may be caused by polarised parts of macromolecules aligning with the poles of the electromagnetic field [78].

5. Mechanical treatment

5.1. Ultrasonic treatment

Ultrasonic treatment acts to mechanically disrupt the cell structure and floc matrix. There are two key mechanisms associated with ultrasonic treatment; cavitation, which is favoured at low frequencies, and chemical reactions due to the formation of $OH^{\bullet}$, $HO_2^{\bullet}$, $H^{\bullet}$ radicals at high frequencies. In sludge treatment, low frequencies (20–40 kHz) are the most efficient. The mechanical phenomena of sludge sonication leads to sludge floc disintegration and microorganisms lyses, according to the treatment time and power, equating to specific energy applied [79]. The energy input for lysis is high, and inactivation of microorganisms was observed prior the occurrence of cell lysis [79]. Moreover, Salsabil et al. [80] showed by flow cytometry experiments that sonication at 20 kHz and 60 W, (TS = 7.8 g L^{-1}) did not produce cell membrane breakage. According to Li et al. [81], cells started to lyse only when the disintegration degree (solubilised COD divided by the maximum soluble COD obtained by alkaline hydrolysis) was over 40%.

Therefore, a threshold for specific energy is often reported for sludge solubilisation. This threshold specific energy ranges from 1000 to 16,000 kJ kg^{-1} TS and depends on sludge TS concentration. Indeed, the higher the sludge concentrations, the lower the specific threshold energy (higher efficiency); since cavitation bubbles have higher probabilities of contacting sludge particles. However, according to Show et al. [82] the optimal range of solids content for sonication lies between 2.3% and 3.2% TS; if the solids concentration is too high, increased viscosity hinders cavitation bubble formation. For a given specific energy, power input is more effective than retention time (i.e., a high power is more effective in disruption than a long retention time) [83].

For research, ultrasounds have widely been applied as pretreatment of anaerobic digestion; main results are summarised in Table 3. Considering energy consumption and enhancement of anaerobic digestion performance, applied specific energies are usually in the range from 1000 to 16,000 kJ kg^{-1} TS (the same as solubilisation threshold) although biogas production increases with the energy input [80]. Taken as a whole, biogas enhancement ranges from 24% to 140% in batch systems and from 10% to 45% in continuous or semi-continuous systems (Table 3). However, this latter may

Table 3
Mechanical pretreatments: ultrasound.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Mixed sludge	31 kHz 3.6 kW, 64 s	Continuous, HRT: 22 days 37 °C	Increase of VS removal from 45.8%[a] to 50.3% (+9%)	[90]
Mixed sludge (25 g TS L^{-1})	9 kHz 200 W, 30 min	Batch, 11 days 36 °C	Increase of CH_4 production from 210[a] to 345 mL g^{-1} VS_{in} (+64%)	[91]
Activated sludge (SRT: 16 days)	41 kHz 150 min	Semi-continuous, HRT: 8 days 37 °C	Increase of VS removal from 21.5%[a] to 33.7% (+36%)	[92]
Activated sludge (9.38 g TS L^{-1})	20 kHz 0.33 W mL^{-1}, 20 min	Batch, 100 days 35 °C	Increase of CH_4 production from 143[a] to 292 g kg^{-1} TS_{in} (+104%)	[79]
Sewage sludge (54 g TS L^{-1})	20 kHz 200 W, 30 min	Batch, 33 days 37 °C	Increase of biogas production (+138%)	[93]
Mixed sludge	20 kHz 180 W, 60 s	Batch, 28 days 35 °C	Increase of biogas production (+24%)	[94]
Activated sludge (27 g TS L^{-1})	20 kHz, 7000 and 15,000 kJ kg^{-1}TS	Batch, 16 days 35–37 °C	Increase of biogas production (+40%)	[95]
Activated sludge	5000 kJ kg^{-1} TS	Semi-continuous HRT: 20 days	Increase of biogas production (+36%)	[96]
Activated sludge (17.1 g TS L^{-1})	20 kHz 108,000 kJ kg^{-1} TS	Batch, 50 days 37 °C	Increase of biogas production (+84%)	[80]
Activated sludge (2.14% TS)	20 kHz 9690 kJ kg^{-1} TS	Batch, 35 days 36 °C	Increase of biogas production (+44%)	[97]
Activated sludge	30 kWh m^{-3} sludge	Batch	Increase of biogas production (+42%)	[98]
Activated sludge	30 kWh m^{-3} sludge	Continuous, HRT: 20 days	Increase of biogas production (+37%) Increase of VS removal (+25%)	
Activated sludge	Sonication of 25% of WAS	WWTP 330,000 PE	Increase of biogas production (+30%)	[88]
Mixed sludge(1.5% VSS)	20 kHz W cm^{-2}, 1.5 s	5000 m^3 egg-shape digester HRT: 22.5 days, 29–33 °C	Increase of biogas production (+45%)	[89]

[a] Performance of anaerobic digestion without pretreatment.

Table 4
Mechanical pretreatment: lysing-centrifuge.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge		Batch, 25 days 35 °C	Increase of biogas production from 91[a] to 168 mL g^{-1} COD_{in} (+85%)	[99]
Mixed sludge (only activated sludge lyses)		Batch, 25 days 35 °C	Increase of biogas production from 170[a] to 210 mL g^{-1} COD_{in} (+24%)	
Sewage sludge Liberec	39 m^3 h^{-1} 3140 rpm	Continuous HRT: 40 days	Increase of biogas production from 0.335[a] to 0.422 Nm^3 kg^{-1} VS_{in} (+26%)	[100]
Sewage sludge Furstenfeldbruck	12 m^3 h^{-1} 2250 rpm	Continuous HRT: 35 days	Increase of biogas production from 0.462[a] to 0.529 Nm^3 kg^{-1} VS_{in} (+15%)	
Sewage sludge Aachen-Soers	200 $m^3.h^{-1}$	Continuous HRT: 19 days 38 °C	Increase of biogas production from 0.326[a] to 0.402 Nm^3 kg^{-1} VS_{in} (+23%)	
Sewage sludge Prague		Semi- continuous, Two-stage HRT: 19 days, from 35 to 55 °C	Increase of biogas production from 0.470[a] to 0.650 Nm^3 kg^{-1} VS_{in} (+38%)	[60]

[a] Performance of anaerobic digestion without pretreatment.

be due either to VS destruction enhancement or to the increase of the digester organic load. Sonication has also been applied within the activated sludge process (configurations T1 or T2 in Fig. 1) [84]. Regarding the effect on waste activated sludge dewaterability, experimental results are contradictory, with positive or negative impacts. Kim and Kim [85] measured sludge dewaterability after sonication at increasing times; and found that dewaterability was first diminished and then improved with sonication time. In particular, Li et al. [81] showed that sludge dewaterability was only improved when sludge disintegration degree was 2–5%. Settling may also be improved by sonication [86]. Another advantage of sonication is the mitigation of sludge bulking problems [87,88] and potential digester foaming.

Ultrasounds have been implemented extensively in industry, mainly as pretreatment for anaerobic digestion (Table 3). For example, the implementation in Bamberg's WWTP (280,000 PE, Germany) has been described by Neis et al. [88]. However, this performance seems quite high as compared to other studies (Table 6). Xie et al. [89] estimated the energy ratio between net energy generation and electricity consumption by an ultrasound device. In this full scale experiment carried out in Singapore, the methane production increased by 45%, with an energy ratio of 2.5 (assuming an electricity yield of 2.2 kWh m^{-3} CH_4).

5.2. Lysis-centrifuge

Lysis-centrifuge operates directly on the thickened sludge stream in a dewatering centrifuge [99]. After this, it is then re-suspended with the liquid stream. Table 4 shows the main results published on this system. It has been implemented in several wastewater treatment plants as a pretreatment for anaerobic digestion: Liberec (100,000 PE, Czech Republic), Furstenfeldbruck (70,000 PE) and Aachen-Soers (650,000 PE) in Germany [100]. The increase of biogas production is 15–26% (Table 4).

5.3. Liquid shear

Liquid shear depends on high liquid flows due to a high pressure system to provide mechanical disruption to cells and flocs. Table 5 summarises the results of different technologies that have been used.

5.3.1. Collision plate

Sludge is pressurised to 30–50 bar by a high pressure pump and jetted to the collision plate after going through a nozzle. Thus, sludge undergoes a rapid depressurisation and is jetted on the plate with velocities of 30–100 m s^{-1}. This process has only been applied

Table 5
Mechanical pretreatment: high pressures.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge	Plate collision ΔP: 30 bar	Batch, 26 days 35 °C	Increase of VS removal from 35%[a] to 50% (+43%)	[101]
Activated sludge (14–18 g TS L^{-1})	Plate collision ΔP: 30 bar	CSTR, HRT: 13, 8, 6 days 35 °C	VS removal of 30%	[102]
Activated sludge (SRT: 3 days)	Homogeniser ΔP: 400 bar	Fixed biomass reactor, HRT: 2.5 days 35 °C	Increase of VS removal from 40%[a] to 51% (+28%)	[103]
Activated sludge (SRT: 13 days)	Homogeniser ΔP: 400 bar	Fixed biomass reactor, HRT: 2.5 days 35 °C	Increase of VS removal from 15%[a] to 28% (+87%)	
Activated sludge	Homogeniser ΔP: 300 bar (750 kJ kg^{-1} TS)	CSTR, HRT: 10–15 days 35 °C	Increase of CH_4 production from 129[a] to 206 mL g^{-1} VS_{in} (+60%)	[104]
Activated sludge	Homogeniser ΔP: 300 bar (750 kJ kg^{-1} TS)	Fixed biomass reactor, HRT: 2–5 days 35 °C	CH_4 production of 178 mL g^{-1} VS_{in}	
Mixed sludge	Homogeniser ΔP: 600 bar	CSTR, HRT: 20 days 36 °C	Increase of biogas production from 350[a] to 413 mL g^{-1} VSS_{in} (+18%)	[53]

[a] Performance of anaerobic digestion without pretreatment.

