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Invited article

An executive review of sludge pretreatment by sonication

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ABSTRACT

Ultrasonication (US), which creates hydro-mechanical shear forces in cavitation, is an advanced technology in sludge pretreatment. However, there are many factors affecting the efficacy of cavitation and ultrasonication disintegration of sludge as a consequence. The objective of this work is to present an extensive review of evaluation approaches of sludge US pretreatment efficiency. Besides, optimization methodologies of related parameters, the differences of optimum values and the similarities of affecting trends on cavitation and sludge pretreatment efficiency were specifically pointed out, including ambient conditions, ultrasonic properties, and sludge characteristics. The research is a prerequisite for optimization of sludge US pretreatment efficiency in lab-scale and practical application. There is not-yet a comprehensive method to evaluate the efficiency of sludge US pretreatment, but some main parameters commonly used for this purpose are degree of sludge disintegration, proteins, particle size reduction, *etc.* Regarding US parameters, power input P_{US}, intensity I_{US}, and frequency F_S seem to have significant effects. However, the magnitude of the effect of P_{US} and probe size in terms of I_{US} has not been clearly detailed. Investigating very low F_S seems interesting but has not yet been taken into consideration. In addition, static pressure effect has been marginally studied only and investigation on the effect of pH prior to US process has been restricted. Their effects therefore should be varied separately and simultaneously with other related parameters, *i.e.* process conditions, ultrasonic properties, and sludge characteristics, to optimize sludge US pretreatment process.

Introduction

Anaerobic digestion of sludge, an efficient technology for sludge treatment, facilitating mass reduction, odor removal, pathogen decrease, and energy recovery in the form of methane, is a complex and slow process. Hydrolysis is known as the rate-limiting step, and thus requires a pretreatment of sludge which ruptures the cell wall and facilitates the release of intracellular matter into the aqueous phase to improve biodegradability and enhance anaerobic digestion.

There are some very popular techniques used in sludge pretreatment, such as biological, thermal, mechanical, chemical, and electrical methods (Carrère et al., 2010; Kopplow et al., 2004; Rittmann et al., 2008; Salerno et al., 2009; Keles et al., 2010; Mahmoud et al., 2010; Pham, 2011; Rynkiewicz, 2011). In their review, Pilli et al. (2011) claimed ultrasonication (US) to be a

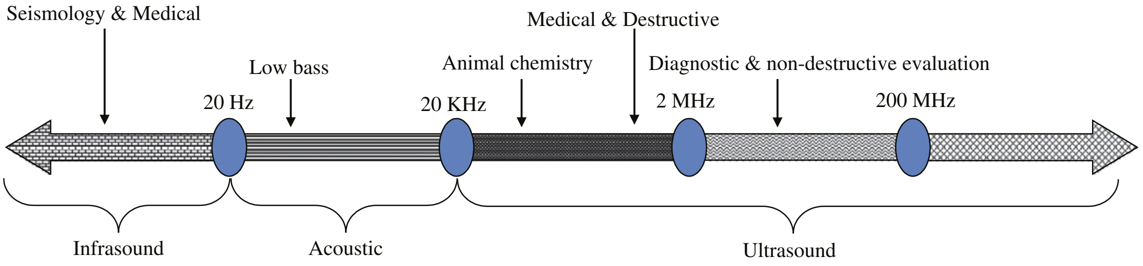

Fig. 1 – Diagram of sonication range (Pilli et al., 2011).

feasible and promising mechanical disruption technique for sludge disintegration and microorganism lysis according to the treatment time and power, equating to specific energy input (ES). Some positive characteristics of this method are efficient sludge disintegration (Pilli et al., 2011), improvement in biodegradability and bio-solid quality (Khanal et al., 2007), increase in biogas/ methane production (Onyeche et al., 2002; Barber, 2005; Khanal et al., 2007), no need for chemical additives (Mao et al., 2004), less sludge retention time (Tiehm et al., 1997), and sludge reduction (Onyeche et al., 2002).

This article presents an extensive review of sludge pretreatment by sonication, including sludge types and properties, brief background of sonication, evaluation approaches of sludge US pretreatment efficiency, and optimization of ultrasonic pretreatment of sludge.

1. Sludge types

1.1. *Primary sludge*

Primary sludge was produced through the mechanical wastewater treatment process, is decayable and must be stabilized before being disposed of (Liu and Liptak, 1999). Primary sludge is easily biodegradable since it consists of more easily digestible carbohydrates and fats. Biogas therefore is more easily produced but the methane content of the gas is lower.

1.2. *Waste activated sludge (WAS)*

WAS coming from the secondary wastewater treatment, consists largely of biological mass and large amount of pathogens, causes odor problems, and thus must be stabilized (Lin et al., 1999). Activated sludge is more difficult to digest than primary sludge.

1.3. *Digested sludge*

The residual product after anaerobic digestion of primary and activated sludge, is reduced in mass, less odorous, and safer in the aspect of pathogens and easier dewatered than the primary and activated sludge types (Liu and Liptak, 1999).

2. Brief background of sonication

The diagram of sonication range is presented in Fig. 1. When propagating in a solution, ultrasound waves generate compressions and rarefactions. If a sufficiently large negative pressure is applied during rarefaction, acoustic cavitation will

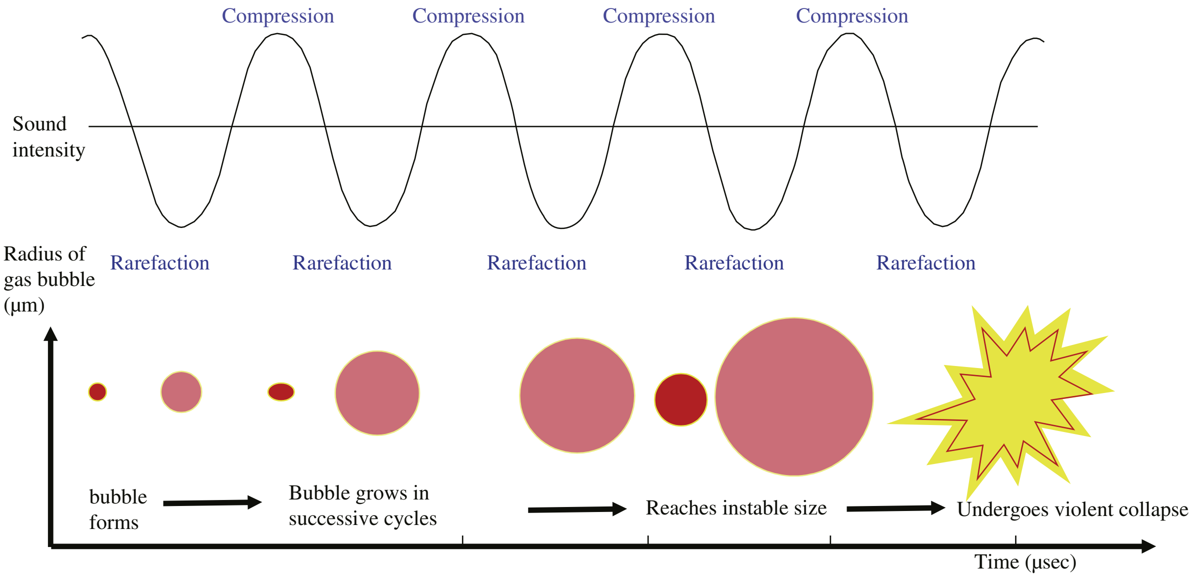

Fig. 2 – Formation and collapse press of a cavity.

Table 1 – Expressions of ultrasonication energy for sludge disintegration.

No.	Parameter	Expression	Reference
1	Specific energy input (J/kg$_{TS}$)	$ES = \frac{P_{US} \times T}{V \times TS}$	Feng et al. (2009a)
2	Ultrasonic dose (J/L)	$DO_{US} = \frac{P_{US} \times T}{V}$	Tiehm et al. (2001)
3	Ultrasonic density (W/L)	$D_{US} = \frac{P_{US}}{V}$	Tiehm et al. (2001)
4	Ultrasonic intensity (W/cm^2)	$I_{US} = \frac{P_{US}}{A}$	Neis et al. (2000)

take place, thereby results in most of US outstanding effects. Micro-bubbles are generated from nuclei, favored by dissolved gas, wall defects, and liquid impurities during the low pressure half periods (bubble formation and expansion). They may oscillate a few periods, undergo a slow average growth due to the so-called "rectified diffusion" process (up to several μm), suddenly reach a critical size, dramatically grow during the low pressure half period, and violently collapse in a very short fraction of the high pressure half period. Most often the bubble breaks up after the collapse point, giving smaller bubbles ready to reproduce the same scenario: oscillatory growth, driven by rectified diffusion, then sudden collapse (as schematized in Fig. 2).

Such a fast collapse being nearly adiabatic gives rise to extreme conditions inside and around the collapsing bubble. Modeling and experimental validations suggest that final collapse leads to a temperature as high as 5000 °K at the bubble center, a pressure of 500 bars, and a high radial velocity — up to the sound speed — then shock waves at the bubble rebound. These cavitation characteristics have different impacts on the sonicated media: high temperature peaks produce very active free radicals (mainly $^{\cdot}OH$ in aqueous media), giving the way to intense radical chemistry either inside or at the interface of the cavitation bubble depending on the volatility of the target dissolved molecules. On the other hand, high pressure, high velocity gradients, and shock waves have mainly physical effects through very strong microturbulence and intense local mixing, increasing heat and mass transfer. These physical effects are even more efficient in multiphase systems and especially on solid surfaces due to asymmetrical collapse with projection of a very fast jet toward the solid close to cavitation bubbles. This is the main cause of ultrasonic cleaning and also of most of ultrasonic solid processing, such as sludge disintegration.

When applied to solid suspension and especially for sludge treatment the power/energy may be expressed in many ways as given in Table 1.

Wang et al. (2005) indicated that the mechanisms implied in US sludge disintegration are hydro-mechanical shear forces, oxidizing effect of $^{\cdot}OH$, $H^{\cdot}$, $N^{\cdot}$, and $O^{\cdot}$ produced under US, and thermal decomposition of volatile hydrophobic substances in the sludge due to the increase in temperature during sonication. The effect of hydro-mechanical shear forces is nevertheless much higher than that of radicals.

3. Evaluation approaches of sludge ultrasonic pretreatment efficiency

Ultrasonic irradiation is a feasible and promising mechanical disruption technique for sludge disintegration, biodegradation acceleration, and anaerobic digestion enhancement. Ultrasonic cell lysis was first studied at lab-scale in the 1960s, but it was initially found uneconomical due to limitations of the US equipment at that time (Roxburgh et al., 2006). In the last 15 years, researches on US application for sludge disintegration have developed, as illustrated in the works by Chiu et al. (1997), Tiehm et al. (1997, 2001), Wang et al. (1999), Neis et al. (2000), Chu et al. (2002), Onyeche et al. (2002), Gonze et al. (2003), Bougrier et al. (2006), *etc*. Advances in US technology in the last decade have enabled commercial applications, especially for wastewater treatment. Fig. 3 depicts options for installation of US systems in waste water treatment plant (Ultrawaves GmbH — Water & Environmental Technologies, 2013).