Table 6
Mechanical pretreatment: grinding.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge (SRT: 7 days)	d_b: 0.25 mm v_b: 10 m s^{-1} 9 min, 60 °C	Batch, 21 days 37 °C	Increase of biogas production (+10%)	[109]
Activated sludge (extended aeration)	d_b: 0.25 mm v_b: 10 m s^{-1} 9 min, 60 °C	Batch, 21 days 37 °C	Increase of biogas production (+24%)	
Anaerobic digested sludge	d_b: 0.25 mm v_b: 10 m s^{-1} 9 min, 60 °C	Batch,21 days 37 °C	Increase of biogas production (+62%)	
Activated sludge (SRT: 3 days)	d_b: 0.35 mm v_b: 6 m s^{-1} E_s: 2000 kJ kg^{-1} TS	Continuous suspended biomass HRT: 7 days	Increase of VS removal from 42%[a] to 47% (+12%)	[110]
Activated sludge (SRT: 3 days)	d_b: 0.35 mm v_b: 6 m s^{-1} E_s: 2000 kJ kg^{-1} TS	Fixed biomass HRT: 2 days	Increase of VS removal from 26%[a] to 37% (+88%)	

v_b is the balls velocity and d_b is the balls diameter.
[a] Performance of anaerobic digestion without pretreatment.

at laboratory scale and allowed a decrease in HRT from 14 to 6 days without affecting anaerobic digestion performance [101,102].

5.3.2. High pressure homogeniser

Sludge pressure is increased up to 900 bar, then sludge goes through an homogenisation valve under strong depressurisation [103]. This process has been tested at full-scale for anaerobic digestion. A fraction of digested sludge was treated at 150 bar and re-introduced in the digester, which led to an increase of biogas production by 30% and a reduction of sludge volume by 23% [105]. However, sludge dewaterability diminished [53].

Several other processes which are based on sludge pressurisation and depressurisation are commercially available. Examples are:

- the *Crown® process* (Biogest company), with operation at 12 bar in several full-scale implementations [106].
- *Cellruptor or Rapid non-equilibrium decompression, RnD® process* (Ecosolids). Sludge is compressed at pressures higher than 1 bar. A gas, which is soluble in the sludge stream, is introduced in the sludge stream. The gas, due to its rapid rate of diffusion across the cell walls, is transported across the cell walls. The gasified sludge stream is then depressurised. This rapid, non-equilibrium decompression causes exceedingly high shear rates and irreversible cell rupture, decreasing particle size, and releasing the interstitial water to the sludge stream. Biogas production can be increased from 0.3–0.6 to 0.48–0.816 m^3 kg^{-1} VS [107].
- *Microsludge® process* (Paradigm Environmental Technologie Inc). Sludge is first treated with chemicals with the aim of adjusting the pH to 11 or 2 in order to weaken cell walls. A high pressure homogeniser at 830 bar then provides cellular disruption. This process was applied in Los Angeles WWTP. Treated waste activated sludge was introduced in a digester together with primary sludge, with a ratio 68/32 (w/w). The degradation of mixed sludge was increased from 50% to 57% [108].

5.4. Grinding

Table 6 shows results obtained during anaerobic digestion of sludge which had been disintegrated by stirred ball mills. Grinding was more beneficial on digested sludge (increase of batch biogas production by 60%) and on waste activated sludge from an extended aeration process (24% increase) than on activated sludge with an higher SRT (7% increase) [109,110]. Kopp et al. [110] underlined higher impacts on methane production when anaerobic digestion was carried out at short HRT (see Table 6), showing an acceleration of anaerobic digestion as the main effect of pretreatment.

6. Chemical treatment

6.1. Oxidation

The most widely used chemical method is ozonation (Table 7). Ozonation leads to partial sludge solubilisation and yield increases with ozone dose. A too high ozone dose will result in reduced apparent solubilisation due to oxidation of the solubilised components [112]. In addition, it is oxidative, and may therefore increase destruction at the expense of methane yield. Several studies have shown an optimal ozone dose for the enhancement of anaerobic biodegradability: 0.1 g O_3 g^{-1} COD [111], 0.2 g O_3 g^{-1} TSS [112], 0.15 g O_3 g^{-1} TS [118] (Table 7). However, sludge ozonation was first used in combination with activated sludge process for wastewater treatment [119]. In this work, the feasibility of operating an activated sludge process without physically wasting excess sludge has been shown. Thickened sludge was dosed with ozone (0.02 g O_3 g^{-1} TS) and re-introduced in the aerated tank. However, the concentration of nitrogen and suspended solids in the effluent slightly increased, although it remained under authorised limits. A review of studies concerning the combination of ozonation with activated sludge process (configurations T1 or T2 in Fig. 1) has been recently proposed by Chu et al. [120]. This process has been commercialised by the Japanese Kurita company and about 30 installations have been implemented [121]. Another industrial process has been proposed by Ondeo-Degremont (Suez): Biolysis® O process [46].

Ozonation has also been combined with anaerobic digestion as a pretreatment [111,112,118] or posttreatment and recycling back to the anaerobic digester [113,114]. The main results are summarised in Table 7. Goel et al. [113] showed better performance and lower ozone consumption in the case of posttreatment and recycling in the digester.

Hydrogen peroxide has also been used as an oxidant [56,116] (Table 7). The COD removal during anaerobic digestion was enhanced by means of oxidation at 90 °C with 2 g H_2O_2 g^{-1} VSS, but not by the oxidation at 37 °C [116]. Moreover, posttreatment on the recirculation loop, treating 20% of the sludge stream, was more efficient than a configuration with pretreatment. However, the process consisting of one anaerobic digester, high temperature oxidation and a second digester led to the highest removal of faecal coliforms [116]. Use of Fenton catalysed oxidation (0.067 g Fe(II) g^{-1} H_2O_2,

Table 7
Chemical pretreatment: oxidation.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Mixed sludge	0.1 g O_3 g^{-1} COD	Batch, 30 days 33 °C	Increase of CH_4 production from 110[a] to 220 mL g^{-1}.COD_{in} (+100%)	[111]
Sewage sludge	0.1 g O_3 g^{-1} TSS	Batch 30 days	Increase of CH_4 production from 82[a] to 173 mL g^{-1}.COD_{in} (+110%)	[112]
Activated sludge (synthetic)	0.05 g O_3 g^{-1} TS	CSTR HRT: 28 days 35 °C	Increase of TS removal from 31%[a] to 59% (+90%)	[113]
Activated sludge (synthetic)	0.045 g O_3 g^{-1} TS recirculation loop at digester outlet	CSTR With supernatant withdraw HRT: 28 days 35 °C	TS removal of 85%	
Activated sludge	0.16 g O_3 g^{-1} TSS recirculation loop at digester outlet (25%)	CSTR 35 °C	Increase of COD removal from 38%[a] to 58% (+53%)	[114]
Activated sludge	0.15 g O_3 g^{-1} TS	Batch, 18 days 35 °C	Increase of biogas production from 150[a] to 367 mL g^{-1} COD_{in} (+145%)	[115]
Activated sludge 17 g L^{-1}	H_2O_2: 150 mmol L^{-1} $FeSO_4$: 5 mmol L^{-1} 90 °C, 60 min	Batch, 24 days 35 °C	Increase of biogas production (+16%)	[56]
Mixed sludge	2 g H_2O_2 g^{-1} VSS 90 °C, 24 h	CSTR, HRT: 30 days 37 °C	Increase of COD removal from 52.2%[a] to 70.1% (+34%)	[116]
Mixed sludge	2 g H_2O_2 g^{-1} VSS 90 °C, 60 h recirculation at digester outlet (20% per day)	CSTR HRT: 30 days 37 °C	Increase of COD removal from 52.2%[a] to 74.6% (+43%)	
Digested mixed sludge (SRT: 15 days)	2 g H_2O_2 g^{-1} VSS 90 °C, 24 h	CSTR, HRT: 15 days 37 °C	Increase of VSS removal from 52.2%[a] to 70.6% (+35%)	
Sewage sludge	Catalytic wet oxidation 270 °C, 86 kg m^{-2} 24 min	UASB, HRT: 24 h, 35 °C Digestion of supernatant of treated sludge	Soluble COD removal of 93.8%	[117]
Activated sludge (wastewater from a crude oil refinery)	Wet air oxidation 200 °C, 20 MPa 30 min	Two-stage UASB, HRT: 3.8 days, 35 °C digestion of liquid after pretreatment (classical batch 33 days without pretreatment)	Increase of CH_4 production from 2419[a] to 33,084 mL kg^{-1} WAS (+27%)	[63]

[a] Performance of anaerobic digestion without pretreatment.

and 60 g H_2O_2 kg^{-1} TS) decreased sludge resistance to dewatering in terms of capillary suction time (CST), but did not have a positive effect on sludge dewatering performance on a belt press simulation [122]. Wet oxidation has also been applied to sewage sludge, with the solubilised fraction subsequently treated in a UASB reactor [63,117] (Table 7).

6.2. Alkali treatments

Alkali treatment is relatively effective in sludge solubilisation, with in order of efficacy being (NaOH > KOH > $Mg(OH)_2$ and $Ca(OH)_2$) [54]. However, too high concentrations of Na^+ or K^+ may cause subsequent inhibition of anaerobic digestion [123]. It is normally combined with thermal treatment. Sludge solubilisation and anaerobic biodegradability increase with alkali dose and temperature, with an upper limit [54,56]. Compared to thermal hydrolysis, alkali treatment temperature is normally lower, and the increase in temperature normally driven by chemical processes (from 170 to 120–130 °C). Performance improvements in methane production are summarised in Table 8. They are moderate as compared to solubilisation performance [50,56]. Alkali treatment (pH of 12, NaOH) combined with microwave irradiation (160 °C) led to methane production 10% higher than microwave irradiation alone [124]. However, since the addition of alkali increases mineral content of digested sludge, it reduces the interest of cotreatment on sludge reduction [56]. In addition, sludge dewaterability may be diminished by KOH addition [125]. Dogan and Sanin [124] observed an improvement on the dewaterability (measured by CST) by about 22% after anaerobic digestion of pretreated sludge (pH of 12, 160 °C microwave) compared to anaerobic digestion of waste activated sludge.

7. Comparison of treatment methods

The results of sludge cotreatments may not be compared directly from different studies as they depend on the sludge (primary, waste activated, sludge age, sludge concentration ...) and on the anaerobic digestion process parameters (batch or continuous, HRT, temperature). The basis of comparison for pretreatment methods can thus be divided into a number of different components including:

(a) Whether the treatment method is aimed at activated or primary sludges.
(b) Treatment effectiveness – whether it increases just degradation rate, or increases the overall amount of material available (bioavailability).
(c) Cost of treatment, particularly energy cost, and secondary costs caused by nutrient release or generation of byproducts (e.g., melanoidins).
(d) Chemical consumption, particularly for oxidative or chemical treatment.

For point (a), as outlined previously in the paper, most treatment methods are orientated towards activated sludges, and are often applied to primary sludges for secondary considerations such as improved dewaterability or pathogen removal. Most analyses of comparative performance have focused also on activated sludge, and the main focus for comparative analysis has been pretreat-

Table 8
Thermo-chemical pretreatment.

Substrate	Treatment conditions	Anaerobic digestion conditions	Results	Reference
Activated sludge ($43\,g\,L^{-1}$)	$300\,meq\,HCl\,L^{-1}$ 175 °C, 60 min	Batch, 25 days 35 °C	Increase of COD conversion to CH_4 from 48%[a] to 75% (+56%)	[49]
Activated sludge ($43\,g\,L^{-1}$)	$300\,meq\,NaOH\,L^{-1}$ 175 °C, 60 min	Batch, 25 days 35 °C	Increase of COD conversion to CH_4 from 48%[a] to 78% (+62%)	
Activated sludge ($7\,g\,L^{-1}$)	$0.3\,g\,NaOH\,g^{-1}$ VSS 130 °C, 5 min	Batch, 10 days 37 °C	Increase of COD conversion to CH_4 from 32%[a] to 42% (+31%)	[50]
Activated sludge (industrial, $8.4\,g\,L^{-1}$)	$0.3\,g\,NaOH\,g^{-1}$ VSS 130 °C, 5 min	Batch, 10 days 37 °C	Increase of COD conversion to CH_4 from 11%[a] to 25% (+127%)	
Activated sludge ($17\,g\,L^{-1}$)	$7\,g\,NaOH\,L^{-1}$ 121 °C, 30 min	Batch, 7 days 37 °C	Increase of biogas production from 3 657[a] to 5 037 $L\,m^{-3}$ sludge (+38%)	[54]
Activated sludge	$7\,g\,NaOH\,L^{-1}$ 121 °C, 30 min	Two-stage: Acidogenic: HRT: 6 days, 37 °C Methanogenic: HRT: 12 days, 41 °C	Increase of CH_4 production from 290[a] to $520\,L\,kg^{-1}$ VS_{in} (+79%)	[126]
Activated sludge ($30\,g\,L^{-1}$)	$45\,meq\,NaOH\,L^{-1}$ 55 °C, 240 min	Batch, 20 days 35 °C	Increase of CH_4 production from165[a] to $310\,L\,kg^{-1}$ VS_{in} (+88%)	[127]
Activated sludge ($100\,g\,L^{-1}$)	pH: 11 90 °C, 10 h	Batch, 15 days 55 °C	CH_4 production of 280 $L.kg^{-1}$ VS_{in}	[128]
Activated sludge ($17\,g\,L^{-1}$)	$1.65\,g\,KOH\,L^{-1}$ pH: 10 130 °C, 60 min	Batch 24 days 35 °C	Increase of biogas production (+30%)	[56]
Activated sludge ($17\,g\,L^{-1}$)	$1.65\,g\,KOH\,L^{-1}$ pH: 10 130 °C, 60 min	CSTR, HRT: 20 days 35 °C	Increase of CH_4 production from 88[a] to $154\,mL\,g^{-1}$ COD_{in} (+75%)	
Activated sludge	Microwave, 160 °C pH: 12 by NaOH 16 min	Batch 37 °C	Increase of CH_4 production (+19%)	[124]
Activated sludge	Microwave, 160 °C pH: 12 by NaOH 16 min	Semi-continuous, HRT: 15 days 37 °C	Increase of CH_4 production from 144[a] to $220\,mL\,g^{-1}$ VS_{in} (+53%)	

[a] Performance of anaerobic digestion without pretreatment.

ment versus no-pretreatment. Bougrier et al. [59] compared the effect of ultrasound, thermal hydrolysis and ozonation pretreatment on the same activated sludge sample prior to batch mesophilic anaerobic digestion. In terms of solubilisation, the thermal treatment was the most efficient. The thermal treatment also led to a strong decrease of apparent viscosity, and a strong increase in filterability. All three pretreatments improved biogas production. For ozonation (0.10 and $0.16\,g\,O_3\,g^{-1}$ TS), this enhancement was low (246–272 $mL\,CH_4\,g^{-1}$ COD_{in} against 221 $mL\,CH_4\,g^{-1}$ COD_{in} for the raw sludge) compared to sonication (with a specific energy of 6250 and 9350 $kJ\,kg^{-1}$ TS) and thermal hydrolysis (at 170 or 190 °C), which both resulted in the same outcomes (325–334 $mL\,CH_4\,g^{-1}$ COD_{in}). Ultrasonic treatment provided minimal solubilisation of sludge and particle size reduction, but improved biodegradability of the particulate fraction. Thermal hydrolysis increased solubilisation, but did not enhance degradability of residual particulates [59].

Salsabil et al. [129] compared thermal treatment (from 40 to 120 °C), ozonation ($0.1\,g\,O_3\,g^{-1}$ TS), and sonication ($200{,}000\,kJ\,kg^{-1}$ TS) on the basis of TSS reduction after subsequent batch anaerobic digestion. Increase of TSS removal was 30% with sonication and 20% with ozonation and thermal treatments at 90 or 120 °C. However, specific energy of sonication was extremely high compared to other studies.

Kim et al. [54] compared thermal (121 °C), chemical ($7\,g\,L^{-1}$ NaOH), ultrasonic (42 kHz, 120 min) and thermochemical (121 °C, $7\,g\,L^{-1}$ NaOH) pretreatment prior to batch anaerobic digestion. They obtained the best results with the thermal (3390 $L\,CH_4\,m^{-3}$ WAS) and thermochemical (3367 $L\,CH_4\,m^{-3}$ WAS) pretreatments, followed by the ultrasounds (3007 $L\,CH_4\,m^{-3}$ WAS) and chemical (2827 $L\,CH_4\,m^{-3}$ WAS) pretreatments, the production from raw sludge being equal to (2507 $L\,CH_4\,m^{-3}$ WAS).

Barjenbruch and Kopplow [53] compared thermal treatment (80–121 °C), high pressure homogenisation (600 bar) and enzymatic treatment (carbohydrase addition) for pretreatment prior to continuous anaerobic digestion with 10 days HRT. An increase of biogas production was observed in the following order: low intensity thermal treatment at 90 and 121 °C (>20% increase) > high pressure and thermal treatment at 80 °C (>16–17% increase) > enzymatic treatment (>13% increase).

Yang et al. [63] studied thermal pretreatment and wet air oxidation followed by anaerobic digestion of the liquid fraction in a two stage UASB reactor. Although some COD was oxidised to CO_2 during pretreatment, wet air oxidation (200 °C, 20 MPa) led to better results than thermal treatment (200 °C): 385 versus 261 $mL\,biogas\,g^{-1}$ COD_{in} and 3084 versus 2775 $mL\,CH_4\,kg^{-1}$ WAS. Moreover, wet air oxidation showed better filterability of the residue compared with thermal treatment.

Muller et al. [130] considered a 250,000 PE virtual WWTP to compare stirred ball milling, ozonation, lysate centrifugation and sonication. The authors provided several classifications of pretreatments according to:

- Energy demand: lysate centrifuge < stirred ball mill < sonication < ozonation.
- Increase of sludge degradation: ozonation > stirred ball mill > sonication > lysate centrifuge.
- Increase in polymer demand for dewatering: lysate centrifuge < stirred ball mill < sonication < ozonation.
- Increase in polymer demand: lysate centrifuge < stirred ball mill < sonication < ozonation.
- Increase in soluble COD and ammonia concentrations in supernatant after dewatering: sonication < lysate centrifuge < stirred ball mill < ozonation.