Ultrawaves and Sonix™, whose configurations are described in Fig. 4, have the largest number of full-scale trials and full-scale installations in wastewater treatment, *i.e.* over 30 installations in Europe, the United States, Asia, and Australia. Ultrawaves is a commercial business born from the research

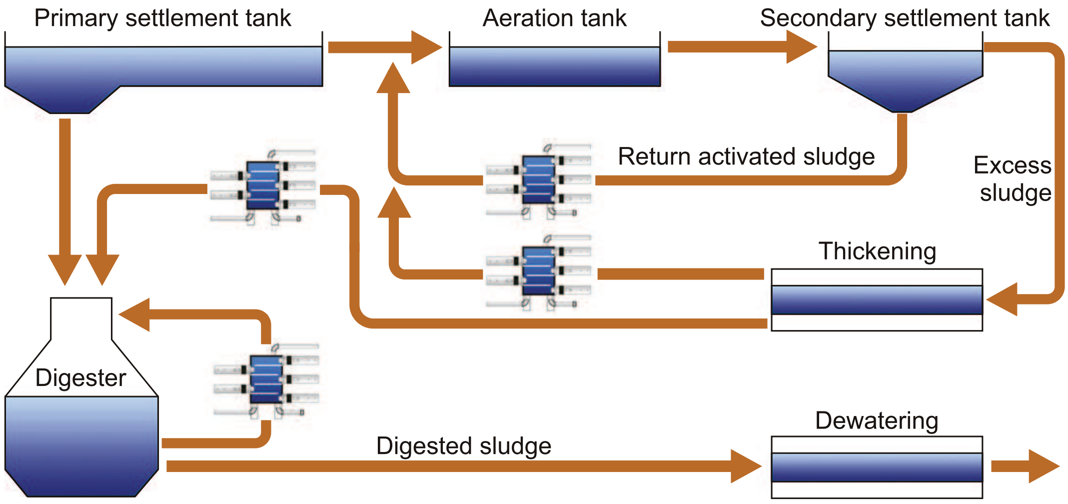

Fig. 3 – Integration of the ultrasonication technology in waste water treatment plant (WWTP) (Ultrawaves GmbH — Water & Environmental Technologies, 2013).

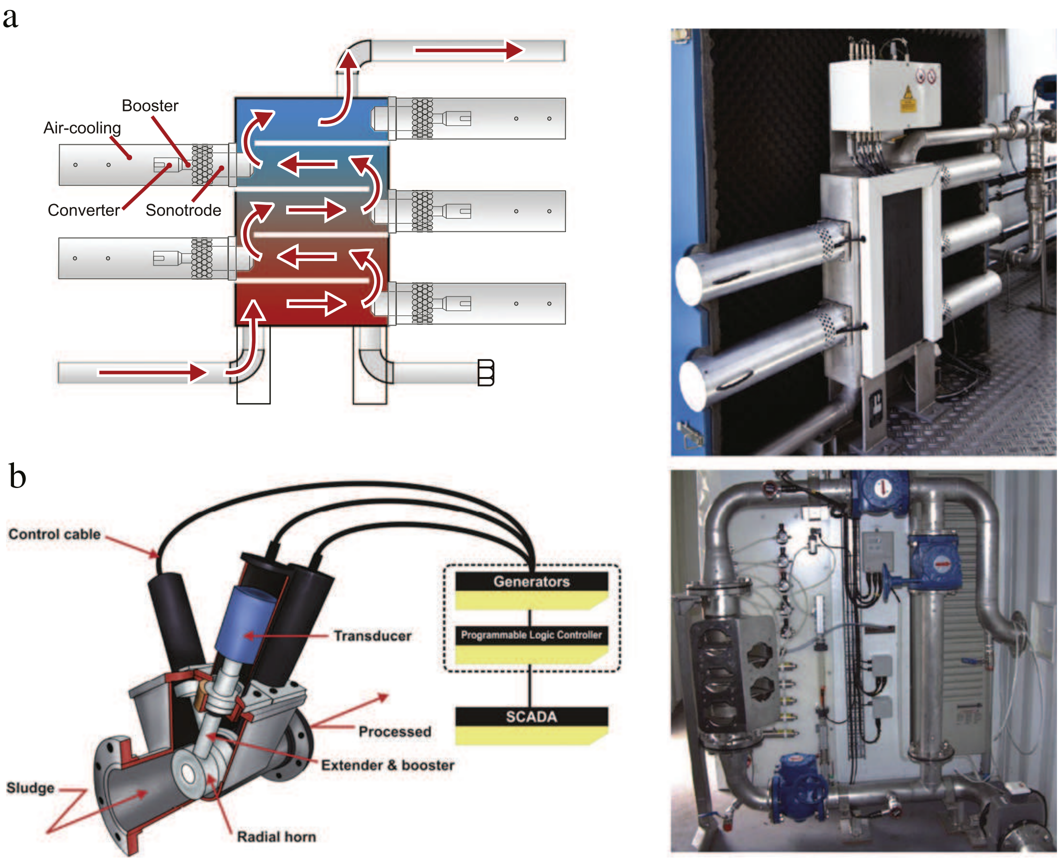

Fig. 4 – Configurations of (a) ultrawaves and (b) Sonix™ reactor.

activities at the Technical University of Hamburg–Harburg, has different trademarks such as Eimco Sonolyser, Dumo, Euro-open KFT, Sonoflux (sold by Stereau in France), *etc.* Sonix™ technology is supplied under licence from Sonico, a joint venture company between Purac Ltd. and Atkins Water. Sonotronic Nagel is a worldwide provider and manufacturer of ultrasonic equipment serving a variety of industries for the last 30 years. Sonolyzer technology is the product of years of development between Ultrawaves and Sonotronic Nagel. For WAS pretreatment, US installations have been applied in many WWTP, especially in Germany, since 2000 with different capacities (Table 2). In general, US system has been operated at 20 kHz and P_{US} up to 48 kHz. According to Roxburgh et al. (2006), the largest installation is at Mangere WWTP in New Zealand, from Sonico.

Some achievements from Sonix™ (a high-power US system for conditioning sludge) have been reported. For instance, TS and VS reduction in digesters were 40% and 50%, respectively for untreated sludge and 60% and 70%, respectively, for sonicated sludge (Hogan et al., 2004). Xie et al. (2007) showed an increase in biogas production of 15%–58% (average of 45%) in the full-scale US installation for mixed sludge treatment. For the full-scale part-stream US plants in Germany, Austria, Switzerland, Italy, and Japan, biogas, VS reduction, and sludge dewaterability were increased by 20%–50% (volume/kg fed), 20%–50%, and 3%–7%, respectively (Barber, 2005).

It is clear that many processing factors significantly affect cavitation and consequently the efficiency of sludge pretreatment. Therefore, assessment, comparison, and selection of optimal ultrasonic conditions for actual application of sludge pretreatment are sorely necessary. An extensive review of approaches to evaluate sludge ultrasonic pretreatment efficiency is presented with regard to changes in the following aspects.

(1) Physical properties: particle size, sludge mass and volume reduction, dewaterability, settleability, turbidity, and microscopic examination.
(2) Chemical properties: increase in soluble chemical oxygen demand (SCOD), nucleic acids, proteins, polysaccharides, release of NH_3, total organic carbon (TOC), *etc.*
(3) Biological properties: heterotrophic count and specific oxygen uptake rate.

3.1. Physical change-based evaluation of sludge ultrasonication (US) pretreatment efficiency

3.1.1. Particle size reduction

Ultrasonication pretreatment is very effective in reducing the particle size of sludge particles. The efficiency of size reduction depends on US parameters and sludge characteristics.

The floc size reduction improves (sludge disintegration efficiency also improves) with the increase in both P_{US} and D_{US} (Show et al., 2007; Pilli et al., 2011), *e.g.* 60% and 73% at 2 and 4 W/mL, respectively (Mao et al., 2004). Chu et al. (2001)

Table 2 – Full scale ultrasonication applications.

	waste water treatment plant (WWTP)	Country	Capacity (PE)	US system	Application year	Substrate/stage	Reference
1	Heiligenstadt	Germany	52000	Ultrawaves (20 kHz, 5 generators, 5 kW/ generator, V = 29 L)	2003	Return sludge (for aerobic stabilization)	Ultrawaves — Royce Water Technologies
2	Leinetal	Germany	50000		2003		
3	Tanba City	Japan			2004		
4	Bamberg	Germany	230000		2004	Primary and Thickened WAS for anaerobic digestion	Rossier et al., 2007; Ultrawaves — Royce Water Technologies
5	Meldorf	Germany	70000		2004	(for anaerobic digestion)	
6	Zeist	Netherlands	75000		2005		
7	Hennef	Germany	65000		2006		
8	Kleinsteinbach	Germany	40000		2006		Ultrawaves — Royce Water Technologies
9	Marselisborg-Arhus	Denmark	220000		2006		
10	Pecs	Hungary	200000		2006	Return sludge (for aerobic stabilization)	
11	Datansha	China	550000		2006		
12	Bath	England	550000		2006		Rossier et al. (2007)
13	Slupsk	Germany	250000		2007		
14	Detmold	Germany	95000	DMS, 14 Kw	2000	Mixed sludge	Rossier et al. (2007)
15	Mannheim	Germany	725000	DMS, 24 kW	2001	Primary: WAS = 1:1, for anaerobic digestion	Bartholomew (2002); Rossier et al. (2007)
16	Russelsheim	Germany	80000	DMS, 10 kW	2001	Mixed sludge	
17	Wiesbaden	Germany	360000	DMS, 48 kW	2002	Mixed sludge	
18	Kavlinge	Sweden	100000	Sonix™, 3–6 kW	2002	WAS	Rossier et al. (2007)
19	Mangere	New Zealand	800000	Sonix™	2005	WAS	
20	Rzeszow	Poland	220000	VTA GSD	2003		Rossier et al. (2007)
21	Villach	Austria	200000		2003		
22	Eberstadt	Germany	200000		2003	Primary: WAS = 1/3:2/3	
23	Zemtralklarwerk Darmstadt	Germany	240000		2004		
24	Halle Nord	Germany	300000		2004		
25	GroBostheim	Germany	35000		2004		
26	Kitzbuhel	Austria	46500		2005		
27	Winsen/Luhe	Germany	50000		2005		
28	Penthaz	Switzerland	10000		2006		
29	Obersee	Germany	25000		2006		
30	Sud	Germany	40000	6 kW	2000	WAS for anaerobic digestion	Bartholomew (2002)
31	Darmstadt	Germany	180000	16 kW	2000	Primary: WAS = 1/3:2/3, for anaerobic digestion	

WAS: waste activated sludge.

showed that after 40 min US at 0.11 W/mL, the architecture of flocs was basically the same as that of the raw sludge. Meanwhile, the structural integrity of flocs was almost completely broken down after 40 min US at 0.33 W/mL. Thereby, there is a critical P_{US} value beyond which the sludge flocs could be sufficiently disintegrated.