Overall, all pretreatment options require significant and comparative resources (see next section). The performance level is reflected in the intensity of treatment, with lower energy methods such as sonication and mechanical pretreatment mainly affecting hydrolysis rate, and to a limited extent (20–30% improved VS destruction), but high impact methods such as thermal hydrolysis and oxidation having significant improvement, but with a substantial energy (and possibly capital) input.

8. Energy aspects

One of the most significant inputs, environmentally, and financially is energy. While the cost of treatment may be disposal driven, in energy terms, energy utilised should hopefully match the energy produced by increases in biogas production. The energy input depends heavily on method, and may be a function of sludge composition, operating and ambient conditions, and equipment used, amongst others.

A summary of performance and energy outcomes for the major digester options is given in Table 9. This is information standardised from the various sources on the basis of kg VS. Assumptions used are given as follows. Energy consumption in anaerobic digesters is electrical and thermal. Electrical requirements are mainly feed and mixing, and are approx. 0.1–0.2 kWh m^{-3} d^{-1} [131,132]. 0.12 kWh m^{-3} d^{-1} has been used in our analysis. The analysis also assumes a hydraulic retention time of 20 days for mesophilic, or 15 days for thermophilic. Heating requirements are thermal capacity plus approx. 10% losses in mesophilic or 20% in thermophilic. This is consistent with reasonable insulation [132]. Heat recovery could reduce this further, but this is likely not necessary (see below). Performance measures were taken from references in the far column, and were available thermal or electrical consumption values. A nominal VS:TS ratio of 80% was used, with a COD:VS ratio of 1.5 g COD g VS^{-1}. Calorific values and heat capacities have been taken from standard texts. In general, where a range of performance measures have been used, the more widely industrially applied examples, or best conditions have been applied. For example, 200 bar has been used as a reference case for high pressure homogenisation, while approx 170 °C has been used for thermal hydrolysis. Energy has not been split between electrical-available, and thermal available energy, but most cogeneration engines would produce approx. 30–40% as electricity, and 40–50% as heat. Thus one advantage of thermal treatments is the use of heat energy which is produced from biogas. This thermal energy is generally in excess as compared to the wastewater treatment plant needs.

As shown in Table 9, generally mesophilic and thermophilic produce adequate thermal energy, and an excess of electrical energy. This is consistent with knowledge of our systems, and only thermophilic systems in cold climate, or with poorly degradable feeds struggle to produce sufficient energy for self-heating [133]. Ferrer et al. [39] compared to the base case, three broad classes of pretreatment:

(a) Mild impact temperature–biological thermal pretreatment. This is comparable in performance and energy consumption to single stage thermophilic.
(b) High impact temperature–thermal hydrolysis. This uses much more thermal energy, but improved performance means there is sufficient energy available. Therefore, this can be applied, with minimal loss, or increase in total electrical energy yield.
(c) Mild impact mechanical. All the mechanical forms of solubilisation have moderate increases in overall yield, with generally, consumption of approx. 0.3 kWh kg VS^{-1}. This produces an increase in gas production of approx. 0.5 kWh kg VS^{-1}, which given a 30% electrical yield, would mean that the energy balance is generally negative.

Energy balances for mechanical options can be improved by thickening [98,134], but at the expense of increased viscosity and energy consumption [135]. Ozonation and chemical processes have not been included in this analysis, due to a lack of information.

All options for pretreatment have substantial capital cost, with thermal hydrolysis being more capitally intensive than mechanical options [136]. Often, the cost of ancillary items such as odour control, and sludge receivals in a centralised facility exceed substantially the cost of the actual pretreatment equipment [136]. Investment and operation costs of lysing-centrifuges are amongst the lowest, especially when they are implemented by adapting a lysing device to existing machinery. Other options to improve digester retention time, such as gravity belt thickening [132] may improve digestion performance substantially at minimal capital cost as an alternative to pretreatment.

9. Nutrients issue

Activated sludges have a nitrogen content of 11%, and a phosphorous content of 5% [132]. Naturally, an improvement in activated sludge destruction increases release of these compounds, which then need to be treated, either in a dedicated process, or through the main treatment process. Byproducts such as melanoidins can also carry embodied nitrogen [72]. While this can increase the cost of pretreatment, especially if nutrients are removed in the main process, it also offers the possibility for nutrient recovery from a concentrated stream.

9.1. Release of nutrients

Release of nutrients has been observed consistently due to most pretreatment processes. For biological, in a two stage thermophilic

Table 9
Energy analysis.

Pretreatment method	Treatment conditions	Feed concentration	VS destruction	Electrical consumed (kWh kg^{-1} VS fed)	Thermal consumed (kWh kg^{-1} VS fed)	Max biogas (kWh kg^{-1} TS fed)	References
None-mesophilic		6%	40%	0.04	0.5	1.9	[5,111]
None-thermophilic		6%	50%	0.03	1.0	2.4	[137]
Biological (thermal)	70 °C 9–48 h	6%	50%	0.03	1.0	2.4	[4,34,39]
Thermal hydrolysis	170 °C 15–30 min	9%	60%	0.04	2.0	2.9	[2,51,60,62,138]
Sonication	100 W, 16 s, 30 kW m^{-3}	6%	50%	0.37	0.5	2.4	[98,134]
Ball milling		6%	50%	1.04	0.5	2.4	[134]
High pressure	200 bar	6%	50%	0.33	1.0	2.6	[135]

(60 °C)–mesophilic process, Watts et al. [139] observed a 35–40% VS removal treating secondary sludge and a phosphorus release ranging from 10% to 20%. Muller [140] observed an increase by a factor of 10 of nitrogen concentration and a factor of 3 for phosphorus in the supernatant after disintegration by high-pressure homogeniser (80 MPa). In another study [141], sonication at 500 kWh m^{-3} over 1 h (a very high dose) of a waste activated sludge originating from a biological nitrogen and phosphorus removal process (BNPR) led to the release of 57% of the organic content (measured as COD), 70% of total nitrogen and more than 60% phosphorus. Most released nitrogen was organic-N (84.9%), followed by NH_3-N (14.9%) and trace amounts of nitrate and nitrite. 80% of the released phosphorus was as PO_4-P [141]. Microwave advanced oxidation ($MW/H_2O_2/O_3$) caused release of 30% of TP and 20% of TKN and 37% of total COD [142]. This brief review has focused mainly on release during pretreatment. Treatment during the actual digestion process would be in excess to this, and indeed, for more aggressive treatment methods (e.g., thermal hydrolysis) can result in in-digester total ammonia concentrations in excess of 3 g L^{-1}, with consequent ammonia inhibition.

9.2. Nutrients removal or recovery

Nutrient impact depends on whether the sludge minimisation process is a main stream cotreatment, or pretreatment method (Fig. 1). The impacts for cotreatment are (a) impact of released nutrients and organics, and (b) impact on the activated sludge population. As an example, according to Boehler and Siegrist [143] nitrifiers are partially killed by cotreatment ozonation, which can therefore lead to a decrease of the sludge age, increasing risk of nitrification failure. In contrast, in another study, nitrification potential was shown to remain constant after the introduction of ozonation [143,144]. The recycling of treated sludge may also lead to an increase of denitrification rates, as disintegrated carbon material can be used as carbon source for the denitrification process [140,145]. The release of phosphorus is more problematic as an additional physical-chemical precipitation of phosphorus is necessary to deal with phosphorus removal in the activated sludge process combined with ozonation [146] or sonication [147].

Pretreatment of sludge will result in increased nitrogen and phosphorous release as discussed above. Nitrogen is mainly in ammonium form and phosphorus in phosphates. These streams can be treated in the main treatment (activated sludge or BNPR process) via recycling of reject water, but this has a direct cost in terms of carbon, electricity, and phosphorous precipitant, and relies on the main process having sufficient capacity. It is generally more efficient to apply a dedicated process directly on the concentrated reject water. Nitrogen can be removed by dedicated processes such as nitrification/denitrification, Anammox, Cannon, NOx processes, as described in Ahn's review [148]. Morse et al. [149] have reviewed the technologies for phosphorous removal and recovery: biological phosphorus removal, chemical precipitation (mainly with Ca, Al or Fe addition) or crystallisation. Among them, struvite ($MgNH_4PO_4{\cdot}6H_2O$) crystallisation is highly recommended, in particular to recover phosphorus from digested effluents [150–155]. The increased concentrations of nitrogen and phosphorus in the anaerobic supernatant make this process feasible in contrast to water line struvite recovery. However, the stoichiometry of struvite precipitation means that only minimal amounts of nitrogen are removed.

In a number of cases, pretreatment has been applied specifically for nutrient release and recovery:

- Addition of a strong base (NaOH), strong acid (HCl), organic acid (citric acid), and sodium acetate [156].
- Microwave/advanced oxidation process [157,158].
- Sonication [141].
- Ozonation [159].
- Heat pretreatment (70 °C) and addition of $CaCl_2$ for recovery of P before anaerobic digestion [160].

10. Conclusions

Sludge minimisation may be either combined with the activated sludge process (cotreatment) in the wastewater treatment line or as a pretreatment to anaerobic digestion. If combined to the activated sludge process, the cotreatment objective is to avoid or to mitigate excess sludge production. However, excess sludge reduction should not impact seriously the main process. In particular, cotreatment will generally decrease effective sludge age by destruction of nitrifiers, increase effective nitrogen and phosphorous load, and increase the effective concentration of micropollutants and metals, which would normally adsorb onto the sludge.

If combined to sludge anaerobic digestion, the pretreatment objective is to not only reduce the final amount of sludge to be disposed of, but also to increase methane production. Various methods may have the effect of:

- Increasing degradability extent, leading higher energy recovery and lower residual digested sludge.
- Increasing degradation kinetics, making it possible to decrease sludge retention time in the digester, therefore making it possible to reduce digester volume or increase the organic load rate of a given digester.

Mechanical and low intensity processes such as biological pretreatment, sonication, and high pressure treatment generally increase rate, while high intensity processes such as thermal hydrolysis increase extent and rate. Mechanical processes have low energy requirements, as electricity. Low intensity thermal phased pretreatment, has higher energy requirements, but as thermal energy, which is generally available at a lower cost. Thermal hydrolysis has a high energy requirement, as thermal energy. Overall performance and feasibility depends on a wide range of factors, not just encompassed by energy use and performance. For instance, sludge dewaterability is a key parameter, and the polymer dose and total solids content in the sludge cake can be improved or degraded depending on cotreatment conditions. As a result of cotreatments, a fraction of nitrogen, phosphorus and refractory COD is released into the dewatered sludge liquid phase. If this phase is returned to the main activated sludge process, the degradation of such additional load may increase aeration costs. However the recovery of phosphorus and a part of nitrogen by struvite crystallisation is a sustainable option made possible by digestion.

References

[1] USEPA, Standards for the Use or Disposal Sewage Sludge, Final Rules 40 CFR Part 257 (1993) US Environmental Protection Agency.
[2] L. Appels, J. Baeyens, J. Degreve, R. Dewil, Principles and potential of the anaerobic digestion of waste-activated sludge, Prog. Energy Combust. Sci. 34 (6) (2008) 755–781.
[3] D.J. Batstone, S. Tait, D. Starrenburg, Estimation of hydrolysis parameters in full-scale anerobic digesters, Biotechnol. Bioeng. 102 (5) (2009) 1513–1520.
[4] S.I. Perez-Elvira, P.N. Diez, F. Fernandez-Polanco, Sludge minimisation technologies, Rev. Environ. Sci. Bio/Technol. 5 (4) (2006) 375–398.
[5] H.Q. Ge, P.D. Jensen, D.J. Batstone, Pre-treatment mechanisms during thermophilic-mesophilic temperature phased anaerobic digestion of primary sludge, Water Res. 44 (1) (2010) 123–130.
[6] J.M. Gossett, R.L. Belser, Anaerobic digestion of waste activated sludge, J. Environ. Eng. ASCE 108 (EE6) (1982) 1101–1120.
[7] S.G. Pavlostathis, E. Giraldo-Gomez, Kinetics of anaerobic treatment: A critical review, Crit. Rev. Environ. Control 21 (5–6) (1991) 411–490.
[8] V. Urbain, J.C. Block, J. Manem, Bioflocculation in activated-sludge – an analytic approach, Water Res. 27 (5) (1993) 829–838.