Besides, the particle size also reduces owing to the increase in US duration (Tiehm et al., 1997; Show et al., 2007), but beyond 10 min of sonication, it can exhibit a reverse trend (Gonze et al., 2003) due to re-flocculation of the particles. However, this phenomenon was not recorded by Show et al. (2007) even after 20 min of sonication.

In terms of ES, 1000 kJ/kg_{TS} may be the disruption threshold of usual fls (Feng et al., 2009a). Following the increase in ES, US causes a decrease in particle size (Tiehm et al., 2001; Gonze et al., 2003; Feng et al., 2009a). For example, the volume occupied by particles of less than 1 μm increased from 0.1% in the raw sludge to 1.5% in the pretreated one at ES of 14,550 kJ/kg_{TS} (Bougrier et al., 2005). Mean particle size of sludge decreased from 33.8 μm to 10.1–13.3 μm when ES increase in the range of 0–15,000 kJ/kg_{TS} (El-Hadj et al., 2007).

Show et al. (2007) and Na et al. (2007) agreed that flocs above 4.4 μm showed more disruption probability as they exhibit a larger surface area and less strong binding forces.

With regard to the sludge type, the particles of flocculated sludge in anaerobic digestion were reduced by more than 50% in size after US compared to those of raw sludge (Chu et al., 2002). Similarly, within 20 min of sonication, the disintegration was more significant in secondary sludge (85%) than in primary sludge (71%) because the former contains mostly biomass (microbial cells) whereas the latter mainly consists of settle-able solids (fibers and less degradable cellulosic material) (Mao et al., 2004).

For sludge TS concentration, the size reduced more in lower TS sample. d_{50} of sludge with 2% TS decreased by 6.5 fold at 0.67 W/mL. Higher TS concentrations (4% and 6%) required more D_{US} (0.83 W/mL and 1.03 W/mL, respectively) to reach the same level of particle size reduction (Akin et al., 2006).

In short, US pretreatment significantly decreases the particle size of sludge, especially in the very first period of sonication. Sludge particle size reduction is sometimes used to assess the degree of sludge disintegration.

3.1.2. *Sludge mass reduction or solubilization*

The sludge mass reduction results mainly from solubilization of the organic matters and is usually measured by the decrease in the suspended solid (SS) concentration. During US (0–30 min, 0.5 W/mL, 9.945 g_{SS}/L of raw sludge), SS reduction increase was almost linear with US duration, indicating the continuous and stable sludge floc disintegration, mass reduction, and cell lysis (Zhang et al., 2007). This parameter was also presented as matter solubilization in the work by Bougrier et al. (2006).

Apart from SS concentration, total dissolved solids also reflect the mass transfer from the solid into the aqueous phase. Feng et al. (2009a) proved the amount of soluble matters in the supernatant to be strongly affected by US, *e.g.* in ES range of 500–26,000 kJ/kg_{TS}, the increase in total dissolved solids was 3%–46% as compared to untreated sludge.

Other parameters used to assess the sludge reduction, subsequently the efficiency of sludge US disintegration, were the solubilization of total solids (S_{TS}) and of volatile solids (S_{VS}). Salsabil et al. (2009) observed that S_{TS} increased linearly with in ES (3600–10,8000 kJ/kg_{TS}) and reached 14.7% at ES_{max}. Meanwhile, S_{VS} initially increased fast in the ES range of 0–31,500 kJ/kg_{TS} (reaching 15.8%) and then slowed down at higher ES values (reaching 23% at ES_{max}). The main purpose of sludge disintegration is to transfer organic matters from the solid to the aqueous phase. The increase in soluble organic compounds can be correlated with VS reduction (as both COD and VS represent the organic matters of sludge). A higher S_{VS} is important for eliminating/shortening the hydrolysis step of anaerobic digestion. In addition, increasing VS reduction directly improves methane production during anaerobic digestion. Therefore, S_{VS} is comparatively more meaningful than S_{TS} in terms of sludge disintegration (Salsabil et al., 2009; Erden and Filibeli, 2009).

3.1.3. *Dewaterability of sludge*

The capillary suction time (CST) and the specific resistance to filtration (SRF) tests are both commonly used to estimate sludge dewaterability. Most authors agree with Gonze et al.(2003) that are two opposite effects of US on sludge dewaterability: positive for short time US (or low ES) then negative for longer US duration (higher ES).

Feng et al. (2009b) found an increase of sludge dewaterability for an ES range of 0–2200 kJ/kg_{TS}, but a decrease when ES exceeded 2200 kJ/kg_{TS}, especially beyond 4400 kJ/kg_{TS}. Li et al. (2009) indicated that when DD_{COD} was too low (<2%), floc structure exhibited a limited change and sludge dewaterability was almost unchanged. When DD_{COD} was proper (2%–5%), the incompact sludge flocs can be disrupted to smaller fragments and then be re-flocculated to tighter particles with the help of conditioning agents, subsequently resulting in an improvement of sludge dewaterability. When DD_{COD} was high (>7%), sludge particle size was significantly decreased, a number of fine particles were then produced, leading to the deterioration of sludge dewaterability.

According to Chu et al. (2001), sludge dewaterability decreases gradually with an increase in US duration because of the subsequent increase in small particles. After 5 min of sonication at 0.528 W/mL, Wanget al. (2006b) observed that SRF and CST increased from 1.67×10^{12} m/kg and 82 sec, respectively for raw sludge to 1.33×10^{14} m/kg and 344 sec, respectively for pretreated sludge. They linked this phenomenon to floc structure disruption, cell lysis, and release of biopolymers from extracellular polymeric substances (EPS) and bacteria into aqueous phase.

The authors stated that sludge particles are disintegrated to smaller size with higher surface area causing adsorption of more water, thus slowing the release of water from sludge. Moreover, the release of EPS in the solution creates a thin layer on the surface of the filtrating membrane acting as a barrier against the water, consequently reducing sludge dewaterability (Chen et al., 2001; Houghton and Stephenson, 2002; Wang et al., 2006b; Feng et al., 2009b). It was proved that both EPS and particle size have effects on sludge dewaterability but the former is considered prevalent (Feng et al., 2009b).

On the other hand, SRF and CST increase with the decrease in free water of the sludge, which means dewaterability shows a positive correlation with free water content. Nevertheless, despite US transforms interstitial water retained by EPS and inside cells into free water, the negative adsorption effect is predominant; thereby sludge dewaterability is deteriorated at high ES.

3.1.4. *Settleability and turbidity of sludge*

Settling velocity is one of the most important settling parameters of sludge in routine press control and plays an important role in controlling the excess sludge emission and sludge bulking (Feng et al., 2009a). The settleability of sludge is not enhanced by US treatment (Chu et al., 2001). It is deteriorated when increasing ES due to the breakdown of flocs, decrease in particle size, and increase in EPS concentration in the liquid phase (Feng et al., 2009a). On the contrary, the turbidity of sludge usually increases with ES due to particle size reduction (Tiehm et al., 2001) and subsequent release of micro-particles into supernatant, which settle very slowly (Feng et al., 2009a). Sludge settle ability and turbidity are rarely used individually, but combined with other parameters to evaluate the efficiency of sludge US pretreatment.

3.1.5. *Microscopic examination of sludge*

Microscope imaging displays sludge floc and cellular level before and after sonication, thus it can be used to evaluate the disintegration degree of sludge (Chu et al., 2001; Khanal et al., 2006). US pretreatment reduces average size of flocs and creates a lot of separate cells and short filaments pieces, Actinomyces (Dewil et al., 2006). Feng et al. (2009a) found that neither the floc structure nor the microbial cells were totally disintegrated, even at ES of 26,000 kJ/kg_{TS} (TS of 14.4 g/L), because there was still a network of filamentous bacteria in the photomicrographs of the treated sludge. Under different experimental conditions, at higher ES, Chu et al. (2001) observed flocs and cell walls to be almost completely broken down after 40 min of US at 0.33 W/mL (P_{US} of 82.5 W, ES of

96,100 kJ/kg_{TS}, TS of 8.3 g/L). It is therefore clear that US has considerable effects on microbial disruption but the efficiency of the disruption should be presented enclosed with press parameters (P_{US}, ES, TS, *etc.*).

3.2. Chemical change-based evaluation of sludge ultrasonication pretreatment efficiency

Chemical evaluation mainly focuses on sludge disintegration efficiency (Khanal et al., 2007), reflected by the degree of sludge disintegration (DD_{COD}) based on a chemical digestion reference. Besides, the ratio of soluble COD to total COD (SCOD/TCOD) is also used as it represents the release of organic matters from solid to liquid phase after US (TCOD being not significantly affected by US as oxidation remains very limited). Apart from SCOD, nucleic acids, EPS, ammonium nitrogen, and total organic carbon (TOC) concentrations are also considered as the important parameters in chemical evaluation.

3.2.1. Degree of disintegration (DD_{COD})

Both cellular/extracellular matter and organic debris/EPS of sludge are disintegrated by US, leading to the solubilization of solid matters and the increase in organic matters/EPS concentrations in aqueous phase; thereby SCOD of sludge increases (Zhang et al., 2007). That is why the release of those components, especially SCOD can be used to assess sludge disintegration efficiency (Tiehm et al., 2001; Rai et al., 2004; Wang et al., 2006a; Nickel and Neis, 2007).

There are different approaches to determine DD_{COD} after US.

$$DD_{COD} = \frac{SCOD_{US} - SCOD_0}{SCOD_{NaOH} - SCOD_0} \times 100\%$$

(Li et al., 2009)where $SCOD_{US}$ (mg/L) is supernatant COD of the sonicated sample; $SCOD_0$ (mg/L) is supernatant COD of original sample; $SCOD_{NaOH}$ is the COD release in the supernatant after NaOH digestion (the sludge sample being mixed with 0.5 mol/L NaOH at room temperature for 24 hr)

$$DD_{COD} = \frac{SCOD_{US} - SCOD_0}{TCOD - SCOD_0} \times 100\%$$

(Bougrier et al., 2006; Zhang et al., 2007)

$$DD_{COD} = \frac{SCOD_{US} - SCOD_0}{COD_{MAX}} \times 100\%$$

(Braguglia et al., 2008) where COD_{max} is COD of the reference sample after complete chemical solubilization with H_2SO_4.

It was proved that US sludge disintegration depends on various factors, such as F_S, I_{US}, US duration, D_{US}, ES, temperature, TS, sludge type/properties, *etc.*, among which US duration, ES, TS, and temperature are the most important (Grönroos et al., 2005).