[9] F. Jorand, P. Guicherd, V. Urbain, J. Manem, J.C. Block, Hydrophobicity of activated-sludge flocs and laboratory-grown bacteria, Water Sci. Technol. 30 (11) (1994) 211–218.
[10] B.M. Wilén, B. Jin, P. Lant, The influence of key chemical constituents in activated sludge on surface and floculating properties, Water Res. 37 (9) (2003) 2127–2139.
[11] S. Comte, G. Guibaud, M. Baudu, Effect of extraction method on EPS from activated sludge: An HPSEC investigation, J. Hazard. Mater. 140 (1–2) (2007) 129–137.
[12] B. Frohlund, T. Griebe, P.H. Nielsen, Enzyme activity in activated-sludge floc matrix, Appl. Microbiol. Biotechnol. 43 (4) (1995) 755–761.
[13] Y.Y. Li, T. Noike, Upgrading of anaerobic digestion of waste activated sludge by thermal pretreatment, Water Sci. Technol. 26 (3–4) (1992) 857–866.
[14] X.Q. Zhang, P.L. Bishop, Biodegradability of biofilm extracellular polymeric substances, Chemosphere 50 (1) (2003) 63–69.
[15] Z.W. Wang, Y. Liu, J.H. Tay, Biodegradability of extracellular polymeric substances produced by aerobic granules, Appl. Microbiol. Biotechnol. 74 (2) (2007) 462–466.
[16] C. Park, R.F. Helm, J.T. Novak, Investigating the fate of activated sludge extracellular proteins in sludge digestion using sodium dodecyl sulfate polyacrylamide gel electrophoresis 80 (12) (2008) 2219–2227.
[17] A. Mottet, E. François, E. Latrille, J.P. Steyer, S. Déléris, F. Vedrenne, H. Carrère, Estimating anaerobic biodegradability indicators for waste activated sludge, Chem. Eng. J. 160 (2) (2010) 488–496.
[18] M. Henze, W. Gujer, T. Mino, M. van Loosdrecht, Activated Sludge Models ASM1, ASM2, ASM2d, and ASM3, (2000), IWA Scientific and Technical Report: London.
[19] G.F. Parkin, W.F. Owen, The fundamentals of anaerobic digestion of wastewater sludge, J. Environ. Eng. 112 (1986) (1986) 867–920.
[20] S.K. Bhattacharya, R.L. Madura, D.A. Walling, J.B. Farrel, Volatile solids reduction in two phase and conventional anaerobic sludge digestion, Water Res. 30 (5) (1996) 1041–1048.
[21] V.A. Vavilin, B. Fernandez, J. Palatsi, X. Flotats, Hydrolysis kinetics in anaerobic degradation of particulate organic material: An overview, Waste Manag. 28 (6) (2008).
[22] W. Rudolfs, H. Heukelelian, Thermophilic digestion of sewage sludge solids. I. -preliminary paper, Indust. Eng. Chem. 22 (1930) 96–99.
[23] R. Roberts, L. Son, W.J. Davies, C.F. Forster, Two stage, thermophilic/mesophilic anaerobic digestion of sewage sludge, TransIChem 77 (B) (1999) 93–97.
[24] J. Oles, N. Dichtl, H.H. Niehoff, Full scale experience of two stage thermophilic/mesophilic sludge digestion, Water Sci. Technol. 36 (6–7) (1997) 449–456.
[25] N. Aoki, M. Kawase, Development of high-performance thermophilic two-phase digestion process (review paper), Water Sci. Technol. 23 (1991) 1147–1156.
[26] I. Ferrer, F. Vázquez, X. Font, Long term operation of a thermophilic anaerobic reactor: Process stability and efficiency at decreasing sludge retention time, Bioresour. Technol. 101 (9) (2010) 2972–2980.
[27] Y.C. Song, S.J. Kwon, J.H. Woo, Mesophilic and thermophilic temperature co-phase anaerobic digestion compared with single stage mesophilic- and thermophilic digestion of sewage sludge, Water Res. 38 (2004) 1653–1662.
[28] S. Ponsá, I. Ferrer, F. Vázquez, X. Font, Optimization of the hydrolytic-acidogenic anaerobic digestion stage (55 °C) of sewage sludge: Influence of pH and solid content, Water Res. 42 (14) (2008) 3972–3980.
[29] H. Ge, P.D. Jensen, D.J. Batstone, Pre-treatment mechanisms during thermophilic-mesophilic temperature phased anaerobic digestion of primary sludge, Water Res. 44 (1) (2010) 123–130.
[30] H.B. Nielsen, Z. Mladenosvska, B.K. Ahring, Bioaugmentation of a two stage thermophilic (68 °C/55 °C) anaerobic digestion concept for improvement of the methane yield from cattle manure, Biotechnol. Bioeng. 97 (6) (2007) 1638–1643.
[31] N. Cabirol, M.R. Oropeza, A. Noyola, Removal of helminth eggs,and fecal coliforms by anaerobic thermophilic sludge digestion, Water Sci. Technol. 45 (10) (2002) 269–274.
[32] C. De León, D. Jenkins, Removal of fecal coliforms by thermophilic anaerobic digestion, Water Sci. Technol. 46 (10) (2002) 147–152.
[33] H. Hartmann, B.K. Ahring, A novel process configuration for anaerobic digestion of source-sorted household waste using hyper-thermophilic post-treatment, Biotechnol. Bioeng. 90 (7) (2005) 830–837.
[34] J.Q. Lu, H.N. Gavala, I.V. Skiadas, Z. Mladenovska, B.K. Ahring, Improving anaerobic sewage sludge digestion by implementation of a hyper-thermophilic prehydrolysis step, J. Environ. Manage. 88 (4) (2008) 881–889.
[35] H.N. Gavala, U. Yenal, I.V. Skiadas, P. Westermann, B.K. Ahring, Mesophilic and thermophilic anaerobic digestion of primary and secondary sludge. Effect of pre-treatment at elevated temperature, Water Res. 37 (19) (2003) 4561–4572.
[36] M. Climent, I. Ferrer, M.D. Baeza, A. Artola, F. Vazquez, X. Font, Effects of thermal and mechanical pretreatments of secondary sludge on biogas production under thermophilic conditions, Chem. Eng. J. 133 (1–3) (2007) 335–342.
[37] D. Bolzonella, P. Pavan, M. Zanette, F. Cecchi, Two-phase anaerobic digestion of waste activated sludge: Effect of an extreme thermophilic prefermentation, Ind. Eng. Chem. Res. 46 (21) (2007) 6650–6655.
[38] I. Ferrer, S. Ponsa, F. Vazquez, X. Font, Increasing biogas production by thermal (70 °C) sludge pre-treatment prior to thermophilic anaerobic digestion, Biochem. Eng. J. 42 (2) (2008) 186–192.
[39] I. Ferrer, E. Serrano, S. Ponsa, F. Vazquez, X. Font, Enhancement of thermophilic anaerobic sludge digestion by 70 °C pre-treatment: energy considerations, J. Residuals Sci. Technol. 6 (1) (2009) 11–18.
[40] R. Borja, B. CJ, A. Garrido, Kinetics of black-olive wastewater treatment by the active sludge system, Process Biochem. 29 (7) (1994) 587–593.
[41] S. Subramanian, N. Kumar, S. Murthy, J.T. Novak, Effect of anaerobic digestion and anaerobic/aerobic digestion processes on sludge dewatering, J. Residuals Sci. Technol. 4 (1) (2007) 17–23.
[42] N. Shiota, A. Akashi, S. Hasegawa, A strategy in wastewater treatment process for significant reduction of excess sludge production, Water Sci. Technol. 45 (12) (2002) 127–134.
[43] S. Hasegawa, N. Shiota, K. Katsura, A. Akashi, Solubilization of organic sludge by thermophilic aerobic bacteria as a pretreatment for anaerobic digestion, Water Sci. Technol. 41 (3) (2000) 163–169.
[44] Y. Sakai, T. Aoyagi, N. Shiota, A. Akashi, S. Hasegawa, Complete decomposition of biological waste sludge by thermophilic aerobic bacteria, Water Sci. Technol. 42 (9) (2000) 81–88.
[45] C. Dumas, S. Perez, E. Paul, X. Lefebvre, Combined thermophilic aerobic process and conventional anaerobic digestion: Effect on sludge biodegradation and methane production, Bioresour. Technol. 101 (8) (2010) 2629–2636.
[46] S. Déléris, A. Larose, V. Geaugey, T. Lebrun. Innovative strategies for the reduction of sludge production in activated sludge plant: BIOLYSIS® O and BIOLYSIS® E. in Biosolids 2003: Water Sludge as a Resource (2003). Trondheim (Norway).
[47] I.V. Skiadas, H.N. Gavala, J. Lu, B.K. Ahring, Thermal pre-treatment of primary and secondary sludge at 70 °C prior to anaerobic digestion, Water Sci. Technol. 52 (1–2) (2005) 161–166.
[48] R.T. Haug, D.C. Stuckey, J.M. Gossett, P.L. Mac Carty, Effect of thermal pretreatment on digestibility and dewaterability of organic sludges, J. Water Pol. Control Fed. (January) (1978) 73–85.
[49] D.C. Stuckey, P.L. Mc Carty, Thermochemical pretreatment of nitrogenous materials to increase methane yield, Biotechnol. Bioeng. Symp. (1978).
[50] S. Tanaka, T. Kobayashi, K.I. Kamiyama, L.N.S. Bildan, Effects of thermochemical pretreatment on the anaerobic digestion of waste activated sludge, Water Sci. Technol. 35 (8) (1997) 209–215.
[51] U. Kepp, I. Machenbach, N. Weisz, O.E. Solheim, Enhanced stabilisation of sewage sludge through thermal hydrolysis – three years of experience with full scale plant, Water Sci. Technol. 42 (9) (2000) 89–96.
[52] C. Fjordside, An operating tale from Næstved Sewage Treatment Plant, in: Municipal wastewater treatment Nordic Conference, Copenhagen (Denmark), 2001.
[53] M. Barjenbruch, O. Kopplow, Enzymatic, mechanical and thermal pretreatment of surplus sludge, Adv. Environ. Res. 7 (3) (2003) 715–720.
[54] J. Kim, C. Park, T.H. Kim, M. Lee, S. Kim, S.W. Kim, J. Lee, Effects of various pretreatments for enhanced anaerobic digestion with waste activated sludge, J. Biosci. Bioeng. 95 (3) (2003) 271–275.
[55] M. Dohanyos, J. Zabranska, J. Kutil, P. Jenicek, Improvement of anaerobic digestion of sludge, Water Sci. Technol. 49 (10) (2004) 89–96.
[56] A. Valo, H. Carrère, J.P. Delgenès, Thermal, chemical and thermo-chemical pretreatment of waste activated sludge for anaerobic digestion, J. Chem. Technol. Biotechnol. 79 (11) (2004) 1197–1203.
[57] S. Graja, J. Chauzy, P. Fernandes, L. Patria, D. Cretenot, Reduction of sludge production from WWTP using thermal pretreatment and enhanced anaerobic methanisation, Water Sci. Technol. 52 (1–2) (2005) 267–273.
[58] C. Bougrier, C. Albasi, J.P. Delgenès, H. Carrère, Effect of ultrasonic, thermal and ozone pre-treatments on waste activated sludge solubilisation and anaerobic biodegradability, Chem. Eng. Process. 45 (8) (2006) 711–718.
[59] C. Bougrier, J.P. Delgenes, H. Carrere, Combination of thermal treatments and anaerobic digestion to reduce sewage sludge quantity and improve biogas yield, Process Saf. Environ. Protect. 84 (B4) (2006) 280–284.
[60] J. Zabranska, M. Dohanyos, P. Jenicek, J. Kutil, J. Cejka, Mechanical and rapid thermal disintegration methods of enhancement of biogas production- Full scale applications, in: IWA Specialized conference: Sustainable sludge management: state of the art, challenges and perspectives, Moscow, Russia, 2006.
[61] J. Chauzy, Cretenot D, Bausseon A, and D. S. Anaerobic digestion enhanced by thermal hydrolysis: First reference BIOTHELYS® at Saumur, France. Facing sludge diversities: challenges, risks and opportunities (2007). Antalya, Turkey.
[62] F. Fernandez-Polanco, R. Velazquez, S.I. Perez-Elvira, C. Casas, D. del Barrio, F.J. Cantero, M. Fdz-Polanco, P. Rodriguez, L. Panizo, J. Serrat, P. Rouge, Continuous thermal hydrolysis and energy integration in sludge anaerobic digestion plants, Water Sci. Technol. 57 (8) (2008) 1221–1226.
[63] X. Yang, X. Wang, L. Wang, Transferring of components and energy output in industrial sewage sludge disposal by thermal pretreatment and two-phase anaerobic process, Bioresour. Technol. 101 (8) (2010) 2580–2584.
[64] C. Eskicioglu, K.J. Kennedy, R.L. Droste, Enhanced disinfection and methane production from sewage sludge by microwave irradiation, Desalination 248 (1–3) (2009) 279–285.
[65] J. Zheng, K.J. Kennedy, C. Eskicioglu, Effect of low temperature microwave pretreatment on characteristics and mesophilic digestion of primary sludge, Environ. Technol. 30 (4) (2009) 319–327.
[66] R.A. Fisher, S.J. Swanwick, High temperature treatment of sewage sludge, Water Pollut. Control 71 (3) (1971) 255–370.
[67] N.J. Anderson, D.R. Dixon, P.J. Harbour, P.J. Scales, Complete characterisation of thermally treated sludges, Water Sci. Technol. 46 (10) (2002) 51–54.

[68] C. Bougrier, J.P. Delgenès, H. Carrère, Effects of thermal treatments on five different waste activated sludge samples solubilisation, physical properties and anaerobic digestion, Chem. Eng. J. 139 (2) (2008) 236–244.
[69] M. Weemaes, W. Verstraete, Evaluation of current wet sludge disintegration techniques, J. Chem. Technol. Biotechn. 73 (8) (1998) 83–92.
[70] E. Neyens, J. Baeyens, A review of thermal sludge pre-treatment processes to improve dewaterability, J. Hazard. Mater. 98 (1–3) (2003) 51–67.
[71] H. Carrere, C. Bougrier, D. Castets, J.P. Delgenes, Impact of initial biodegradability on sludge anaerobic digestion enhancement by thermal pretreatment, J. Environ. Sci. Health Part A-Toxic/Hazard. Subst. Environ. Eng. 43 (13) (2008) 1551–1555.
[72] J. Dwyer, D. Starrenburg, S. Tait, K. Barr, D.J. Batstone, P. Lant, Decreasing activated sludge thermal hydrolysis temperature reduces product colour, without decreasing degradability, Water Res. 42 (18) (2008) 4699–4709.
[73] C. Eskicioglu, K.J. Kennedy, R.L. Droste, Initial examination of microwave pretreatment on primary, secondary and mixed sludges before and after anaerobic digestion, Water Sci. Technol. 57 (3) (2008) 311–317.
[74] A. Mottet, J.P. Steyer, S. Deleris, F. Vedrenne, J. Chauzy, H. Carrere, Kinetics of thermophilic batch anaerobic digestion of thermal hydrolysed waste activated sludge, Biochem. Eng. J. 46 (2) (2009) 169–175.
[75] D.J. Batstone, C. Balthes, K. Barr, Model Assisted Startup of Anaerobic Digesters Fed with Thermally Hydrolysed Activated Sludge, Water Sci. Technol, in press, doi:10.2166/wst.2010.487.
[76] K. Panter, H. Kleiven, Ten years experience of full scale thermal hydrolysis projects, in: 10th European Biosolids and Biowastes Conference, Wakefield, United Kingdom, 2005.
[77] S.I. Perez-Elvira, F. Fernandez-Polanco, M. Fernandez-Polanco, P. Rodriguez, P. Rouge, Hydrothermal multivariable approach. Full-scale feasibility study, Electron. J. Biotechnol. 11 (2008) 7–8.
[78] C. Eskicioglu, N. Terzian, K.J. Kennedy, R.L. Droste, M. Hamoda, Athermal microwave effects for enhancing digestibility of waste activated sludge, Water Res. 41 (11) (2007) 2457–2466.
[79] C.P. Chu, D.J. Lee, B.V. Chang, C.S. You, J.H. Tay, "Weak" ultrasonic pretreatment on anaerobic digestion of flocculated activated biosolids, Water Res. 36 (11) (2002) 2681–2688.
[80] M.R. Salsabil, A. Prorot, M. Casellas, C. Dagot, Pre-treatment of activated sludge: Effect of sonication on aerobic and anaerobic digestibility, Chem. Eng. J. 148 (2–3) (2009) 327–335.
[81] H. Li, Y.Y. Jin, R.B. Mahar, Z.Y. Wang, Y.F. Nie, Effects of ultrasonic disintegration on sludge microbial activity and dewaterability, J. Hazard. Mater. 161 (2–3) (2009) 1421–1426.
[82] K.-Y. Show, T. Mao, D.-J. Lee, Optimisation of sludge disruption by sonication, Water Res. 41 (20) (2007) 4741–4747.
[83] A. Gronroos, H. Kyllonen, K. Korpijarvi, P. Pirkonen, T. Paavola, J. Jokela, J. Rintala, Ultrasound assisted method to increase soluble chemical oxygen demand (SCOD) of sewage sludge for digestion, Ultrason. Sonochem. 12 (1–2) (2005) 115–120.
[84] S. Vaxelaire, E. Gonze, G. Merlin, Y. Gonthier, Reduction by sonication of excess sludge production in a conventional activated sludge system: Continuous flow and lab-scale reactor, Environ. Technol. 29 (12) (2008) 1307–1320.
[85] Y.U. Kim, B.I. Kim, Effect of ultrasound on dewaterability of sewage sludge, Japan J. Appl. Phys. Part 1 42 (9A) (2003) 5898–5899.
[86] X. Feng, H.Y. Lei, J.C. Deng, Q. Yu, H.L. Li, Physical and chemical characteristics of waste activated sludge treated ultrasonically, Chem. Eng. Process. 48 (1) (2009) 187–194.
[87] B. Wunsch, W. Heine, U. Neis, Combating bulking sludge with ultrasound, in: U. Neis (Ed.), Ultrasound in Environmental Engineering II, 2002.
[88] U. Neis, K. Nickel, A. Lunden, Improving anaerobic and aerobic degradation by ultrasonic disintegration of biomass, J. Environ. Sci. Health Part A-Toxic/Hazard. Subst. Environ. Eng. 43 (13) (2008) 1541–1545.
[89] R. Xie, Y. Xing, Y.A. Ghani, K.E. Ooi, S.W. Ng, Full-scale demonstration of an ultrasonic disintegration technology in enhancing anaerobic digestion of mixed primary and thickened secondary sewage sludge, J. Environ. Eng. Sci. 6 (5) (2007) 533–541.
[90] A. Tiehm, K. Nickel, U. Neis, The use of ultrasound to accelerate the anaerobic digestion of sewage sludge, Water Sci. Technol. 36 (11) (1997) 121–128.
[91] Q. Wang, M. Kuninobo, K. Kakimoto, H.I. Ogawa, Y. Kato, Upgrading of anaerobic digestion of waste activated sludge by ultrasonic pretreatment, Bioresour. Technol. 68 (1999) 309–313.
[92] A. Tiehm, K. Nickel, M. Zellhorn, U. Neis, Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization, Water Res. 35 (8) (2001) 2003–2009.
[93] T.I. Onyeche, O. Schlafer, H. Bormann, C. Schroder, M. Sievers, Ultrasonic cell disruption of stabilised sludge with subsequant anaerobic digestion, Ultrasonics 40 (2002) 31–35.
[94] J.B. Bien, G. Malina, J.D. Bien, L. Wolny, Enhancing anaerobic fermentation of sewage sludge for increasing biogas generation, J. Environ. Sci. Health Part A-Toxic/Hazard. Subst. Environ. Eng. 39 (4) (2004) 939–949.
[95] C. Bougrier, H. Carrère, J.P. Delgenès, Solubilisation of waste-activated sludge by ultrasonic treatment, Chem. Eng. J. 106 (2) (2005) 163–169.
[96] C.M. Braguglia, G. Mininni, A. Gianico, Is sonication effective to improve biogas production and solids reduction in excess sludge digestion? Water Sci. Technol. 57 (4) (2008) 479–483.
[97] G. Erden, A. Filibeli, Ultrasonic pre-treatment of biological sludge: consequences for disintegration, anaerobic biodegradability, and filterability, J. Chem. Technol. Biotechnol. 85 (1) (2009) 145–150.
[98] S. Perez-Elvira, M. Fdz-Polanco, F.I. Plaza, G. Garralon, F. Fdz-Polanco, Ultrasound pre-treatment for anaerobic digestion improvement, Water Sci. Technol. 60 (6) (2009) 1525–1532.
[99] M. Dohanyos, J. Zabranska, P. Jenicek, Enhancement of sludge anaerobic digestion by using of a special thickening centrifuge, Water Sci. Technol. 36 (11) (1997) 145–153.
[100] J. Zabranska, M. Dohanyos, P. Jenicek, J. Kutil, Disintegration of excess activated sludge – evaluation and experience of full-scale applications, Water Sci. Technol. 53 (12) (2006) 229–236.
[101] H.B. Choi, K.Y. Hwang, E.B. Shin, Effect on anerobic digestion of sewage sludge pretreatment, Water Sci. Technol. 35 (10) (1997) 207–211.
[102] I.W. Nah, Y.W. Kang, K.Y. Hwang, W.K. Song, Mechanical pretreatment of waste activated sludge for anaerobic digestion process, Water Res. 34 (8) (2000) 2362–2368.
[103] J. Muller, L. Pelletier, Désintégration mécanique des boues activées, L'eau, l'industrie, les nuisances (217) (1998) 61–66.
[104] M. Engelhart, M. Krüger, J. Kopp, N. Dichtl, Effects of disintegration on anaerobic degradation of sewage sludge in downflow stationary fixed film digester, in: II International Symposium on Anaerobic Digestion of Solid Waste, Barcelona, 1999.
[105] T.I. Onyeche, Economic benefits of low pressure sludge homogenization for wastewater treatment plants, in: IWA specialist conferences. Moving forward wastewater biosolids sustainability, Moncton, New Brunswick, Canada, 2007.
[106] Biogest, 2010/03/23.Available from: http://www.biogest.com/.
[107] Ecosolids, 2010/03/23.Available from: http://www.ecosolids.com/.
[108] R.J. Stephenson, S. Laliberte, P.M. Hoy, D. Britch, Full scale and laboratory scale results from the trial of microsludge at the joint water pollution control plant at Los Angeles County, in: IWA Specialist Conferences. Moving Forward Wastewater Biosolids Sustainability, Moncton, New Brunswick, Canada, 2007.
[109] U. Baier, P. Schmidheiny, Enhanced anaerobic degradation of mechanically disintegrated sludge, Water Sci. Technol. 36 (11) (1997) 137–143.
[110] J. Kopp, J. Müller, N. Dichtl, J. Schwedes, Anaerobic digestion and dewatering characteristics of mechanically disintegrated sludge, Water Sci. Technol. 36 (11) (1997) 129–136.
[111] M. Weemaes, H. Grootaerd, F. Simoens, W. Verstraete, Anaerobic digestion of ozonized biosolids, Water Res. 34 (8) (2000) 2330–2336.
[112] I.T. Yeom, K.R. Lee, Y.H. Lee, K.H. Ahn, S.H. Lee, Effects of ozone treatment on the biodegradability of sludge from municipal wastewater treatment plants, Water Sci. Technol. 46 (4–5) (2002) 421–425.
[113] R. Goel, T. Tokutomi, H. Yasui, T. Noike, Optimal process configuration for anaerobic digestion with ozonation, Water Sci. Technol. 48 (4) (2003) 85–96.
[114] A. Battimelli, C. Millet, J.P. Delgenès, R. Moletta, Anaerobic digestion of waste activated sludge combined with ozone post-treatment and recycling, Water Sci. Technol. 48 (4) (2003) 61–68.
[115] C. Bougrier, A. Battimelli, J.P. Delgenes, H. Carrere, Combined ozone pretreatment and anaerobic digestion for the reduction of biological sludge production in wastewater treatment, Ozone-Sci. Eng. 29 (3) (2007) 201–206.
[116] J.A.C. Rivero, N. Madhavan, M.T. Suidan, P. Ginestet, J.M. Audic, Enhancement of anaerobic digestion of excess municipal sludge with thermal and/or oxidative treatment, J. Environ. Eng. -ASCE 132 (6) (2006) 638–644.
[117] J.J. Song, N. Takeda, M. Hiraoka, Anaerobic treatment of sewage treated by catalytic wet oxidation process in upflow anaerobic blanket reactors, Water Sci. Technol. 26 (3–4) (1992) 867–875.
[118] C. Bougrier, A. Battimelli, J.P. Delgenès, H. Carrère, Combined ozone pretreatment and anaerobic digestion for the reduction of biological sludge production in wastewater treatment, Ozone-Sci. Eng. 29 (3) (2007) 201–206.
[119] Y. Sakai, T. Fukasu, H. Yasui, M. Shibata, An activated sludge process without excess sludge production, Water Sci. Technol. 36 (11) (1997) 163–170.
[120] L.B. Chu, S.T. Yan, X.H. Xing, X.L. Sun, B. Jurcik, Progress and perspectives of sludge ozonation as a powerful pretreatment method for minimization of excess sludge production, Water Res. 43 (7) (2009) 1811–1822.
[121] E. Paul, P. Camacho, M. Spérandio, P. Ginestet, Technical and economical evaluation of a thermal, and two oxidative techniques for the reduction of excess sludge production, in: 1st International Conference on Engineering for Waste Treatment, Albi (France), 2005.
[122] G.E. Kaynak, A. Filibeli, Assessment of Fenton process as a minimization technique for biological sludge: Effects on anaerobic sludge bioprocessing, J. Residuals Sci. Technol. 5 (3) (2008) 151–160.
[123] A.H. Mouneimne, H. Carrère, N. Bernet, J.P. Delgenès, Effect of saponification on the anaerobic digestion of solid fatty residues, Bioresour. Technol. 90 (1) (2003) 89–94.
[124] I. Dogan, F.D. Sanin, Alkaline solubilization and microwave irradiation as a combined sludge disintegration and minimization method, Water Res. 43 (8) (2009) 2139–2148.
[125] J.G. Everret, The effect of pH on the heat treatment of sewage sludges, Water Res. 8 (1974) 899–906.
[126] C. Park, C. Lee, S. Kim, Y. Chen, H.A. Chase, Upgrading of anaerobic digestion by incorporating two different hydrolysis processes, J. Biosci. Bioeng. 100 (2) (2005) 164–167.
[127] N.H. Heo, S.C. Park, J.S. Lee, H. Kang, Solubilization of waste activated sludge by alkaline pretreatment and biochemical methane potential (BMP) tests for anaerobic co-digestion of municipal organic waste, Water Sci. Technol. 48 (8) (2003) 211–219.