3.2.2. Nucleic acid assessment

The increase in nucleic acid concentration represents cell lysis, thus it is also used to evaluate the efficiency of sludge US pretreatment. Zhang et al. (2007) measured the concentration of nucleic acids after US treatment and found a linear relationship between cell lysis and D_{US} (0.1–1.5 W/mL for 30 min US) as well as sonication time (0–30 min US at 0.5 W/mL).

3.2.3. Protein assessment

Under US, the activated sludge is disintegrated, cells are ruptured, and consequently EPS and cellular substances are released into the aqueous phase, resulting in an increase in protein and polysaccharide levels. It can be inferred that the rise of soluble protein increases the anaerobic digestion efficiency (Aldin et al., 2008), thus it was used to evaluate the efficacy of sludge US pretreatment (Akin et al., 2006; Wang et al., 2006a; 2006b). Besides, Ca^{2+} and Mg^{2+} play a key role in binding the EPS. Sonication first causes a fast increase in Ca^{2+} and Mg^{2+} concentrations in the aqueous phase, but then these concentrations decrease as the cations are adsorbed by smaller sludge particles formed during US (Wang et al., 2006a).

The amounts of proteins, polysaccharides, and DNA in the supernatant first increase fast when US is applied (Feng et al., 2009a; 2009b). Then the release of proteins and polysaccharide slows down when sludge is almost disintegrated, but DNA concentration drops due to temperature increase during US which would denature the DNA (Wang et al., 2006a). Among those components, protein is the most released due to large quantities of exoenzymes in the flocs: a ratio of protein to polysaccharide of about 5.4 was found by Feng et al. (2009a).

However, the protein measurement is not common and not yet well accepted for evaluating sludge ultrasonic disintegration efficiency. Therefore, COD measurement is preferred for this purpose due to its simplicity and easiness in daily operation (Pilli et al., 2011).

3.2.4. Release of ammonia and soluble organic nitrogen assessment

The ammonia nitrogen concentration increases following the increase in ES due to the disintegration of bacterial cells and release of intracellular organic nitrogen into the aqueous phase, which is subsequently hydrolyzed to ammonia (Khanal et al., 2006; Akin et al., 2006). The disintegration of organic nitrogen from non-biological debris is also an important contribution to ammonia nitrogen (Khanal et al., 2007).

Bougrier et al. (2005) and Salsabil et al. (2009) claimed that total Kjeldahl nitrogen in the whole sludge is constant regardless of ES, which means US does not lead to nitrogen mineralization or volatilization. Following an increase in ES, organic nitrogen in particles decreases meanwhile organic nitrogen in soluble phase and ammonia concentrations increase. Different estimations of solubilization of organic nitrogen were obtained: about 40% at 15,000 kJ/kg_{TS}—220 W (Bougrier et al., 2005) and about 19.6% at 108,000 kJ/kg_{TS}—60 W (Salsabil et al., 2009). Very little organic nitrogen is transformed into ammonium (NH_4^+–N).

In short, the release of ammonia and soluble organic nitrogen in the aqueous phase could be another useful indicator to assess sludge US pretreatment efficacy. However, a correlation between nitrogen release data and subsequent anaerobic digestion efficiency under different conditions is required to obtain a standardized method based on NH_3 data (Pilli et al., 2011).

3.2.5. Sonication duration assessment

In agreement with TCOD, TOC of sludge (solid + liquid) stays almost constant as the organics only pass from solid to liquid phase during US treatment without significant oxidation. After 90 min of sonication at 200 W, Kidak et al. (2009) observed that the solubilization of organics (based on sonication duration measurement in the supernatant) reached 7.9% and 22.8% for industrial and municipal sludge, respectively. This increase of sonication duration in the liquid phase was consistent with the results obtained from the COD analysis.

3.3. *Biological change-based evaluation of sludge ultrasonic pretreatment efficiency*

The breakdown of bacterial cell walls due to US can be evaluated by biological utilization tests. The sludge microbiological activity is characterized using Oxygen Utilization/Uptake Rate (OUR). OUR measurement therefore could be used to evaluate the sludge US disintegration efficiency.

In general, sludge microbial activity decreases when DD_{COD} increases during US sludge treatment. Nevertheless, Li et al. (2009) found that microbial activity was first enhanced and OUR increased about 20–40% when DD_{COD} was in the range 0–20%. This indicates that the flocs were slightly disrupted, but the cell lysis did not occur at this stage. In other words, the microbial activity would go up when the micro-floc aggregates are separated from the sludge flocs. When DD_{COD} was 20%–40%, OUR still increased but by less than 20%, which means that some microorganisms were damaged. When DD_{COD} was over 40%, inactivation of microbes occured, *i.e.* most bacteria were disrupted at different degrees, and sludge microbial activity decreased significantly. In other words, cells started to lyse only when DD_{COD} was over 40% as presented in Fig. 5.

DD_{OUR} is considered as the degree of inactivation and calculated as follows: $DD_{OUR}(\%) = \frac{1\text{-OUR}}{\text{OUR}_0} \times 100$ (Rai et al., 2004) where OUR and OUR_0 is the oxygen uptake rate of sonicated and original sample, respectively.

DD_{OUR} first increases quickly with the increase in ES, but the increase then slows down, above ES of 40 kJ/g_{TS} according to Rai et al.(2004). It could be inferred that DD_{OUR} is directly proportional to DD_{COD}. However, Zhang et al. (2007) observed a big difference between DD_{OUR} (95.5%) and DD_{COD} (30.1%), indicating some chemical reactions might have happened and inhibited cell metabolisms without disrupting the sludge structure. Akin et al. (2006) also noticed that microbes were inactivated well prior to their disintegration, *e.g.* the percentage of microbial inactivation ranged from 53% to 69% (corresponding to different TS) after 60 s of US and the OUR values changed insignificantly for longer duration. According to Pilli et al. (2011), OUR data therefore should not be used to assess the degree of sludge disintegration.

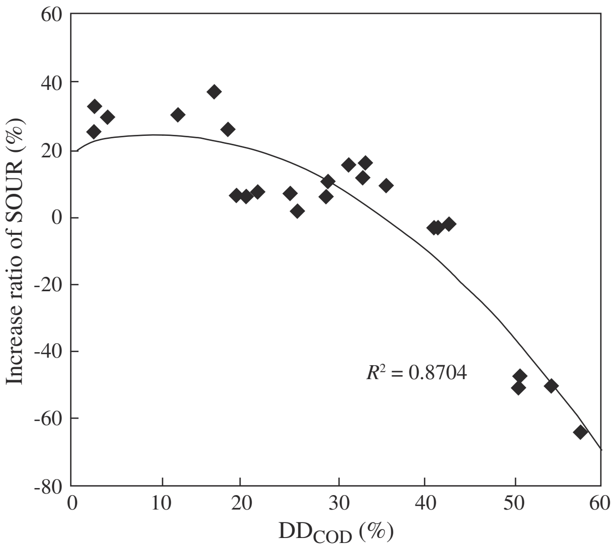

Fig. 5 – Relationship between sludge microbial activity and disintegration degree during ultrasonic treatment (Li et al., 2009).

Chu et al. (2001) proposed the following scenario to describe the sonication of a biological sludge. In the first stage (0–20 min), mechanical forces break down the porous flocs into small particles and release extracellular polymers. In the second stage (20–60 min), the biomass is inactivated and organic matters are dissolved. In the final stage (>60 min), sonication has essentially no effect on sludge if the bulk temperature has been controlled; if it is not controlled, the total coliform could be disinfected effectively if time exceeds 60 min. Of course, these results based on US time only give the general trend.

Zhang et al. (2007) showed that the sludge inactivation efficiency increased significantly after 10 min of sonication and the biomass inactivation stage was 10–30 min, which was different from Chu et al. (2001) maybe due to the different D_{US} applied: 0.5 W/mL as compared to 0.3 W/mL by Chu et al. (2001). After 30 min of sonication, the sludge OUR decrease ratio was 95.5%, which indicated that biological cells were almost completely inactivated. The above hypothesis was therefore modified as follows: sludge disintegration and cell lysis occur continuously during sonication, but sludge inactivation occurs mainly in the second stage (10–30 min). It could be concluded that D_{US} and US duration are important parameters affecting inactivation of sludge.

Besides, Li et al. (2009) mentioned two main stages in US sludge pretreatment process: sludge flocs are changed and disintegrated at first, and then the exposed cells are disrupted. In the first stage, some organic matters contained in the flocs are dissolved, SCOD increases slightly, and OUR also increases due to the enhancement of oxygen and nutrients consumption. In the second stage, some cells are exposed and damaged by US cavitation, leading to the release in intracellular organic matters, the further increase in SCOD, and the significant decrease in OUR. Due to the heterogeneity of sludge and the differences in the external resistances of many types of zoogloea and bacteria, activation and inactivation might both occur at the same time and the comprehensive effectiveness is under the influence of various US parameters.

3.4. *Effects of US on sludge degradability and methane production in anaerobic digester*

The main purpose of US is to accelerate the sludge biodegradability to favor the methane production at lower hydraulic retention time (HRT) in anaerobic digester. To evaluate the effect of US on anaerobic digestion of sludge, Tiehm et al. (1997) proved sonicated sludge to be digested stably even in the experiment of 8-day residence time, in which biogas was produced more than double (2.2 times) that in the control digester. Similarly, Neis et al. (2000) and Bunrith (2008) showed the increase in degradation rates of sludge due to US, allowing shorten the retention time, and then eventually reduce the digester volume for same digestion efficiency. Besides, biogas production rate in sonicated sludge was also increased at lower HRT (Shimizu et al., 1993; Neis et al., 2000). It could be inferred that raw sludge was degraded better under the effect of US disintegration, which helped increase the biogas production substantially.

Many researches also indicated the VS reduction (which directly converting to increased methane production) and biogas production in anaerobic digestion to be enhanced after US (Tiehm et al., 1997; Bunrith, 2008; Pilli et al., 2011). Related to the effect of US frequency, as found with DD_{COD}, owing to the increase in frequency (41–1068 kHz), the VS reduction decreased (Tiehm et al., 2001). For effects of US time, and then of ES, Wang et al. (1999), Tiehm et al. (2001), Bougrier et al. (2005) and Grönroos et al. (2005) found VS reduction, biogas production in anaerobic digestion digester and the methane percentage in the biogas increased gradually following the increase in US time. In other words, anaerobic sludge stabilization was intensified with the increase in DD_{COD}.

4. Optimization of ultrasonic pretreatment of sludge

The ambient conditions of the sonicated system can significantly affect the intensity of cavitation and consequently affect the efficiency (rate and/or yield) of the desired operation. The cavitation effect is influenced by many factors: gas and particulate matter, solvent, field type (standing or progressive wave), types of US cavitation (related to F_S, D_{US}, I_{US}), attenuation, temperature, external pressure, and sample preparation, *etc.* (Lorimer and Mason, 1987; Thompson and Doraiswamy, 1999; Pilli et al., 2011). This section aims at presenting main parameters significantly affecting the cavitation in order to optimize sludge US pretreatment efficacy.

4.1. *Ultrasonic frequency*

Acoustic cavitation is a phenomenon that is mainly related to the sound pressure amplitude, its frequency, through the bubble size variations (Leighton, 2007). For a given frequency and sound pressure amplitude, there is a critical size range in which the initial size of the bubbles must fall to nucleate cavitation (Leighton, 1994). This range increases with the increase in acoustic pressure amplitude and the decrease in frequency.

Sound frequency has a significant effect on the cavitation process because it alters the critical size of the cavitation bubble (Thompson and Doraiswamy, 1999). In general, the increase in acoustic frequency leads to the decrease in cavitation physical effects (Crum, 1995; De La Rhebrhard et al., 2012) due to the decrease in radius range that will provide cavitation (Leighton, 2007). It was added that at very high frequencies, the finite time of the rarefaction cycle is too short to allow a bubble to grow and collapse (Lorimer and Mason, 1987). Moreover, even if a bubble is produced during rarefaction, the compression cycle occurs too fast to collapse the bubble (Thompson and Doraiswamy, 1999). On the other hand, at higher sound frequencies, although cavitation is less violent, there are more cavitation events and thus more radicals to be produced and consequently a promotion of chemical reactions (Crum, 1995). Meanwhile, lower sound frequencies have stronger shock waves and favor mechanical effects (Zhang et al., 2008a). This more violent collapse at low frequencies is due to the resonance bubble size being inversely proportional to the acoustic frequency (Laborde et al., 1998).

The optimum frequency is system-specific and depends on whether intense temperatures and pressures (enhanced by lower frequencies) or single electron transfer reactions (enhanced by higher frequencies) are looked for. The choice of frequency therefore depends on the expected type of US effects: mechanical, due to shock waves and high local shear stresses, or chemical, connected to free radical formation. For example, 20–60 kHz are used for ultrasonic cleaning baths (Lorimer and Mason, 1987; Entezari et al., 1997) or metal corrosion (Whillock and Harvey, 1997b; Dhe et al., 2003), 20–600 kHz for sonochemical degradation of carbon tetrachloride (Francony and Pétrier, 1996), trichloroethylene (Drijvers et al., 1996), methylene blue (Kobayashi et al., 2012), or ethylbenzene sulfonate (Deojay et al., 2011), 20–900 kHz for sonochemical oxidation of iodide (Entezari and Kruus, 1996) or large-scale sonochemical reactors (Asakura et al., 2008). However, in several reactions, the alteration of frequency (20–900 kHz) has no apparent effect, such as in the dissociation of carbon disulfide (Entezari et al., 1997).

With regard to sludge pretreatment, ultrasound mechanically disrupts the floc matrix and cell structure. Tiehm et al. (2001) and Zhang et al. (2008a) found that DD_{COD} decreased owing to the increase in frequency (41–1068 kHz and 25–150 kHz, respectively), indicating that mechanical effects, instead of free radicals, are responsible for the biodegradability enhancement. It is therefore important to note that in most works sludge disintegration is the most significant at low frequencies (Pham et al., 2009; Carrère et al., 2010; Pilli et al., 2011). However, the lowest investigated values of frequency in this field have been restricted to around 20–25 kHz. Lower frequency could then be interesting in sludge disintegration and needs detailed investigation.

4.2. *Temperature*

Theory-based, increasing temperature will decrease surface tension and raise the equilibrium vapor pressure of the medium, leading to easier bubble formation. However, these kinds of cavitation bubbles contain more vapors that reduce the US energy produced by cavitation because they cushion the implosion, thus reducing the amount of free radicals produced within the bubble and also mechanical effects as shock waves. Besides, great numbers of cavitation bubbles generated simultaneously will provoke attenuation or dampening effect on the propagation of US energy from the emitter through the system (Lorimer and Mason, 1987).

Nevertheless, in terms of sludge disintegration, it is important to note that sludge ultrasonic pretreatment efficacy increases following an increase in the bulk temperature as temperature alone favors COD release. It was proved that the US treatment has two simultaneous effects: (i) vigorous agitation caused by the formation and explosion of tiny bubbles and (ii) the increase in the bulk temperature. Chu et al. (2001), Grönroos et al. (2005), Li et al. (2009) and Kidak et al. (2009) concluded that the higher the temperature of sludge samples, the more efficient the US disintegration was. This is opposite to most power US applications as cavitation intensity is higher at low temperature.

Li et al. (2009) indicated that the temperature effect is limited when US duration is short. For example, after 1 min of US at 4 W/mL, DD_{COD} was 9% for both samples without and with temperature control (20°C). On the other hand, after 5 min at 0.8 W/mL, DD_{COD} was 27% and 23% for the uncontrolled and controlled temperature samples, respectively. It was also noted that cavitation explosion and bulk temperature increase have equal influence on sludge floc disintegration and cell lysis (Chu et al., 2001).

It could be suggested that for any scale up operation, on one hand, the process should be carried out without cooling to make use of thermal solubilization; on the other hand, the extreme temperature must be controlled neither to damage the mechanical equipment nor to fully inhibit transient cavitation. In other words, the US system should be controlled at the possible highest temperature in order to both take advantage of US (cavitation and temperature effects) and to maintain the system (Kidak et al., 2009). This suggests a probable optimum temperature.

4.3. *Hydrostatic pressure*

Despite ultrasonic sludge treatment has reached commercial developments and given rise to many works, none of them has been carried out to investigate the effect of pressure. Changing the hydrostatic pressure will change the resonance condition of cavitation bubbles *via* their equilibrium radius and then may drive the system toward resonance conditions (Thompson and Doraiswamy, 1999). At resonance conditions, the rate and yield of reactions will increase (Cum et al., 1988, 1990, 1992). More probably, both the cavitation threshold and the intensity of cavity collapse should increase following an increase in external pressure (Lorimer and Mason, 1987), suggesting a possible optimum pressure. Brett and Jellinek (1956) stated that bubbles could be visible for gas-applied pressure as high as 16 atm. Nevertheless, nearly all the US experiments have been carried out at atmospheric pressure. Only a few studies have been focusing on how increasing static pressure affects cavitation.

Whillock and Harvey (1997a) investigated the effects of hydrostatic pressure on the corrosion of 304 L stainless steel in an ultrasonic field. An increase in pressure up to 4 bar at a constant temperature caused a strong increase in corrosion rate. Hydrostatic pressure retards both cavity nucleation (reduction of the total number of cavities) and cavity growth (decrease in the sizes of cavities). As a result, larger US intensity is required to induce bubble oscillations and implosions. More recent pressure effects again focused attention. Gaitan et al. (2010) found that the collapse strength is intensified at elevated static pressures in part due to an increased differential pressure between the external liquid and the interior of the bubble. Bader et al. (2012b) extended the work of Gaitan et al. (2010) and found the increase in the collapse strength of transient cavitation events at elevated static pressure (up to 300 bar) to be more strongly dependent on the increased acoustic energy stored in the resonant system (*i.e.* increased peak negative pressure) rather than the increased differential pressure. The overpressure acts to suppress cavitation and increase the amount of stored energy which leads to an increase in the collapse strength and therefore shock wave amplitudes. Besides, the cavitation threshold increases linearly with the static pressure, thus the acoustic pressure amplitude required to reach the cavitation threshold also increases (Bader et al., 2012a). Yasui et al. (2011) showed an enhancement of acoustic energy radiated by a bubble per acoustic cycle either by the excess static pressure for relatively high acoustic amplitudes and low viscosities of liquids or by a reduced static pressure for relatively low acoustic amplitudes and high viscosities. The optimal static pressure which maximizes the acoustic energy increases as the acoustic amplitude increases or viscosity of liquid decreases, which qualitatively agrees with Sauter et al. (2008).

Closer to the present subject, Neppiras and Hughes (1964) investigated the influence of pressure (up to 5.8 atm) on the disintegration of yeast cells and found an optimum value of 4 atm. As mentioned the effect of pressure on sludge pretreatment has hardly been investigated but should deserve attention.

4.4. *Energy aspects*

Concerning the economy of the process of US sludge disintegration, the operation cost is directly linked to ES — the US energy per unit weight of dry sludge provided to the suspension. The fact that US sludge disruption is an energy-driven process was effectively proved by the usual verification that ES is by far the main parameter (Tiehm et al., 2001; Rai et al., 2004; Grönroos et al., 2005; Bougrier et al., 2005; Khanal et al., 2006; Feng et al., 2009a; *etc.*), even if many authors remained concerned with only US power, time of irradiation, and to a less extend US intensity and US dose (Mao et al., 2004; Wang et al., 2005; Zhang et al., 2007; Na et al., 2007; El-Hadj et al., 2007; *etc.*).

Knowing this fundamental result, the questions to be solved are: is there an optimum power or power density, an optimum sludge concentration, and later how to extrapolate? What is the effect of the equipment size?

4.4.1. *Ultrasonic power*

As a general trend it is usually accepted that US power has positive effect in most US applications taking advantage of either chemical or physical effects. Nevertheless, very high power or intensity may be detrimental. Ratoarinoro et al. (1995) and Contamine et al. (1994) explained that at high $P_{US,}$ the formation of a dense cloud of cavitation bubbles around the probe blocks the energy transmitted from emitter to the solution. The optimum P_{US} also depends on F_S: different optimal values were found for P_{US} depending on F_S when investigating the corrosion rate of 304 L stainless steel; no optimum value was observed at 20 kHz (Whillock and Harvey, 1997b).

In the case of sludge pretreatment, it is proved that the solubilization of organics increases when applying elevated P_{US} or D_{US}. For example, at ES of 100,000 kJ/kg_{TS}, DD_{COD} were 52.3% and 71.3% for P_{US} of 100 W and 200 W, respectively (Kidak et al., 2009). At the same ES of 40 kWh/kg_{TS}, SCOD increased by 1.2–1.9 fold corresponding to the D_{US} range of 0.18–0.52 W/mL (Show et al., 2007), and by 1.2–4.8 fold for 2–4 W/mL (Mao et al., 2004). Chu et al. (2001) indicated the total solubilized COD fraction (SCOD/TCOD) during 40 min at 0.33 W/mL to be much higher than that during 2 hr at 0.11 W/mL.

According to Kidak et al. (2009), at a given ES, "high P_{US}–short US duration" should be preferred for heterogeneous sludge like municipal sludge, in agreement with Grönroos et al. (2005), Zhang et al. (2007), and Show et al. (2007). Conversely, "low P_{US} and long US duration" better works for homogenous sludge like industrial sludge. It could be reasoned that particles in municipal sludge (like fibrous particles coming from toilet papers) are resistant to US disruption; thus P_{US} should be increased to break these particles. On the other hand, the settled bacteria (the major components in industrial sludge) are broken to soluble materials even at low P_{US}; more solubilization consequently could be obtained when increasing the US duration.