[128] A.G. Vlyssides, P.K. Karlis, Thermal alkaline solubilization of waste activated sludge as pre-treatment of anaerobic digestion, Bioresour. Technol. 91 (2004) 201–206.
[129] M.R. Salsabil, J. Laurent, M. Casellas, C. Dagot, Techno-economic evaluation of thermal treatment, ozonation and sonication for the reduction of wastewater biomass volume before aerobic or anaerobic digestion, J. Hazard. Mater. 174 (1–3) (2010) 323–333.
[130] J.A. Muller, A. Winter, G. Strunkmann, Investigation and assessment of sludge pre-treatment processes, Water Sci. Technol. 49 (10) (2004) 97–104.
[131] P.F. Greenfield, D.J. Batstone, Anaerobic digestion: impact of future greenhouse gases mitigation policies on methane generation and usage, Water Sci. Technol. 52 (1–2) (2005) 39–47.
[132] G. Tchobanoglous, F. Burton, H. Stensel, Metcalf and Eddy Inc. Wastewater Engineering, Treatment and Reuse, New York, NY (US) McGraw-Hill, 2003.
[133] R.E. Speece, Anaerobic Biotechnology and Odor/Corrosion Control for Municipalities and Industries 2 ed, Tennessee Archae Press, Nashville, 2008.
[134] M. Boehler, H. Siegrist, Potential of activated sludge disintegration, Water Sci. Technol. 53 (12) (2006) 207–216.
[135] T.I. Onyeche, S. Schafer, Energy production and savings from sewage sludge treatment, in: Biosolids: Wastewater Sludge as a Resource, Trondheim (Norway), 2003.
[136] K.G. Barr, D.O. Solley, D.J. Starrenburg, R.G. Lewis, Evaluation, selection and initial performance of a large scale centralised biosolids facility at Oxley Creek Water Reclamation Plant, Brisbane, 2008, pp. 1579–1586.
[137] G.D. Zupancic, M. Ros, Two stage thermophilic sludge digestion - solids degradation, heat and energy considerations, in: Biosolids: Wastewater Sludge as a Resource, Trondheim (Norway), 2003.
[138] C. Bougrier, J.P. Delgenes, H. Carrere, Impacts of thermal pre-treatments on the semi-continuous anaerobic digestion of waste activated sludge, Biochem. Eng. J. 34 (1) (2007) 20–27.
[139] S. Watts, G. Hamilton, J. Keller, Two-stage thermophilic-mesophilic anaerobic digestion of waste activated sludge-from a biological nutrient removal plant, Water Sci. Technol. 53 (8) (2006) 149–157.
[140] J.A. Muller, Pretreatment processes for the recycling and reuse of sewage sludge, Water Sci. Technol. 42 (9) (2000) 167–174.
[141] X.X. Wang, Z.F. Qiu, S.G. Lu, W.C. Ying, Characteristics of organic, nitrogen and phosphorus species released from ultrasonic treatment of waste activated sludge, J. Hazard. Mater. 176 (1–3) (2010) 35–40.
[142] G.Q. Yin, P.H. Liao, K.V. Lo, An ozone/hydrogen peroxide/microwave-enhanced advanced oxidation process for sewage sludge treatment, J. Environ. Sci. Heal. A. 42 (8) (2007) 1177–1181.
[143] M. Boehler, H. Siegrist, Potential of activated sludge ozonation, Water Sci. Technol. 55 (12) (2007) 181–187.
[144] M.H. He, C.H. Wei, Performance of membrane bioreactor (MBR) system with sludge Fenton oxidation process for minimization of excess sludge production, J. Hazard. Mater. 176 (1–3) (2010) 597–601.
[145] M.A. Dytczak, K.L. Londry, H. Siegrist, J.A. Oleszkiewicz, Ozonation reduces sludge production and improves denitrification, Water Res. 41 (3) (2007) 543–550.
[146] M. Salhi, S. Deleris, H. Debellefontaine, P. Ginestet, E. Paul, More insights into the understanding of reduction of excess sludge production by ozone, in: Biosolids: Wastewater Sludge as a Resource, Trondheim (Norway), 2003.
[147] G.M. Zhang, P.Y. Zhang, J.M. Yang, Y.M. Chen, Ultrasonic reduction of excess sludge from the activated sludge system, J. Hazard. Mater. 145 (3) (2007) 515–519.
[148] Y.H. Ahn, Sustainable nitrogen elimination biotechnologies: A review, Process Biochem. 41 (8) (2006) 1709–1721.
[149] G.K. Morse, S.W. Brett, J.A. Guy, J.N. Lester, Review: Phosphorus removal and recovery technologies, Sci. Total Environ. 212 (1) (1998) 69–81.
[150] K.S. Le Corre, E. Valsami-Jones, P. Hobbs, S.A. Parsons, Phosphorus recovery from wastewater by struvite crystallization: A review, Crit. Rev. Environ. Sci. Technol. 39 (6) (2009) 433–477.
[151] J. Suschka, A. Machnicka, K. Grubel, Surplus activated sludge disintegration for additional nutrients removal, Arch. Environ. Prot. 33 (2) (2007) 55–65.
[152] N. Marti, J. Ferrer, A. Seco, A. Bouzas, Optimisation of sludge line management to enhance phosphorus recovery in WWTP, Water Res. 42 (18) (2008) 4609–4618.
[153] N. Marti, L. Pastor, A. Bouzas, J. Ferrer, A. Seco, Phosphorus recovery by struvite crystallization in WWTPs: Influence of the sludge treatment line operation, Water Res. 44 (7) (2010) 2371–2379.
[154] C. Zhang, Y.G. Chen, Simultaneous nitrogen and phosphorus recovery from sludge-fermentation liquid mixture and application of the fermentation liquid to enhance municipal wastewater biological nutrient removal, Environ. Sci. Technol. 43 (16) (2009) 6164–6170.
[155] L. Pastor, D. Mangin, J. Ferrer, A. Seco, Struvite formation from the supernatants of an anaerobic digestion pilot plant 101 (1) (2010) 118–125.
[156] P.H. Liao, D.S. Mavinic, F.A. Koch, Release of phosphorus from biological nutrient removal sludges: A study of sludge pretreatment methods to optimize phosphorus release for subsequent recovery purposes, J. Environ. Eeng. Sci. 2 (5) (2003) 369–381.
[157] W.T. Wong, W.I. Chan, P.H. Liao, K.V. Lo, A hydrogen peroxide/microwave advanced oxidation process for sewage sludge treatment 41 (11) (2006) 2623–2633.
[158] A.A. Kenge, P.H. Liao, K.V. Lo, Factors affecting microwave-enhanced advanced oxidation process for sewage sludge treatment, J. Environ. Sci. Heal. A 44 (11) (2009) 1069–1076.
[159] T. Kondo, S. Tsuneda, Y. Ebie, Y. Inamori, K. Xu, Improvement of nutrient removal and phosphorus recovery in the anaerobic/oxic/anoxic process combined with sludge ozonation and phosphorus adsorption, J. Water Environ. Technol. 7 (2) (2009) 135–142.
[160] N. Takiguchi, M. Kishino, A. Kuroda, J. Kato, H. Ohtake, A laboratory-scale test of anaerobic digestion and methane production after phosphorus recovery from waste activated sludge, J. Biosci. Bioeng. 97 (6) (2004) 365–368.