It is clear that P_{US} and D_{US} are important parameters in WAS disintegration that must be considered in terms of cost-benefit purpose in full-scale application.

4.4.2. Ultrasonic intensity

Above the cavitation threshold, increasing I_{US} leads to a rise in the maximum pressure and temperature within a transient collapse (Lorimer and Mason, 1987), improving all mechanical effects, and then the degree of sludge disintegration (DD_{COD}) (Quarmby et al., 1999; Neis et al., 2000; Pilli et al., 2011). For instance, Neis et al. (2000) found that DD_{COD} was more than double by increasing I_{US} from 6 to 18 W/cm^2.

However, Lorimer and Mason (1987) noted that I_{US} cannot be increased indefinitely since a subsequent pressure amplitude increase may result in so large bubbles during rarefaction that the time available for their collapse is insufficient. This is rather similar to the explanation of optimum power due to the damping of US wave by an excess of cavitation bubbles near the emitter (Contamine et al., 1994; Ratoarinoro et al., 1995).

Apart from bubble formation, bubble behavior is also associated with I_{US}. As discussed, the disruptive effect of transient bubbles in a short US duration is more noticeable than that of stable bubbles with long US duration. Thus, I_{US} may be considered as a more predominant parameter than US duration in terms of bubble behavior, thereby the US process can be optimized by increasing I_{US} to minimize energy use (Show et al., 2007).

In addition, I_{US} is the quotient of P_{US} and the surface area of the probe (A). Most researches (Wang et al., 2005; Show et al., 2007; Zhang et al., 2008b; Liu et al., 2009; Li et al., 2010) have varied only $P_{US,}$ meanwhile the magnitude of the effect of each factor needs further investigation in connection with scale-up purpose.

4.4.3. Ultrasonic duration and specific energy input

In earlier studies, sonication time was most often used although as already mentioned ES has more significance and should be preferred. It was proved that the solubilization of WAS increases gradually with an increase in US duration at same US conditions (Lorimer and Mason, 1987; Wang et al., 1999; Mao et al., 2004; Show et al., 2007). For example, to get 50% and 75–80% increase in solubilization, it required at least 30–40 min and 90 min of sonication, respectively (Shimizu et al., 1993).

In terms of ES, different ranges were investigated. Generally, SCOD increases with an increase in ES. Considering together the disintegration efficiency and the energy input, different ES values were suggested: 4000 kJ/L (Na et al., 2007), 10,000 kJ/kg$_{TS}$ (Bougrier et al., 2005), 12,000 kJ/kg$_{TS}$ (Neis et al., 2000), 35,000 kJ/kg$_{TS}$ (Khanal et al., 2006), 50,000 kJ/kg$_{TS}$ (Wang et al., 2006a).

In addition, according to Kidak et al. (2009), higher reactor volume resulted in a decrease in DD_{COD} due to the difficulties in creating homogeneous sonication, as intense damping occurs in the sludge suspension. This is a complex problem faced when trying to scale up this process.

In conclusion, it is clear that experimental results are required to account for P_{US}, I_{US}, and D_{US} (through optimal solid concentration) and not only for ES.

4.5. Sludge type, and total solid concentration of sludge

Mao et al. (2004) proved the SCOD in WAS to be higher than that in primary sludge. Regarding TS concentration, high solid loading in the liquid generally makes more cavitation sites and then more intense hydro-mechanical shear forces (Neis et al., 2000; Mao et al., 2004; Akin et al., 2006; Show et al., 2007; Zhang et al., 2008b; Kidak et al., 2009; Pilli et al., 2011). However, the effect of TS depends on many factors, *e.g.* reactor configuration (reactor size, transducer type), T, P_{US}, and sludge characteristics (Grönroos et al., 2005). An optimum TS concentration can be found, which is explained by opposite effects. The increase in TS provides more cells and aggregates to be in contact with cavitation bubbles; thereby, the P_{US} required to generate cavitation is more efficiently consumed. However, at high sludge loading, the acoustic pressure field decreases faster from the emitter due to the degraded propagation of US waves in a denser suspension. Consequently, acoustic cavitation intensity is reduced. For example, SCOD increased from 1000 to 5800 mg/L when TS varied between 0.98% and 2.6%, but it decreased to 3200 mg/L when TS was 3.6% (Akin et al., 2006). According to Kidak et al. (2009), DD_{COD} hiked up with an increase in TS within the range 4–12 g/L, but it severely decreased at a TS of 24 g/L. Show et al. (2007) found the optimum range of TS to be between 2.3% and 3.2% at constant energy input.

4.6. pH of sludge

According to Wang et al. (2005), the effects of sonication parameters and sludge properties on solubilization of the chemical oxygen demand (COD) can be rated as follows: sludge pH > sludge concentration > ultrasonic intensity > ultrasonic density. This suggests that pH adjustment to a suitable value prior to US pretreatment is an important step.

Sludge cells were proved to be disintegrated and dissolved by acidic treatment, solubilization being only significantly affected by the acid dose (Woodard and Wukash, 1994). The optimal pH values for reducing volatile suspended solids and excess sludge subsequently was found to vary between 1.5 (Woodard and Wukash, 1994) and 3 (Neyens et al., 2003). However, acidic pretreatment alone exhibits a very low performance as compared to US pretreatment for releasing organic matters into the liquid phase and Apul (2009) reported the sludge acidification to be detrimental to US pretreatment performance, especially at low pH values.

On the other hand, alkaline pretreatment enhances sludge solubilization, anaerobic biodegradability, and methane production (Kim et al., 2003; Valo et al., 2004). Besides, the combination of alkaline and US gives better performances of TS solubilization as compared to both thermo-acidic and US-acidic pretreatments (Liu et al., 2008). Moreover, Chu et al. (2001) showed that EPS and gels surrounding cells limit the efficiency of ultrasonic treatment on sludge disintegration. Adjusting the pH of sludge to alkali value promotes EPS hydrolysis and gel solubilization. After that, cell walls cannot maintain an appropriate turgor pressure (Jin et al., 2009) and easily disrupt. Therefore, the combined alkaline-US pretreatment, based on different mechanisms of sludge disintegration (modification of structural properties and intense mechanical shear force), is expected to take advantage of both and achieve a better efficiency of sludge pretreatment. Some synergetic effects were even noticed (Kim et al., 2010). At near-neutral pH conditions (pH 7–8), waste activated sludge (WAS) solubilization obtained from combined, chemical, and US (1.9 W/mL, 60 sec) pretreatments were 18%, 13.5%, and 13%, respectively (Bunrith, 2008). At higher pH values (pH 11–13), the solubilization reached 60%–70% with the combined method (ES 7500–30,000 kJ/kg_{TS}) while it never exceeded 50% in individual pretreatments (Jin et al., 2009; Kim et al., 2010). Methane production yield derived from full stream combined-pretreated sludge (pH 9, ES 7500 kJ/kg_{TS}) was also 55% higher than that from the control (Kim et al., 2010) which seems rather questionable.

The chemicals used for increasing the pH of sludge also affect WAS solubilization efficacy, where NaOH > KOH > Mg $(OH)_2$ and Ca $(OH)_2$ (Kim et al., 2003; Jin et al., 2009). Ca^{2+} and Mg^{2+} are key substances binding cells with EPS. As a result, their presence may enhance the reflocculation of dissolved organic polymers (Jin et al., 2009), leading to a decrease in SCOD. On the other hand, overconcentration of Na^{+} (or K^{+}) was reported to cause subsequent inhibition of anaerobic digestion (Carrère et al., 2010).

4.7. Outlook

Regarding sludge US pretreatment, I_{US} has positive effects, but the respective magnitude of the effect of P_{US} and probe size has not been looked into. Besides, sludge disintegration is known to take advantage of low frequency but audible frequency has not yet been considered. In addition, hydrostatic pressure is an important parameter, but has hardly been investigated in terms of sludge US pretreatment. Thereby several issues need to be elucidated or confirmed in order to optimize sludge disintegration:

- How important are the effects of P_{US}, $I_{US,}$ and F_S on sludge pretreatment efficiency? Which parameter between P_{US} and probe size is more meaningful in terms of I_{US} effect on sludge pretreatment efficiency? Does a very low frequency down to audible range really improve the efficiency of sludge disintegration?
- Is there an optimum hydrostatic pressure for sludge US pretreatment? If any, how do the other parameters (sludge type, sludge concentration, temperature, *ES*, $P_{US,}$ I_{US}, F_S) affect this optimum and what is the expected gain in terms of energy saving?
- How does the US procedure (continuous or sequential treatment at optimum conditions) affect the efficiency of sludge disintegration and *AD* afterwards?

In order to answer these questions, the following tasks might be taken into consideration to optimize sludge pretreatment by sonication: (i) to investigate usual operation parameters: sludge type, TS concentration, sludge pH (alkaline dose, holding time), stirrer speed, and temperature profile, (ii) to quantify the effect of US parameters on sludge disintegration: P_{US}, I_{US}, and F_S, (iii) to study the effect of pressure on sludge US pretreatment at various P_{US}, I_{US}, and F_S, (iv) to finally optimize US process.

5. Conclusions

According to the literature review, studies about US sludge disintegration have expressed US effect using different reference properties. There is still no fully comprehensive method to evaluate the efficiency of sludge US pretreatment. However, some main parameters commonly used for this purpose are DD_{COD}, proteins, particle size reduction, *etc.* due to their simplicity, easiness, and predominant accuracy in daily operation.

Regarding US parameters, apart from ES recognized as the main one, P_{US}, I_{US}, and frequency seem to have significant effects. However, static pressure effect has been only marginally studied due to the complex equipment required. The magnitude of the effect of P_{US} and probe size in terms of I_{US} has not been clearly detailed and should to be investigated at constant ES. Besides, investigation on the effect of pH — alkalization prior to US process has been restricted to limited concerned parameters (initial pH or alkaline dose and ES). In addition, investigating very low frequency (acoustic frequency) seems interesting but has not yet been taken into consideration. Their effects therefore should be varied separately and simultaneously with other related parameters, *i.e.* process conditions, ultrasonic properties, and sludge characteristics, to optimize sludge US pretreatment press.

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REFERENCES

Akin, B., Khanal, S.K., Sung, S., Grewell, D., Van-Leeuwen, J., 2006. Ultrasound pre-treatment of waste activated sludge. Water Sci. Technol. 6 (6), 35–42.

Aldin, S., Elbeshbishy, S., Tu, F., Nakhla, G., Ray, M., 2008. Pre-treatment of primary sludge prior to anaerobic digestion. (Available at: http://www.nt.ntnu.no/users/skoge/prost/

preedings/aiche-2008/data/papers/P137267.pdf. Date assessed: 28 December 2012).

Apul, O.G., 2009. Municipal sludge minimization: evaluation of ultrasonic and acidic pretreatment methods and their subsequent effects on anaerobic digestion. Master Degree Thesis. Middle East Technical University.

Asakura, Y., Nishida, T., Matsuoka, T., Koda, S., 2008. Effects of ultrasonic frequency and liquid height on sonochemical efficiency of large-scale sonochemical reactors. Ultrason. Sonochem. 15 (3), 244–250.

Bader, K.B., Raymond, J.L., Mobley, J., Church, C.C., Felipe Gaitan, D., 2012a. The effect of static pressure on the inertial cavitation threshold. J. Acoust. Soc. Am. 132 (2), 728–737.

Bader, K.B., Mobley, J., Church, C.C., Felipe Gaitan, D., 2012b. The effect of static pressure on the strength of inertial cavitation events. J. Acoust. Soc. Am. 132 (4), 2286–2291.

Barber, W.P., 2005. The effects of ultrasound on sludge digestion. Water Environ. J. 19 (1), 2–7.

Bartholomew, R., 2002. Ultrasound disintegration of sewage sludge: an innovative wastewater treatment technology PA Department of Environmental Protection, Bureau of Water Supply And Wastewater Management Available at: http://www.dep.state.pa.us/DEP/DEPutate/Watermgt/WSM/WSM_TAO/InnovTech/ProjReviews/Ultrasound-Disintegr.htm.

Bougrier, C., Carrère, H., Delgenès, J.P., 2005. Solubilization of waste-activated sludge by ultrasonic treatment. Chem. Eng. J. 106 (2), 163–169.

Bougrier, C., Albasi, C., Delgenès, J.P., Carrère, H., 2006. Effect of ultrasonic, thermal and ozone pre-treatments on waste activated sludge solubilisation and anaerobic biodegradability. Chem. Eng. Press. 45 (8), 711–718.

Braguglia, C.M., Mininni, G., Gianico, A., 2008. Is sonication effective to improve biogas production and solids reduction in excess sludge digestion? Water Sci. Technol. 57 (4), 479–483.

Brett, H.W.W., Jellinek, H.H.G., 1956. Degradation of long-chain molecules by ultrasonic waves. Part VI. Effect of pressure. J. Polym. Sci. 21 (99), 535–545.

Bunrith, S., 2008. Anaerobic digestibility of ultrasound and chemically pretreated waste activated sludge. Master Degree Thesis. Asian Institute of Technology, Thailand (Available at: http://www.faculty.ait.ac.th/visu/Data/AIT-Thesis/MasterThesisFinal/Bunrith.pdf. Date accessed: 28 July 2012).

Carrère, H., Dumas, C., Battimelli, A., Batstone, D.J., Delgenès, J.P., Steyer, J.P., et al., 2010. Pretreatment methods to improve sludge anaerobic degradability: a review. J. Hazard. Mater. 183 (1–3), 1–15.

Chen, Y.G., Yang, H.Z., Gu, G.W., 2001. Effect of acid and surfactant treatment on activated sludge dewatering and settling. Water Res. 35 (11), 2615–2620.

Chiu, Y.C., Chang, C.N., Lin, J.G., Huang, S.J., 1997. Alkaline and ultrasonic pretreatment of sludge before anaerobic digestion. Water Sci. Technol. 36 (11), 155–162.

Chu, C.P., Chang, B.V., Liao, G.S., Jean, D.S., Lee, D.J., 2001. Observations on changes in ultrasonically treated waste-activated sludge. Water Res. 35 (4), 1038–1046.

Chu, C.P., Lee, D.J., Chang, B.V., You, C.S., Tay, J.H., 2002. "Weak" ultrasonic pre-treatment on anaerobic digestion of flocculated activated biosolids. Water Res. 36 (11), 2681–2688.

Contamine, F., Faid, F., Wilhelm, A.M., Berlan, J., Delmas, H., 1994. Chemical reactions under ultrasound: discrimination of chemical and physical effects. Chem. Eng. Sci. 49 (24B), 5865–5873.

Crum, L.A., 1995. Comments on the evolving field of sonochemistry by a cavitation physicist. Ultrason. Sonochem. 2 (2), S147–S152.

Cum, G., Gallo, R., Spadaro, A., 1988. Effect of static pressure on the ultrasonic activation of chemical reactions. Selective oxidation at benzylic carbon in the liquid phase. J. Chem. Soc. Perkin Trans. 2 (3), 375–383.

Cum, G., Gallo, R., Spadaro, A., Galli, G., 1990. Temperature effects in ultrasonically activated chemical reactions. IL Nuovo Cimento D 12 (10), 1423–1430.

Cum, G., Galli, G., Gallo, R., Spadaro, A., 1992. Role of frequency in the ultrasonic activation of chemical reactions. Ultrasonics 30 (4), 267–270.

De La Rhebrhard, S., Suptil, J., Blais, J.F., Naffrechoux, E., 2012. Sonochemical efficiency dependence on liquid height and frequency in an improved sonochemical reactor. Ultrason. Sonochem. 19 (2), 280–285.

Deojay, D.M., Sostaric, J.Z., Weavers, L.K., 2011. Exploring the effects of pulsed ultrasound at 205 and 616 kHz on the sonochemical degradation of octylbenzene sulfonate. Ultrason. Sonochem. 18 (3), 801–809.

Dewil, R., Baeyens, J., Goutvrind, R., 2006. Ultrasonic treatment of waste activated sludge. Environ. Prog. 25 (2), 121–128.

Dhe, M.L., Hihn, J.Y., Mandroyan, A., Viennet, R., Touyeras, F., 2003. Influence of ultrasound power and frequency upon corrosion kinetics of zinc in saline media. Ultrason. Sonochem. 10 (6), 357–362.

Drijvers, D., de Baets, R., de Visscher, A., Van Langenhove, H., 1996. Sonolysis of trichloroethylene in aqueous solution: volatile organic intermediates. Ultrason. Sonochem. 3 (2), S3–S90.

El-Hadj, T.B., Dosta, J., Márquez-Serrano, R., Mata-Álvarez, J., 2007. Effect of ultrasound pretreatment in mesophilic and thermophilic anaerobic digestion with emphasis on naphthalene and pyrene removal. Water Res. 41 (1), 87–94.

Entezari, M.H., Kruus, P., 1996. Effect of frequency on sonochemical reactions. II: temperature and intensity effects. Ultrason. Sonochem. 3 (1), 19–24.

Entezari, M.H., Kruus, P., Otson, R., 1997. The effect of frequency on sonochemical reactions. III: dissociation of carbon disulfide. Ultrason. Sonochem. 4 (1), 49–54.

Erden, G., Filibeli, A., 2009. Ultrasonic pre-treatment of biological sludge: consequences for disintegration, anaerobic biodegradability, and filterability. J. Chem. Technol. Biotechnol. 85 (1), 145–150.

Feng, X., Lei, H.Y., Deng, J.C., Yu, Q., Li, H.L., 2009a. Physical and chemical characteristics of waste activated sludge treated ultrasonically. Chem. Eng. Press. 48 (1), 187–194.

Feng, X., Deng, J.C., Lei, H.Y., Bai, T., Fan, Q.J., Li, Z.X., 2009b. Dewaterability of waste activated sludge with ultrasound conditioning. Bioresour. Technol. 100 (3), 1074–1081.

Francony, A., Pétrier, C., 1996. Sonochemical degradation of carbon tetrachloride in aqueous solution at two frequencies: 20 kHz and 500 kHz. Ultrason. Sonochem. 3 (2), S77–S82.

Gaitan, D.F., Tessien, R.A., Hiller, R.A., Gutierrez, J., Scott, C., Tardif, H., et al., 2010. Transient cavitation in high-quality-factor resonators at high static pressures. J. Acoust. Soc. Am. 127 (6), 3456–3465.

Gonze, E., Pillot, S., Valette, E., Gonthier, Y., Bernis, A., 2003. Ultrasonic treatment of an aerobic activated sludge in batch reactor. Chem. Eng. Press. 42 (12), 965–975.

Grönroos, A., Kyllönen, H., Korpijärvi, K., Pirkonen, P., Paavola, T., Jokela, J., et al., 2005. Ultrasound assisted method to increase soluble chemical oxygen demand (SCOD) of sewage sludge for digestion. Ultrason. Sonochem. 12 (1–2), 115–120.

Hogan, F., Mormede, S., Clark, P., Crane, M., 2004. Ultrasonic sludge treatment for enhanced anaerobic digestion. Water Sci. Technol. 50 (9), 25–32.

Houghton, J.L., Stephenson, T., 2002. Effect of influent organic content on digested sludge extracellular polymer content and dewaterability. Water Res. 36 (14), 3620–3628.

Jin, Y.Y., Li, H., Mahar, R.B., Wang, Z.Y., Nie, Y.F., 2009. Combined alkaline and ultrasonic pretreatment of sludge before aerobic digestion. J. Environ. Sci. 21 (3), 279–284.

Keles, C., Icemer, G.T., Ozen, S., 2010. Inactivation of *Salmonella* spp. by low-frequency electric fields in sewage sludge. Int. J. Civil Environ Eng 10 (6), 13–18.

Khanal, K., Isik, H., Sung, S., Avan Leeuwen, J., 2006. Ultrasonic conditioning of waste activated sludge for enhanced aerobic digestion. Proceedings of IWA Specialized Conference–Sustainable

Sludge Management. State of the Art, Challenges and Perspectives, Moscow, Russia. May 29–31.

Khanal, S.K., Grewell, D., Sung, S., Van Leeuwen, J., 2007. Ultrasound applications in wastewater sludge pretreatment: a review. Crit. Rev. Environ. Sci. Technol. 37 (4), 277–313.

Kidak, R., Wilhelm, A.M., Delmas, H., 2009. Effect of process parameters on the energy requirement in ultrasonical treatment of waste sludge. Chem. Eng. Press. 48 (8), 1346–1352.

Kim, J., Park, C., Kim, T.H., Lee, M., Kim, S., Kim, S.W., Lee, J., 2003. Effects of various pretreatments for enhanced anaerobic digestion with waste activated sludge. J. Biosci. Bioeng. 95 (3), 271–275.

Kim, D.H., Jeong, E., Oh, S.E., Shin, H.S., 2010. Combined (alkaline + ultrasonic) pretreatment effect on sewage sludge disintegration. Water Res. 44 (10), 3093–3100.

Kobayashi, D., Honma, C., Suzuki, A., Takahashi, T., Matsumoto, H., Kuroda, C., et al., 2012. Comparison of ultrasonic degradation rates constants of methylene blue at 22.8 kHz, 127 kHz, and 490 kHz. Ultrason. Sonochemistry 19 (4), 745–749.

Kopplow, O., Barjenbruch, M., Heinz, V., 2004. Sludge pre-treatment with pulsed electric fields. Water Sci. Technol. 49 (10), 123–129.

Laborde, J.L., Bouyer, C., Caltagirone, J.P., Gérard, A., 1998. Acoustic bubble cavitation at low frequencies. Ultrasonics 36 (1–5), 589–594.

Leighton, T.G., 1994. The Acoustic Bubble. Academic Press, London.

Leighton, T.G., 2007. Review: what is ultrasound? Prog. Biophys. Mol. Biol. 93 (1–3), 3–83.

Li, H., Jin, Y.Y., Mahar, R.B., Wang, Z.Y., Nie, Y.F., 2009. Effects of ultrasonic disintegration on sludge microbial activity and dewaterability. J. Hazard. Mater. 161 (2–3), 1421–1426.

Li, C.L., Liu, G.F., Jin, R.F., Zhou, J., Wang, J., 2010. Kinetics model for combined (alkaline + ultrasonic) sludge disintegration. Bioresour. Technol. 101 (22), 8555–8557.

Lin, J.G., Ma, Y.S., Chao, A.C., Huang, C.L., 1999. BMP test on chemically pretreated sludge. Bioresour. Technol. 68 (2), 187–192.

Liu, D.H., Liptak, B.G., 1999. Environmental Engineers' Handbook. 2nd ed. CRC Press LLC, Ba Raton, Florida (Chapter 7: Wastewater Treatment, URL. Available at:http://www.engnetbase.com/books/78/fm.pdf).

Liu, X., Liu, H., Chen, J.H., Du, G.C., Chen, J., 2008. Enhancement of solubilisation and acidification of waste activated sludge by pre-treatment. Waste Manag. 28 (12), 2614–2622.

Liu, C., Xiao, B., Dauta, A., Peng, G.F., Liu, S.M., Hu, Z.Q., 2009. Effect of low power ultrasonic radiation on anaerobic biodegradability of sewage sludge. Bioresour. Technol. 100 (24), 6217–6222.

Lorimer, J.P., Mason, T.J., 1987. Sonochemistry: part 1—the physical aspects. Chem. Soc. Rev. 16, 239–274.

Mahmoud, A., Olivier, J., Vaxelaire, J., Hoadley, A.F.A., 2010. Electrical field: a historical review of its application and contributions in wastewater sludge dewatering. Water Res. 44 (8), 2381–2407.

Mao, T., Hong, S.Y., Show, K.Y., Tay, J.H., Lee, D.J., 2004. A comparison of ultrasound treatment on primary and secondary sludges. Water Sci. Technol. 50 (9), 91–97.

Na, S., Kim, Y.U., Khim, J., 2007. Physiochemical properties of digested sewage sludge with ultrasonic treatment. Ultrason. Sonochem. 14 (3), 281–285.

Neis, U., Nickel, K., Tiehm, A., 2000. Enhancement of anaerobic sludge digestion by ultrasonic disintegration. Water Sci. Technol. 42 (9), 73–80.

Neppiras, E.A., Hughes, D.E., 1964. Some experiments on the disintegration of yeast by high intensity ultrasound. Biotechnol. Bioeng. 6 (3), 247–270.

Neyens, E., Baeyens, J., Weemas, M., De Heyder, B., 2003. Hot acid hydrolysis as a potential treatment of thickened sewage sludge. J. Hazard. Mater. 98 (1–3), 275–293.

Nickel, K., Neis, U., 2007. Ultrasonic disintegration of biosolids for improved biodegradation. Ultrason. Sonochem. 14 (4), 450–455.

Onyeche, T.I., Schläfer, O., Bormann, H., Schröder, C., Sievers, M., 2002. Ultrasonic cell disruption of stabilised sludge with subsequent anaerobic digestion. Ultrasonics 40 (1–8), 31–35.

Pham, A.T., 2011. Sewage sludge electro-dewatering. PhD Thesis. Mikkeli University Consortium, Mikkeli, Finland (Available at: http://www.doria.fi/bitstream/handle/10024/72598/isbn%209789522651693.pdf?sequence=2. Date assessed 29 t. 2013).

Pham, T.D., Shrestha, R.A., Virkutyte, J., Sillanpää, M., 2009. Recent studies in environmental applications of ultrasound. Can. J. Civ. Eng. 36 (11), 1849–1858.

Pilli, S., Bhunia, P., Yan, S., LeBlanc, R.J., Tyagi, R.D., Surampalli, R.Y., 2011. Ultrasonic pretreatment of sludge: a review. Ultrason. Sonochem. 18 (1), 1–18.

Quarmby, J., Scott, J.R., Mason, A.K., Davies, G., Parsons, S.A., 1999. The application of ultrasound as a pre-treatment for anaerobic digestion. Environ. Technol. 20 (11), 1155–1161.

Rai, C.L., Struenkmann, G., Mueller, J., Rao, P.G., 2004. Influence of ultrasonic disintegration on sludge growth reduction and its estimation by respirometry. Environ. Sci. Technol. 38 (21), 5779–5785.

Ratoarinoro, N., Contamine, F., Wilhelm, A.M., Berlan, J., Delmas, H., 1995. Power measurement in sonochemistry. Ultrason. Sonochem. 2 (1), S43–S47.

Rittmann, B.E., Lee, H.S., Zhang, H., Alder, J., Banaszak, J.E., Lopez, R., 2008. Full-scale application of focused-pulsed pre-treatment for improving biosolids digestion and conversion to methane. Water Sci. Technol. 58 (10), 1895–1901.

Rossier, Membrez, Mottet, 2007. Comparaison des technologies de prétraitement des boues d'épuration pour l'augmentation de la production de biogas. (Département de l'environnement, des transports, de l'énergie et de la communication DETEC) Available at:http://www.bfe.admin.ch/php/modules/publikationen/stream.php?extlang=fr&name=fr_897865446.pdf.

Roxburgh, R., Sieger, R., Johnson, B., Rabinowitz, B., Goodwin, S., Crawford, G., et al., 2006. Sludge minimization technologies—doing more to get less. WEFTEC®.06-Water Environment Foundation, pp. 506–525 (http://www.environmental-expert.com/Files%5C5306%5Carticles%5C8538%5C040.pdf).

Rynkiewicz, M., 2011. Application of constant electric field in simultaneous intensification of dewatering of wastewater sludge and filtrate purification. Environ. Prot. Eng. 37 (3), 93–100.

Salerno, M.B., Lee, H.S., Parameswaran, P., Rittmann, B.E., 2009. Using a pulsed electric field as a pretreatment for improved biosolids digestion and methanogenesis. Water Environ. Res. 81 (8), 831–839.

Salsabil, M.R., Prorot, A., Casellas, M., Dagot, C., 2009. Pretreatment of activated sludge: effect of sonication on aerobic and anaerobic digestibility. Chem. Eng. J. 148 (2–3), 327–335.

Sauter, C., Emin, M.A., Schuchmann, H.P., Tavman, S., 2008. Influence of hydrostatic pressure and sound amplitude on the ultrasound induced dispersion and de-agglomeration of nanoparticles. Ultrason. Sonochem. 15 (4), 517–523.

Shimizu, T., Kudo, K., Nasu, Y., 1993. Anaerobic waste-activated sludge digestion—a bioconversion mechanism and kinetic model. Biotechnol. Bioeng. 41 (11), 1082–1091.

Show, K.Y., Mao, T., Lee, D.J., 2007. Optimisation of sludge disruption by sonication. Water Res. 41 (20), 4741–4747.

Thompson, L.H., Doraiswamy, L.K., 1999. Sonochemistry: science and engineering. Ind. Eng. Chem. Res. 38 (4), 1215–1249.

Tiehm, A., Nickel, K., Neis, U., 1997. The use of ultrasound to accelerate the anaerobic digestion of sewage sludge. Water Sci. Technol. 36 (11), 121–128.

Tiehm, A., Nickel, K., Zellhorn, M., Neis, U., 2001. Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization. Water Res. 35 (8), 2003–2009.

Ultrawaves GmbH-Water & Environmental Technologies, 2013. Waste water treatment plants — intensified sludge treatment by ultrasound Available at: http://www.ultrawaves.de.

Ultrawaves–Royce Water Technologies–Biosolids treatment innovation, n. Available at: http://www.roycewater.com.au.

Valo, A., Carrère, H., Delgenès, J.P., 2004. Thermal, chemical and thermo-chemical pre-treatment of waste activated sludge for anaerobic digestion. J. Chem. Technol. Biotechnol. 79 (11), 1197–1203.

Wang, Q.H., Kuninobu, M., Kakimoto, K., Ogawa, H.I., Kato, Y., 1999. Upgrading of anaerobic digestion of waste activated sludge by ultrasonic pretreatment. Bioresour. Technol. 68 (3), 309–313.

Wang, F., Wang, Y., Ji, M., 2005. Mechanisms and kinetics models for ultrasonic waste activated sludge disintegration. J. Hazard. Mater. 123 (1–3), 145–150.

Wang, F., Ji, M., Lu, S., 2006a. Influence of ultrasonic disintegration on the dewaterability of waste activated sludge. Environ. Prog. 25 (3), 257–260.

Wang, F., Lu, S., Ji, M., 2006b. Components of released liquid from ultrasonic waste activated sludge disintegration. Ultrason. Sonochem. 13 (4), 334–338.

Whillock, G.O.H., Harvey, B.F., 1997a. Ultrasonically enhanced corrosion of 304 L stainless steel I: the effect of temperature and hydrostatic pressure. Ultrason. Sonochem. 4 (1), 23–31.

Whillock, G.O.H., Harvey, B.F., 1997b. Ultrasonically enhanced corrosion of 304 L stainless steel II: the effect of frequency, acoustic power and horn to specimen distance. Ultrason. Sonochem. 4 (1), 33–38.

Woodard, S.E., Wukash, R.F., 1994. A hydrolysis/thickening/filtration press for the treatment of waste activated sludge. Water Sci. Technol. 30 (3), 29–38.

Xie, R.J., Xing, Y.J., Ghani, Y.A., Ooi, K.E., Ng, S.W., 2007. 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), 533–541.

Yasui, K., Towata, A., Tuziuti, T., Kozuka, T., Kato, K., 2011. Effect of static pressure on acoustic energy radiated by cavitation bubbles in viscous liquids under ultrasound. J. Acoust. Soc. Am. 130 (5), 3233–3242.

Zhang, P.Y., Zhang, G.M., Wang, W., 2007. Ultrasonic treatment of biological sludge: floc disintegration, cell lysis and inactivation. Bioresour. Technol. 98 (1), 207–210.

Zhang, G.M., Zhang, P.Y., Gao, J., Chen, Y.M., 2008a. Using acoustic cavitation to improve the bio-activity of activated sludge. Bioresour. Technol. 99 (5), 1497–1502.

Zhang, G.M., Zhang, P.Y., Yang, J., Liu, H.Z., 2008b. Energy-efficient sludge sonication: power and sludge characteristics. Bioresour. Technol. 99 (18), 9029–9031.